รายงานวิจัยฉบับสมบูรณ์

เรื่อง

ฤทธิ์ต้านเชื้อจุลชีพของสารสกัดจากต้นติ้วเกลี้ยง และ *Garcinia* sp. และสารอนุพันธ์ chalcones

Antimicrobial activities of the isolated compounds from *Cratoxylum cochinchinense*, *Garcinia* sp. and chalcone derivatives

สุชาดา จันทร์พรหมมา และคณะ

กุมภาพันธ์ 2556

สัญญาเลขที่ RSA 5280033

รายงานวิจัยฉบับสมบูรณ์

เรื่อง

ฤทธิ์ต้านเชื้อจุลชีพของสารสกัดจากต้นติ้วเกลี้ยง และ Garcinia sp. และสารอนุพันธ์ chalcones

Antimicrobial activities of the isolated compounds from *Cratoxylum cochinchinense*, *Garcinia sp*. and chalcone derivatives

คณะผู้วิจัย

1. รองศาสตราจารย์ ดร. สุชาดา จันทร์พรหมมา มหาวิทยาลัยสงขลานครินทร์
2. รองศาสตราจารย์ ดร. ฉัตรชนก กะราลัย มหาวิทยาลัยสงขลานครินทร์

สนับสนุนโดยสำนักงานกองทุนสนับสนุนการวิจัย

ทุนเพิ่มขีดความสามารถด้านการวิจัยของอาจารย์รุ่นกลางในสถาบันอุดมศึกษา

กิตติกรรมประกาศ

คณะผู้วิจัยขอขอบคุณสำนักงานกองทุนสนับสนุนการวิจัย (สกว.) ที่ได้สนับสนุนทุนวิจัย และขอขอบคุณมหาวิทยาลัยสงขลานครินทร์ ที่ได้ให้สนับสนุนทุนวิจัยสมทบจากเงินรายได้มหาวิทยาลัย ทำให้สามารถดำเนินงานวิจัยได้สำเร็จตามความมุ่งหมาย

คณะผู้วิจัยขอขอบคุณ Professor Dr. Hoong-Kun Fun, X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, Penang, Malaysia ที่ได้ให้ความช่วยเหลือในการเก็บข้อมูลการเลี้ยวเบนของรังสีเอกซ์บนผลึกเดี่ยวและคำแนะนำต่าง ๆ

คณะผู้วิจัยขอขอบคุณ ดร.ณวงศ์ บุนนาค และ นางสาวฐิติพร สุวรรณวงศ์ ผู้ช่วยวิจัย ขอขอบคุณ คุณธีรศักดิ์ อนันตพงศ์ ภาควิชาเทคโนโลยีชีวภาพ คณะอุตสาหกรรมเกษตร มหาวิทยาลัยสงขลานครินทร์ ในการทดสอบฤทธิ์ทางชีวภาพ ขอขอบคุณ Assistant Professor Dr. Arun M. Isloor, Department of Chemistry, National Institute of Technology Karnataka, India ในการช่วยเหลือด้านการทดสอบฤทธิ์ต้านการอักเสบ ขอขอบคุณผู้บริหารและเจ้าหน้าที่ของสกว. และผู้บริหารและเจ้าหน้าที่ของมหาวิทยาลัยสงขลานครินทร์ทุกท่าน ที่มีส่วนช่วยในการสนับสนุนอำนวยความสะดวกและช่วยเหลือทำให้งานวิจัยดำเนินไปได้ด้วยดี

สุชาดา จันทร์พรหมมา และ คณะ

บทคัดย่อ

Project Code: RSA 5280033

Project Title: ฤทธิ์ต้านเชื้อจุลชีพของสารสกัดจากต้นติ้วเกลี้ยง และ *Garcinia* sp. และสารอนุพันธ์ chalcones

Investigators: Principle investigator: สุชาดา จันทร์พรหมมา

Co-investigators: ฉัตรชนก กะราลัย

E-mail address: suchada.c@psu.ac.th

Objectives:

1. เพื่อหาฤทธิ์การต้านเชื้อจุลชีพของสารที่สกัดและแยกได้จากต้นติ้วเกลี้ยงและ *Garcinia sp*.
2. เพื่อสังเคราะห์และหาฤทธิ์การต้านเชื้อจุลชีพของสารอนุพันธ์ chalcones
3. เพื่อหาโครงสร้างผลึกของสารสังเคราะห์และสารผลิตภัณฑ์ธรรมชาติบริสุทธิ์ที่สามารถแยกและตกผลึกได้ เพื่อเป็นฐานข้อมูลของโครงสร้างสารผลิตภัณฑ์ธรรมชาติในการใช้ประโยชน์ เพื่อศึกษาหาความสัมพันธ์ของโครงสร้างและการออกฤทธิ์

Methodology: สกัดแยก (สารผลิตภัณฑ์ธรรมชาติ) สังเคราะห์สารอนุพันธ์ chalcones และหาโครงสร้างสารบริสุทธิ์ที่แยกหรือสังเคราะห์ได้ด้วยเทคนิคทางสเปกโตสโกปี ตกผลึก และหาโครงสร้างผลึกของสารด้วยเทคนิคการเลี้ยวเบนของรังสีเอกซ์บนผลึกเดี่ยว ทดสอบฤทธิ์ทางชีวภาพของสารที่แยกหรือสังเคราะห์ได้ คือ ฤทธิ์ต้านเชื้อแบคทีเรีย และฤทธิ์ยับยั้งเชื้อรา และศึกษาสมบัติฟลูออเรสเซนต์ของสารอนุพันธ์ chalcones

Results: จากการสกัดและแยกองค์ประกอบทางเคมีจากส่วนสกัดหยาบจากผลและยางของต้นติ้วเกลี้ยง รากและผลของต้นติ้วขน ด้วยวิธีทางโครมาโทกราฟี สามารถแยกสารประกอบประเภท xanthones จากผลอ่อนและยางของต้นติ้วเกลี้ยง 20 สาร จากรากต้นติ้วขนได้ 14 สาร และจากผลอ่อนของต้นติ้วขนได้ 9 สาร ทำการสังเคราะห์สารอนุพันธ์ chalcones จำนวน 27 สาร และหาโครงสร้างสารโดยใช้ข้อมูลทางสเปกโทรสโกปี และหาโครงสร้างด้วยเทคนิคการเลี้ยวเบนของรังสีเอกซ์บนผลึกเดี่ยวของสารที่ตกผลึกได้ และทำการทดสอบการออกฤทธิ์ทางชีวภาพ คือ ฤทธิ์การยับยั้งเชื้อแบคทีเรียและฤทธิ์การยับยั้งเชื้อรา พบสารออกฤทธิ์ทางชีวภาพที่น่าสนใจหลายสาร จากการศึกษาสมบัติฟลูออเรสเซนต์ของสารอนุพันธ์ chalcones พบสารที่แสดงสมบัติฟลูออเรสเซนต์

Discussion and Conclusion: จากการศึกษาองค์ประกอบทางเคมีและการทดสอบฤทธิ์ทางชีวภาพของสารที่แยกได้จากพืชดังกล่าวพบว่าได้สารชนิดใหม่หลายชนิดและสารที่แยกได้หลายตัวมีฤทธิ์ทางชีวภาพที่น่าสนใจและสารบางตัวมีฤทธิ์ยับยั้งแบบจำเพาะเจาะจงต่อเชื้อแบคทีเรีย และพบสารที่ออกฤทธิ์ต้านการอักเสบที่น่าสนใจอีกด้วย สำหรับสารสังเคราะห์อนุพันธ์ chalcones ไม่พบสารออกฤทธิ์ยังยั้งแบคทีเรียที่น่าสนใจ จึงได้ทำการศึกษาสมบัติฟลูออเรสเซนต์ของสาร พบสารอนุพันธ์ chalcones ที่มีสมบัติฟลูออเรสเซนต์

คำหลัก Natural Products, *Cratoxylum cochinchinense*, *Cratoxylum formosum* ssp. *pruniflorum*, crystal structure, antibacterial, anti-inflammatory, bioactivity, chalcones และ fluorescence

ABSTRACT

Project Code: RSA 5280033

Project Title: Antimicrobial activities of the isolated compounds from *Cratoxylum cochinchinense*, *Garcinia* sp. and chalcone derivatives

Investigators: Principle investigator: Suchada Chantrapromma

Co-investigators: Chatchanok Karalai

E-mail address: suchada.c@psu.ac.th

Objectives: 1. To investigate chemical constituents from the resin and green fruits of *Cratoxylum cochinchinense*; the roots and green fruit of *Cratoxylum formosum* ssp. *pruniflorum.*

2. To synthesis and study for bioactivity of chalcones

3. To study the crystal structures of the isolated compounds and the synthesized chalcones which can be crystallized.

Methodology: Extraction, isolation and characterization, and evaluation of antibacterial and antifungal activities of the isolated compounds. Synthesis chalcones by condensation reaction and study for antibacterial activity and fluorescent property. Crystallization and determination of the crystal structures by single crystal x-ray structure determination.

Results: The crude extracts of the resin and green fruit of *Cratoxylum cochinchinense*; and the roots and green fruit of *Cratoxylum formosum* ssp. *pruniflorum* were subjected to column chromatography. Twenty xanthones from resin and green fruit of *Cratoxylum cochinchinense*; fourteen xanthones from the roots and nine xanthones from the green fruit of *Cratoxylum formosum* ssp. *pruniflorum* were isolated. Twenty-seven chalcones were synthesized. The structures of the compounds were evaluated by spectroscopic methods. In addition, the structures of the compounds that can be crystallized out were also determined by single crystal X-ray diffraction. Antibacterial and antifungal activities of the isolates were evaluated. Many compounds exhibited antibacterial activity. In addition fluorescent properties of chalcones were also carried out. Some of chacones possess fluorescent property.

Discussion and Conclusion: From the studies of chemical constituents and their biological activities from these plants, several new compounds have been isolated. Several compounds possess interesting biological activities and some of them show specific antibacterial activity. Unfortunately, the biological study of chalcones found that they are not active against the tested bacterial strains. However some of the chalcones possess fluorescent property.

Keywords: Natural Products, *Cratoxylum cochinchinense*, *Cratoxylum formosum* ssp. *pruniflorum*, crystal structure, antibacterial, anti-inflammatory, bioactivity, chalcones และ fluorescence

สารบัญ

บทนำ

โครงการวิจัยนี้ประกอบด้วย 2 โครงการย่อย โดยโครงการย่อยที่ 1 ได้ทำการศึกษาองค์ประกอบทางเคมีและการออกฤทธิ์ทางชีวภาพของยางและผลอ่อนของต้นติ้วเกลี้ยง (*Cratoxylum cochinchinense*) และรากและผลอ่อนของต้นติ้วขน *Cratoxylum formosum ssp. pruniflorum* ในส่วนของพืชสกุล Garcinia sp. ดังปรากฏในชื่อโครงการ เนื่องจากการประสบปัญหาในการเก็บพันธุ์พืชในเขตวนอุทยานน้ำตกโตนงาช้างที่ จังหวัด สงขลา จึงได้ทำวิจัยในส่วนของรากและผลอ่อนของต้นติ้วขนเพิ่มเติมจากที่ทำเฉพาะติ้วเกลี้ยง ส่วนโครงการย่อยที่ 2 เป็นการสังเคราะห์และศึกษาฤทธิ์ทางชีวภาพและสมบัติฟลูออเรสเซนต์ของสารอนุพันธ์ chalcones

ติ้วเกลี้ยงและติ้วขนเป็นพืชในสกุล *Cratoxylum* ซึ่งพืชในสกุลนี้เป็นพืชที่น่าสนใจเนื่องจากมีรายงานสารออกฤทธิ์ทางชีวภาพหลายชนิด เช่น สารที่มีฤทธิ์ในการยับยั้งเชื้อแบคทีเรีย เชื้อรา เซลล์มะเร็ง และ ยับยั้งอนุมูลอิสระ เป็นต้น จึงได้ทำการหาองค์ประกอบทางเคมีจากต้นไม้ทั้งสองชนิดนี้เพื่อหาสารออกฤทธิ์ทางชีวภาพและได้พบสารออกฤทธิ์ทางชีวภาพชนิดใหม่หลายชนิด อีกทั้งได้ทำการสังเคราะห์และศึกษาฤทธิ์ทางชีวภาพของสารอนุพันธ์ chalcones และเนื่องจากผลการออกฤทธิ์ทางชีวภาพของสารอนุพันธ์ chalcones ที่ได้ในโครงการนี้ไม่เป็นที่น่าพอใจจึงได้ทำการศึกษาเพิ่มเติมในส่วนของฤทธิ์ต้านการอักเสบ และสมบัติฟลูออเรสเซนต์ของสารอนุพันธ์ chalcones อีกด้วย

โครงการย่อยที่ 1 ฤทธิ์ต้านเชื้อจุลชีพของสารที่สกัดและแยกได้จากต้นติ้วเกลี้ยงและติ้วขน

ติ้วเกลี้ยง (*Cratoxylum cochinchinense*) และติ้วขน (*Cratoxylum formosum* subsp. *pruniflorum*) เป็นต้นไม้ซึ่งอยู่ในวงศ์ Guttiferae สกุล *Cratoxylum* ถูกใช้เป็นยาพื้นบ้านในการรักษาโรค เช่น แก้โรคท้องร่วง ขับลม แก้ไข้ และ ใช้เป็นยาบำรุงกำลัง

1. ติ้วเกลี้ยง

ชื่อวิทยาศาสตร์ *Cratoxylum cochinchinense* (Lour.) *Blume*

ชื่อวงศ์ GUTTIFERAE

ชื่อพื้นเมือง ติ้วขาว ติ้วส้ม ติ้วเกลี้ยง ติ้วใบเลื่อม ขี้ติ้ว มูโต๊ะ กุ่ยฉ่องบ้าง กวยโซง กุยฉ่องเซ้า ตาวติ้วขน ติ้วแดง ติ้วยาง ติ้วเลือด ติ้วเหลือง แต้วหิน เน็กเครแย่ ราเง้ง

ลักษณะทางพฤกษศาสตร์

ต้น ผลัดใบ สูง 8 - 15 ม. ลำต้นค่อนข้างตรง เรือนยอดเป็นรูปกรวยคว่ำ กิ่งอ่อนเกลี้ยง และปลายกิ่งมักกลายสภาพเป็นหนามแข็งอยู่ตามลำต้นและกิ่งใหญ่ เปลือกนอก สีเทาเรียบหรือแตกเป็น

ร่องและเป็นสะเก็ดไปตามยาวของลำต้น เปลือกในสีเหลืองอ่อน จะมีน้ำยางเหนียว ๆ สีเหลือง อมแดง ซึมออกมาเมื่อถูกตัด

ใบ ทรงใบรูปรีหรือรูปหอก กว้าง 2 - 3.5 ซม. ยาว 4.5 - 10 ซม. โคนและปลายใบสอบ บางทีปลายใบมน ส่วนโคนใบสอบเรียว เนื้อใบบาง เกลี้ยงและมีต่อมใส ๆ กระจายทั่วไป เส้นแขนงใบมี 8 - 10 คู่ ปลายเส้นจะโค้งจรดกับเส้นถัดขึ้นไปก่อนถึงขอบใบ เส้นใบย่อยแบบเส้นร่างแหจะเห็นชัดทางด้านหลังใบ ส่วนทางท้องใบมักมีสีจางนวล ๆ ใบอ่อนจะออกสีชมพูเรื่อ ๆ ใบแก่เมื่อแก่จัดก่อนผลัดใบจะมีสีแสดหรือสีแดงอิฐ ขอบใบเรียบ ก้านใบค่อนข้างสั้น ยาวไม่เกิน 5 มม.

ดอก 2 - 5 ดอก ตามง่ามใบเหนือรอยแผลใบและตามปลายกิ่ง ทั้งกลีบรอง กลีบดอก และกลีบดอกมีอย่างละ 5 กลีบ แต่ละกลีบเป็นอิสระแก่กัน เกสรผู้มีมากและรวมกันเป็นกลุ่มสามกลุ่ม รังไข่รูปรีเกลี้ยง ๆ ภายในแบ่งเป็น 3 ช่อง แต่ละช่องมีไข่อ่อนมาก หลอดรังไข่มีสามหลอด ดอกสีส้มหรือแดงเข้มแกมขาว มีกลิ่นหอมอ่อน ๆ

ผล เกลี้ยงเป็นมัน พอแก่จะแตกออกเป็น 3 เสี่ยง เมล็ดรูปขอบขนานเล็ก ๆ มีปีกรูปมนบาง ๆ ทางด้านบนหนึ่งปีก กลีบขั้วจุกผลจะหุ้มติดผลประมาณ 2/3 ของความยาวผล ผลแก่สีน้ำตาลดำ เมล็ดจำนวนมาก

ระยะเวลาในการออกดอกและเป็นผล ออกดอกตลอดปี และมักมีดอกและผลในเวลาเดียวกัน

ด้านสมุนไพร ใบอ่อนและยอดอ่อนรับประทานสดช่วยระบายท้อง ต้นและราก ผสมลำต้นกำแพงเจ็ดชั้น ต้มน้ำดื่มแก้กระษัยเส้น เป็นยาระบาย

ด้านเป็นพืชอาหาร ใบอ่อนและยอดอ่อน กินกับอาหารพื้นเมืองทางภาคเหนือ และภาคตะวันออกเฉียงเหนือ

2. ต้นติ้วขน

ชื่อวิทยาศาสตร์ *Cratoxylum formosum* ssp. *pruniflorum*

ชื่อวงศ์ GUTTIFERAE

ชื่อพื้นเมือง กุยฉ่องเช้า (กะเหรี่ยงลำปาง) กวยโชง (กะเหรี่ยงกาญจนบุรี) ตาว (สตูล) ติ้วแดง ติ้วยาง ติ้วเลือด (พายัพ) ติ้วส้ม (นครราชสีมา) ติ้วเหลือง แต้ว **ติ้วขน** (ไทย) เตา (เลย) เน็คเคร่แย (ละว้า เชียงใหม่) ราเง้ง (เขมรสุรินทร์) มูโต๊ะ (นราธิวาส มาเลเซีย) ขี้ติ้ว ตาว (สตูล)

ลักษณะทางพฤกษศาสตร์

ต้น เป็นไม้ต้นขนาดเล็กถึงขนาดใหญ่ ผลัดใบ สูง 8 - 15 ม. เรือนยอด เป็นพุ่มกลม โปร่ง กิ่งอ่อนมีขนนุ่มทั่วไป กิ่งเล็กตามลำต้นและกิ่งใหญ่มักลายสภาพเป็นหนามแข็ง ๆ เปลือกนอก สีน้ำตาลปนดำ แตกเป็นสะเก็ดเล็ก ๆ ห้อยย้อยลง เปลือกใน สีน้ำตาลแกมเหลือง และมีน้ำยางสีเหลืองปนแดงซึมออกมา เมื่อตัดใหม่ ๆ กระพี้ขาวแยกจากแก่นเห็นได้ชัด

ใบ ใบเดี่ยว ติดตรงข้ามกันเป็นคู่ ๆ ทรงใบรูปรแกมรูปไข่กลับและรูปขอบขนาน ขนาด 2 - 5 X 3 - 13 ซม. โคนสอบเรียว ส่วนที่ค่อนไปทางปลายใบโตออก ปลายสุดสอบเข้า เนื้อบางมีต่อมใส ๆ

กระจายเต็มไปหมด หลังใบมีขนสาก ท้องใบมีขนนุ่มหนาแน่น ใบอ่อนออกสีชมพูเรื่อ ๆ ส่วนใบแก่ก่อนผลัดใบจะมีสีแดงหรือแดงอิฐ ขอบใบเรียบ ก้านใบยาวไม่เกิน 1 ซม. เส้นแขนงใบ 7 - 13 คู่ เส้นโค้งและปลายเส้นจะโค้งจรดกับเส้นถัดไปก่อนถึงขอบใบ

ดอก ออกเป็นกลุ่ม ก้านช่อสั้นออกตามง่ามใบ ดอกสีขาวหรือชมพูอ่อน ๆ กลิ่นหอมอ่อน ๆ กลีบดอกยาวเป็นสองเท่าของกลีบรองกลีบดอก กลีบรองฐานดอกและกลีบดอกมี 5 กลีบ แต่ละกลีบเป็นอิสระแก่กัน กลีบรองกลีบดอกมีขนประปราย ส่วนกลีบดอกเกลี้ยง เกสรผู้จำนวนมากสีเหลืองอ่อน แบ่งออกเป็นสามกลุ่ม รังไข่ รูปรี ๆ เกลี้ยง ภายในแบ่งเป็น 3 ช่อง แต่ละช่องมีไข่อ่อนมาก หลอดรังไข่มี 3 หลอด

ผล ผลแห้ง รูปรี ๆ ยาวประมาณ 2 ซม. หรือย่อมกว่าเล็กน้อย แข็ง มีคราบสีนวล ติดตามผิว แก่จัดแตกอ้าออกตามรอยประสานเป็น 3 แฉก เผยให้เห็นเมล็ดรูปขอบขนานเล็ก ๆ ที่มีปีกโค้ง ๆ เรียงอัดอยู่แน่น กลีบขั้วจุก ผลจะหุ้มติดผลประมาณ 1/4 ของความยาวผล แหล่งที่พบ ป่าเต็งรัง

ระยะเวลาในการออกดอกและเป็นผล ออกดอกระหว่างเดือน มกราคม-เมษายน และเป็นผลระหว่าง เดือนกุมภาพันธ์-พฤษภาคม ก่อนออกดอกจะทิ้งใบหมด ดอกจะเริ่มออกพร้อมกับการผลิใบใหม่ จึงมักมีทั้งดอกและผลติดอยู่ในเวลาเดียวกัน

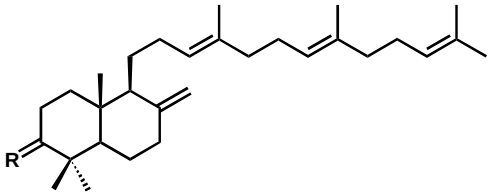
ด้านสมุนไพร ใบอ่อนและยอดอ่อนรับประทานสดช่วยระบายท้อง รากและใบใช้ต้มกินแก้ปวดท้อง เปลือกและใบตำผสมกับน้ำมันมะพร้าวทาแก้โรคผิวหนัง น้ำยางจากลำต้นใช้รักษาบาดแผลและทาแก้ฝ่าเท้าแตก

ด้านเป็นพืชอาหาร ยอดอ่อน ใบอ่อนและดอก ใช้รับประทานเป็นผัก กับอาหารประเภทส้มตำ ลาบ น้ำตก และอาหารประเภทยำต่าง ๆ หรือใส่ต้มยำต่าง ๆ เพื่อปรุงให้มีรสเปรี้ยวแทนมะนาว แต่นิยมรับประทานน้อยกว่าติ้วขาว เพาะติ้วขนมีรสชาติขมหรือฝาดกว่าติ้วขาว

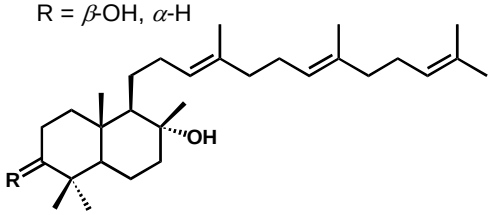
การสืบค้นและทบทวนวรรณกรรม

พืชในสกุล *Cratoxylum* จัดอยู่ในวงศ์ Guttiferae มีรายงานสารออกฤทธิ์ทางชีวภาพ ได้แก่ antibacterial (Munekazu et al., 1996 [**1**]) cytotoxicity activity (Seo et al., 2002; [**2**] Pattanaprateeb et al., 2005 [**3**]) immunomodulatory activity (Pinto et al., 1997 [**4**]) ติ้วเกลี้ยงและติ้วขนเป็นต้นไม้ในวงศ์ Guttiferae สกุล *Cratoxylum* ถูกใช้เป็นยาพื้นบ้านในการรักษาโรค เช่น แก้โรคท้องร่วง ขับลม แก้ไข้ ยาบำรุงกำลัง จากการสำรวจเอกสารที่เกี่ยวข้องกับพืชสกุล *Cratoxylum* มีรายงานการวิจัยดังนี้

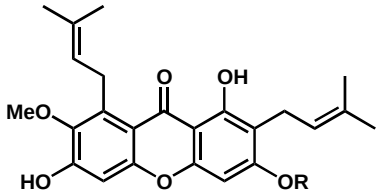
ในปี 1993 Bennett และคณะ ได้ทำการศึกษาในส่วนเปลือกของ *C. cochinchinense* สามารถแยกสารในกลุ่มของ bicyclic triterpenoids และ xanthones ได้ดังนี้



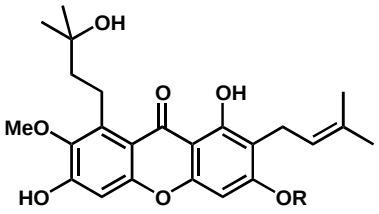
R = H_2
R = β-OH, α-H



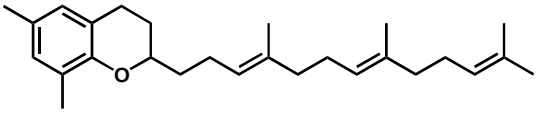
R = O
R = β-OH, α-H

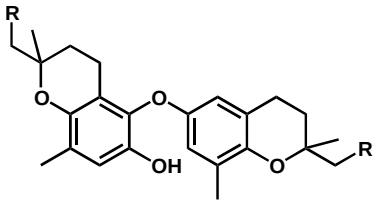


R = H
R = Me

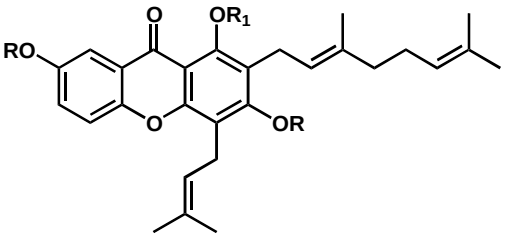


R = H
R = Me

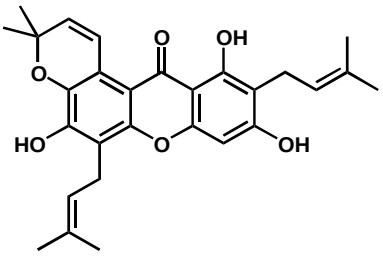


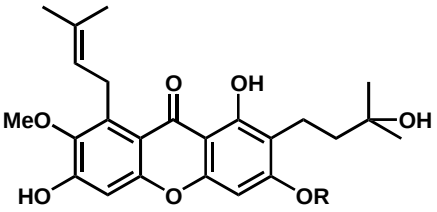


R = Farmesyl

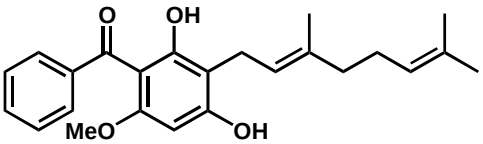


R = H, Me, Me
R_1 = H, H,
Me

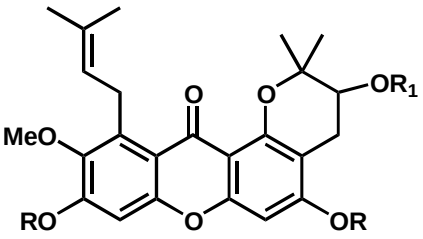




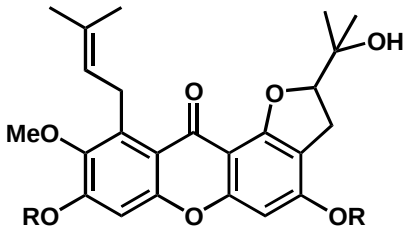
R = H
R = Me

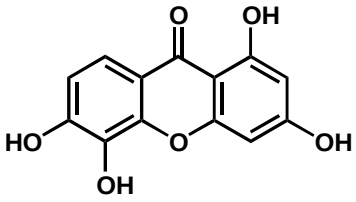


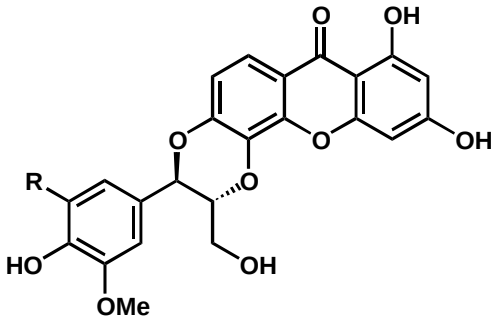
ในปี 1995 Sia และคณะ ได้ทำการศึกษาในส่วนเปลือกของ *C. cochinchinense* สามารถแยกสารในกลุ่มของ xanthones และ bis-xanthones ได้ดังนี้

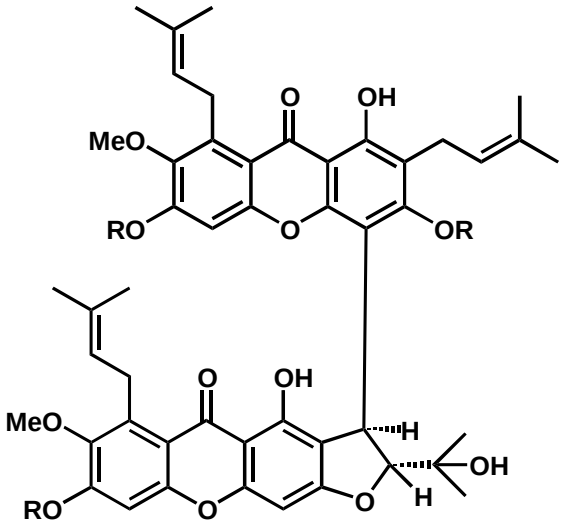


R = H, Me, Me
R_1 = H, H, Ac



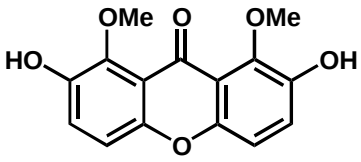


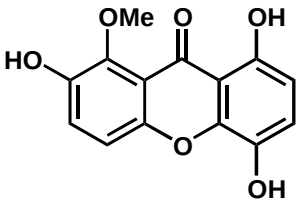


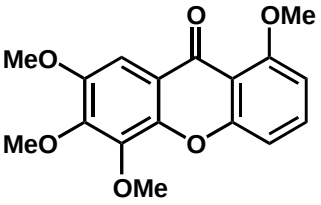


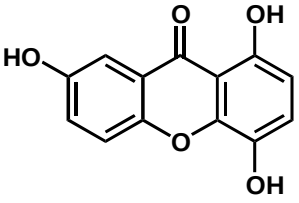
R = Me
R = H

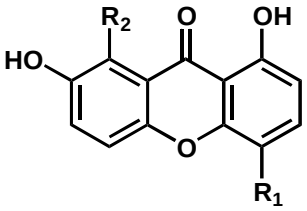
ในปี 1996 Ilnuma และคณะ ได้ทำการศึกษาในส่วนเปลือกของ *C. formosum* สามารถแยกสารในกลุ่มของ xanthones และ flavonoids ได้ดังนี้



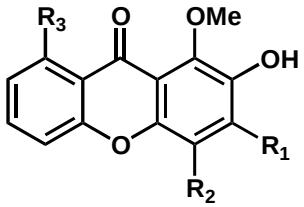




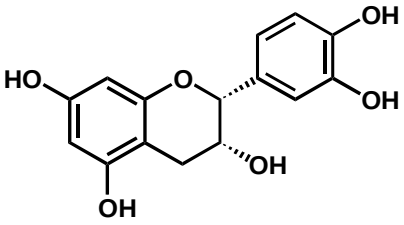


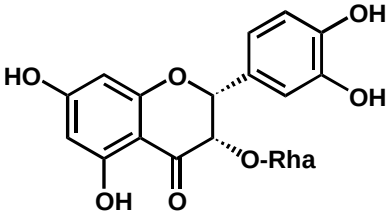


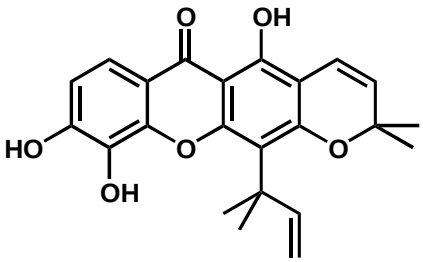
R_1 = OMe, H, H
R_2 = H, H, OMe

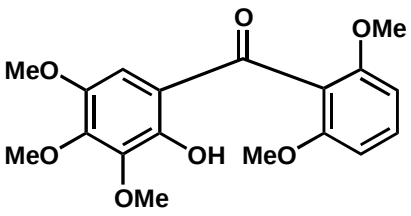


R_1 = OMe, OH, OMe
R_2 = OMe, OH, OMe
R_3 = OMe, H, H

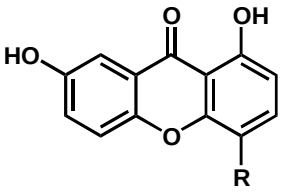




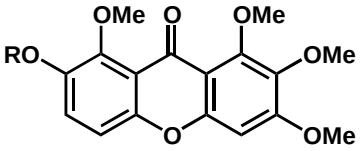




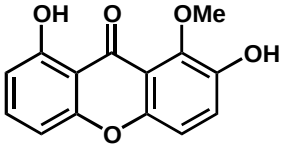
ในปี 1998 Kijjoa และคณะ ได้ทำการศึกษาในส่วนเนื้อไม้ของ *C. maingayi* สามารถแยกสารในกลุ่มของ xanthones ได้ดังนี้



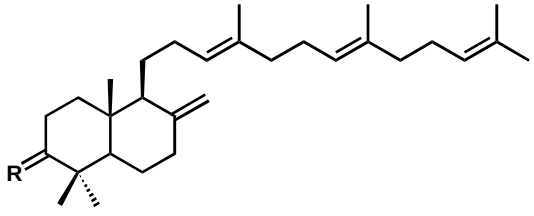
R = H
R = OMe



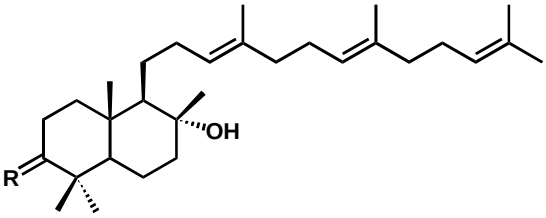
R = H
R = Ac



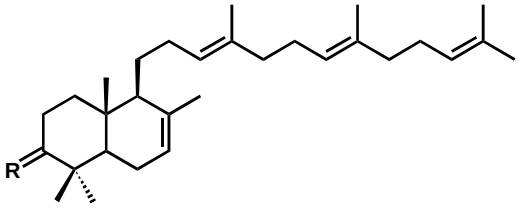
ในปี 1998 Nguyen และคณะ ได้ทำการศึกษาในส่วนเนื้อไม้ของ *C. cochinchinense* สามารถแยกสารในกลุ่มของ bicyclic triterpenoids, xanthones และ anthraquinone ได้ดังนี้



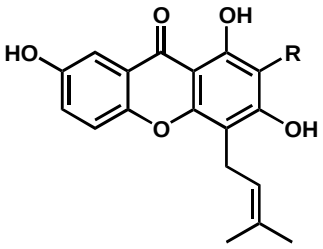
R = α–H, β–OH
R = H_2



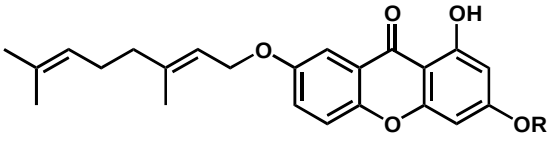
R = H_2
R = O
R = α-H, β-H



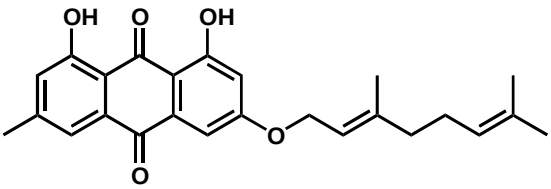
R = α–H, β–H
R = H_2
R = α–H, β–H
R = O



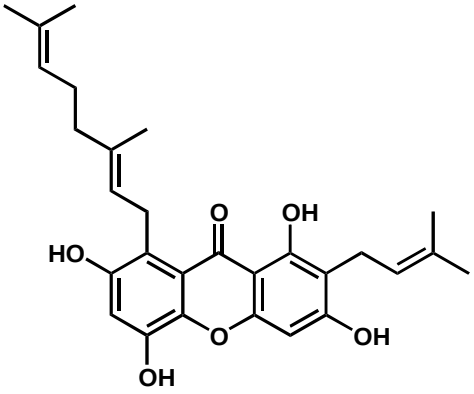
R = geranyl
R = prenyl

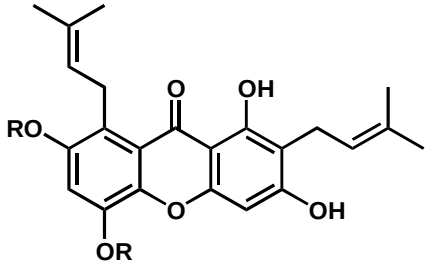


R = H
R = Me

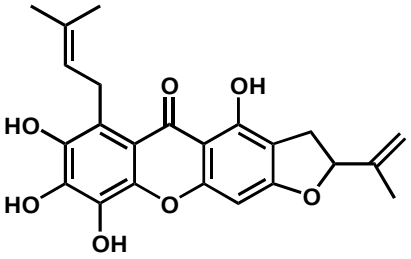


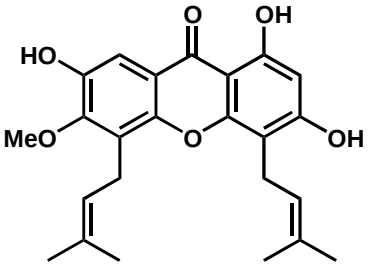
ในปี 2002 Seo และคณะ ได้ทำการศึกษาในส่วนเนื้อไม้ของ *C. sumatranum* สามารถแยกสารในกลุ่มของ anthraquinobenzophenones และ xanthones ได้ดังนี้

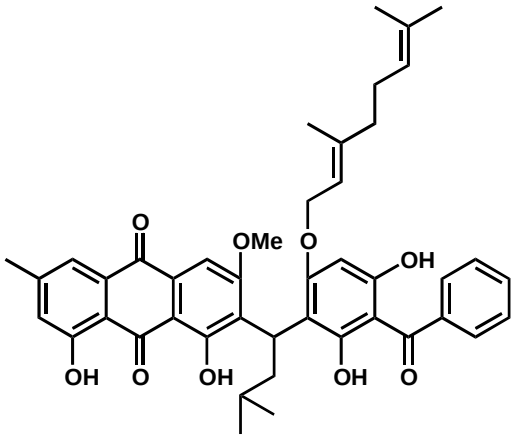


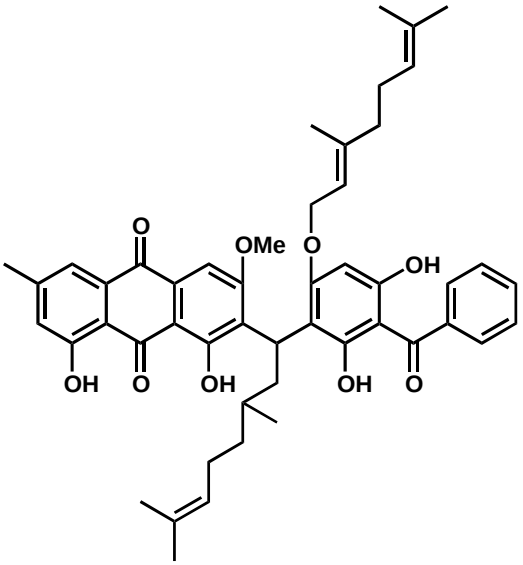


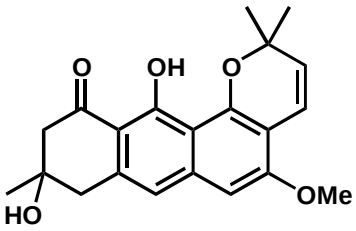
R = H
R = Me

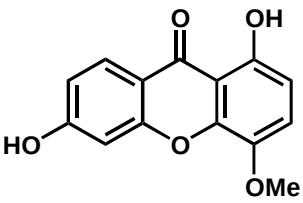




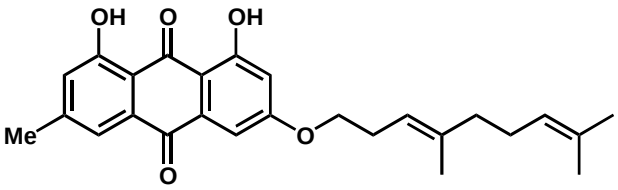


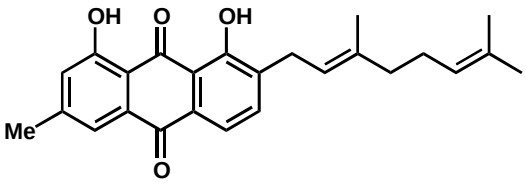


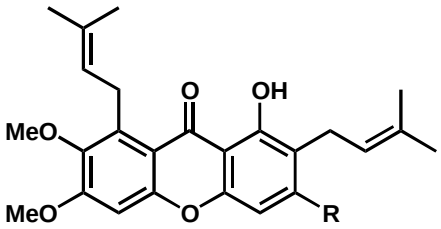


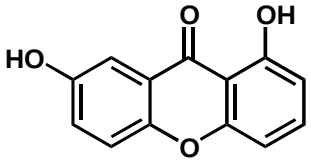


ในปี 2005 Pattanaprateeb และคณะ ได้ทำการศึกษาในส่วนเปลือกของ *C. aborecens* สามารถแยกสารในกลุ่มของ anthraquinones และ xanthones ได้ดังนี้









R = OH
R = OMe

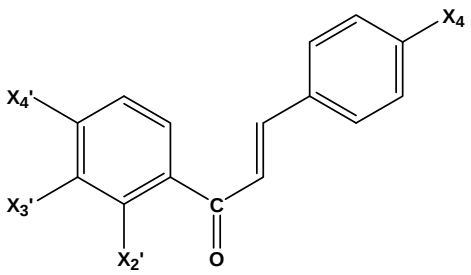
โครงการย่อยที่ 2 ฤทธิ์ต้านเชื้อจุลชีพของสารสังเคราะห์อนุพันธ์ chalcones

สารกลุ่ม chalcones เป็นสารประกอบที่พบได้ในผลิตภัณฑ์ธรรมชาติหลายชนิด ปัจจุบันการศึกษาเกี่ยวกับสารประกอบกลุ่ม chalcones ทั้งที่ได้จากผลิตภัณฑ์ธรรมชาติและสารสังเคราะห์ได้รับความสนใจเพิ่มขึ้น เนื่องจากพบว่าสารกลุ่มนี้มีฤทธิ์ทางเภสัชวิทยาหลายแบบด้วยกัน เช่น antimicrobial, antifungal, antioxidant, cytotoxic and anti-inflammatory (Dimmock et al., 1999; Go et al., 2005) รวมถึงการใช้ประโยชน์ทางด้านการถนอมอาหาร (Dhar, 1981) แม้ว่าสารกลุ่ม chalcones ที่ได้จากธรรมชาติจะแสดงฤทธิ์ที่น่าสนใจแต่ปัญหาคือปริมาณที่ถูกสกัดและแยกจากผลิตภัณฑ์ธรรมชาติจะมีปริมาณที่ไม่มากนัก กอร์ปกับการประยุกต์ใช้ของสารกลุ่มนี้มีหลายอย่างด้วยกัน ดังนั้นกลุ่มผู้วิจัยจึงสังเคราะห์สารอนุพันธ์ chalcones เพื่อทำการศึกษาฤทธิ์การต้านเชื้อจุลชีพเพื่อใช้เป็นข้อมูลประกอบกับสารที่ได้จากผลิตภัณฑ์ธรรมชาติ

การสืบค้นและทบทวนวรรณกรรม

จากการสำรวจเอกสารการวิจัยที่เกี่ยวข้องกับฤทธิ์ทางเภสัชวิทยาสารกลุ่ม chalcones มีดังนี้

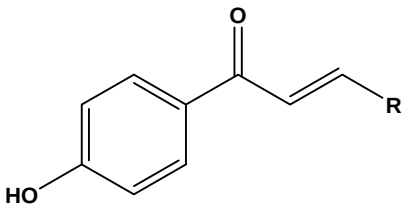
Alcaradz *et al*., 2008. สังเคราะห์และศึกษา bioactivity ของสารประกอบ chalcones โดยศึกษาการออกฤทธิ์ต่อการต้านเชื้อแบคทีเรีย *Staphylococcus aureus*



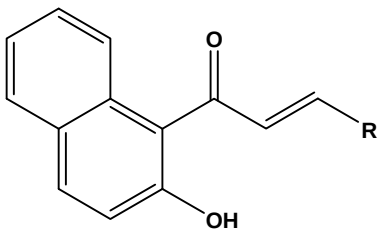
(1) Chalcone	$X_{2'} = X_{3'} = X_{4'} = X_4 = H$			
(2) 4(OH)-chalcone	$X_4 = OH$	$X_{2'} = X_{3'} = X_{4'} = H$		
(3) 4(OCH_3)-chalcone	$X_4 = OCH_3$	$X_{2'} = X_{3'} = X_{4'} = H$		
(4) 4(F)-chalcone	$X_4 = F$	$X_{2'} = X_{3'} = X_{4'} = H$		
(5) 4(Cl)-chalcone	$X_4 = Cl$	$X_{2'} = X_{3'} = X_{4'} = H$		
(6) 2'(OH)-chalcone	$X_{2'} = OH$	$X_{3'} = X_{4'} = X_4 = H$		
(7) 2',4'$(OH)_2$-chalcone	$X_{2'} = X_{4'} = OH$	$X_{3'} = X_4 = H$		
(8) 2',4$(OH)_2$-chalcone	$X_{2'} = X_4 = OH$	$X_{3'} = X_{4'} = H$		
(9) 2'(OH),4'(OCH_3)-chalcone	$X_{2'} = OH$	$X_{3'} = X_4 = H$	$X_{4'} = OCH_3$	
(10) 2'(OH),4(OCH_3)-chalcone	$X_{2'} = OH$	$X_{3'} = X_{4'} = H$	$X_4 = OCH_3$	
(11) 2',4'$(OH)_2$-3'(OCH_3)-chalcone	$X_{2'} = X_{4'} = OH$	$X_{3'} = OCH_3$	$X_4 = H$	

และพบว่า สารประกอบ **6** **7** และ **8** มีฤทธิ์ในการต้านแบคทีเรีย *S. aureus*

Prasad *et al*., 2008. สังเคราะห์สารอนุพันธุ์ของ chalcone เพื่อศึกษาคุณสมบัติในการยับยั้งแบคทีเรีย *Bacillus pumilis*



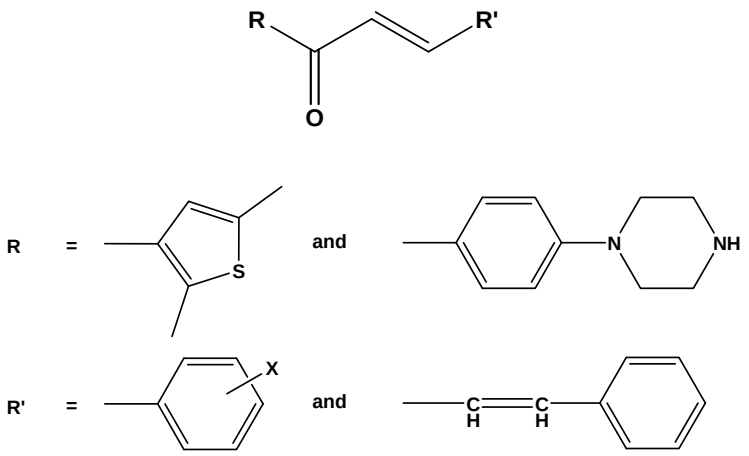
1-11



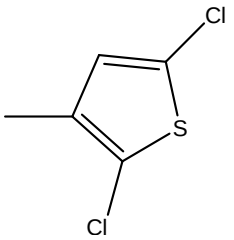
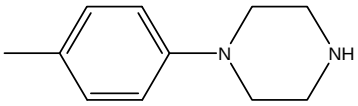
12-24

Compound	R
1	anthryl
2	5-bromo furyl
3	2,4,5-trimethoxy phenyl
4	4-hydroxy-3methoxy phenyl
5	3-bromo-5-chloro-2-hydroxy phenyl
6	2,4-dichloro phenyl
7	3,4,5-trimethoxy phenyl
8	2-pyridyl
9	2-quinolyl
10	4-quinolyl
11	3-pyridyl
12	4-methoxy phenyl
13	3,4,5-trimethoxy phenyl
14	phenyl
15	2-hydroxy phenyl
16	3-bromo phenyl
17	4-dimethyl amino phenyl
18	2,4,5-trimethoxy phenyl
19	4-nitro phenyl
20	4-methyl phenyl
21	9-anthryl
22	2-chloro phenyl
23	2,4-dichloro phenyl
24	3,4-dimethoxy phenyl

Tomar *et al*., 2007. สังเคราะห์สารประกอบอนุพันธ์ chalcone และศึกษาสมบัติ antimicrobial activity ต่อแบคทีเรีย Gram-positive bacteria เช่น แบคทีเรีย *Staphylococcus aureus* และ *Escherichia coli*

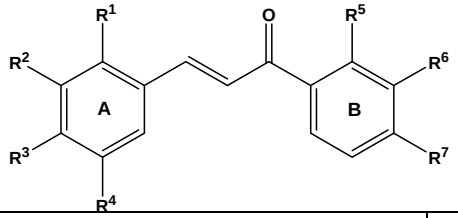


X = H, 3-CH_3, 4-CH_3, 4-OCH_3, 3,4,5-trimethoxy, 4-Cl and 3-NO_2

compound	R	R' (benzaldehyde)
1		H
2		3-CH_3
3		4-CH_3
4		4-OCH_3
5		3,4,5-Trimethoxy
6		4-Cl
7		3-NO_2
8		Cinnamaldehyde
9		H
10		3-CH_3
11		4-CH_3
12		4-OCH_3
13		3,4,5-Trimethoxy
14		4-Cl
15		3-NO_2
16		Cinnamaldehyde

พบว่า สารประกอบ **7 9 10 12** และ **15** มีฤทธิ์ในการต้าน *S. aureus* ได้ดี สารประกอบ **9** และ **15** มีฤทธิ์ในการต้าน *E. coli* ได้ดี ในขณะที่สารประกอบตัวอื่นๆแสดงฤทธิ์ในการต้านเชื้อแบคทีเรียที่ไม่โดดเด่น

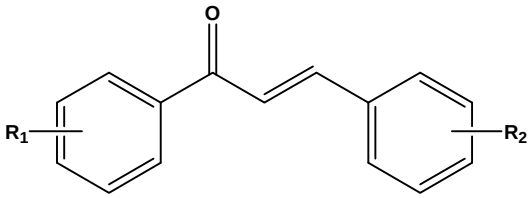
Sivakumar *et al*., 2007. สังเคราะห์และศึกษา bioactivity ของสารประกอบกลุ่ม chalcones โดยศึกษาการมีฤทธิ์ antimycobacterial activity และ quantitative structure-activity relationship (QSAR) ของสาร



Compound	A-ring				B-ring		
	R^1	R^2	R^3	R^4	R^5	R^6	R^7
C_1			NMe_2				
C_2			SMe				
C_3			SMe				NO_2
C_4							
C_5			SMe			OH	
C_6			SMe				OMe
C_7			SMe		OH		
C_8			SMe				Me
C_9			SMe				Cl
C_{10}			SMe			NO_2	
C_{11}			OMe				
C_{12}			SMe		Cl		Cl
C_{13}	OMe						
C_{14}			SMe				Br
C_{15}		OMe	OMe	OMe			
C_{16}			SMe				OH
C_{17}			OMe	OMe	Cl		Cl
C_{18}	OMe		OMe			NO_2	
C_{19}			OMe	OMe		NO_2	
C_{20}			NMe_2			NO_2	
C_{21}	OMe						NO_2
C_{22}			NMe_2		Cl		Cl
C_{23}			OMe				Me
C_{24}			OMe			OH	
C_{25}	OMe					NO_2	

พบว่า สารประกอบ C_{21} และ C_{24} มีประสิทธิภาพมากในการมีฤทธิ์ antitubercular activity

Bowman *et al.,* 2007. สังเคราะห์สารอนุพันธุ์ของ chalcone เพื่อศึกษาคุณสมบัติในการยับยั้งแบคทีเรีย *S. aureus*



4At: R_1 = 4-OH, R_2 = 2,4-Cl

4Be: R_1 = 3-OH, R_2 = 3-Br

4Bl: R_1 = 3-OH, R_2 = 2,3-Cl

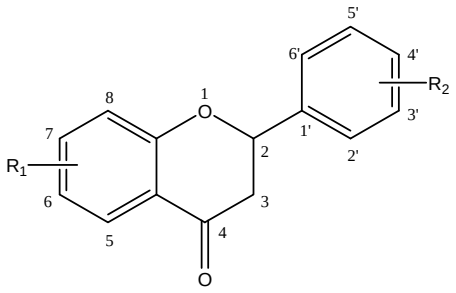
4Bo: R_1 = 3-OH, R_2 = 2,3,6-Cl

4Bv: R_1 = 3-OH, R_2 = 3,5-CF3

4Cf: R_1 = 3-OMe, 4-OH, R_2 = 4-Br

พบว่า สารประกอบ **4Bo** และ **4Bv** มีประสิทธิภาพสูงในการยับยั้งเชื้อแบคทีเรีย *S. aureus*

Moorthy *et al*., 2006. สังเคราะห์สารอนุพันธ์ของ flavanone เพื่อศึกษาคุณสมบัติในการยับยั้งแบคทีเรีย *Staphylococcus aureus*, *Shiegella sonnei*, *Shiegella dysenteriae*, *Salmonella typhimurium*, *Vibrio cholerae* และ *Escherichia coli*

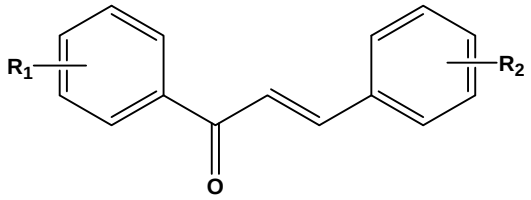


Compound	R_1	R_2
2a	H	H
2b	H	4’-OH
2c	H	4’-OCH_3
2d	H	4’-$N(CH_3)_2$
2e	7-OH	6’-OH

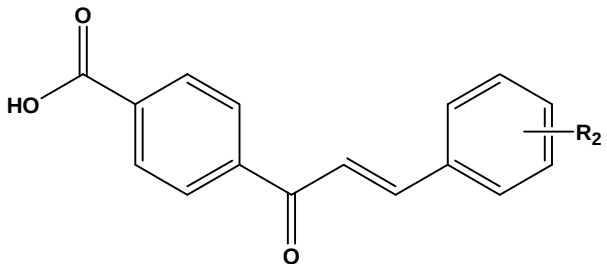
Compound	R_1	R_2
2f	7-OH	4'-$N(CH_3)_2$
2g	7-OH	6'-OH
2h	6-OH	6'-OH
2i	6-OH	4'-OH
2j	6-OH	4'-OCH_3
2k	6-OH	4'-$N(CH_3)_2$

พบว่าสารประกอบ **2b 2e 2f** และ **2h** มีฤทธิ์ในการยับยั้งเชื้อแบคทีเรีย

Nielsen *et al*., 2004. สังเคราะห์สารอนุพันธุ์ของ chalcone เพื่อศึกษาคุณสมบัติในการยับยั้งแบคทีเรีย *S. aureus* ATCC 29213



Compound	**R_1**	**R_2**
1	4-OH	2,4-Di-Cl
2	2-F-4-OH	2,4-Di-Cl
3	3-F-4-OH	2,4-Di-Cl
4	3,5-Di-F-4-OH	2,4-Di-Cl
5	4-COOH	2,4-Di-Cl
6	4-Tetrazolyl	2,4-Di-Cl



Compound	R_2	Compound	R_2
7	H	**19**	3-OPh
8	4-CF_3	**20**	2-CF_3
9	4-Cl	**21**	2-Br
10	4-CH_3	**22**	2-Cl
11	4-OCH_3	**23**	2-OH
12	4-OH	**24**	2-OBu
13	4-OPh	**25**	3,5-Di-CF_3
14	3-CF_3	**26**	3,5-Di-Br
15	3-Br	**27**	3,5-Di-Cl
16	3-Cl	**28**	3,5-Di-CH_3
17	3-NO_2	**29**	3,5-Di-F
18	3-OH	**30**	3,5-Di-OCH_3

พบว่าสารประกอบ **1 2 25** และ **26** มีฤทธิ์ในการยับยั้งเชื้อแบคทีเรีย *S. aureus* ได้ดีมาก

2. ผลการวิจัยและอภิปรายผล (ติ้วเกลี้ยง)

การสกัดและแยกสารจากต้นติ้วเกลี้ยง

2.1 General experimental procedures

Melting points were determined on the Fisher-John melting point apparatus. Optical rotations were measured on a JASCO P-1020 digital polarimeter. UV and IR spectra were recorded on SPECORD S 100 (Analytikjena) and Perkin-Elmer FTS FT-IR spectrophotometer, respectively. The ^{1}H and ^{13}C NMR spectra were recorded on a 500 MHz Varian UNITY INOVA and/or 300 MHz Bruker FTNMR Ultra Shield^{TM} spectrometers in $CDCl_3$ or CD_3OD with TMS as the internal standard. Chemical shifts are reported in δ (ppm) and coupling constants (*J*) are expressed in hertz. EI and HREI mass spectra were measured on a Kratos MS 25 RFA spectrometer. Quick column chromatography (QCC) and column chromatography (CC) were carried out on silica gel 60 F_{254} (Merck) and silica gel 100 (Merck), respectively.

2.2 Plant materials

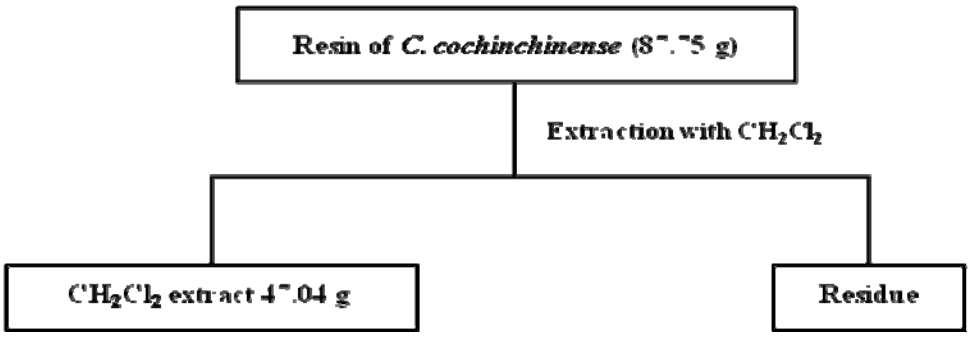
The resin and green fruits of *Cratoxylum cochinchinense*

The resin of *C. cochinchinense* was collected in October 2003 at Prince of Songkla University, Hat-Yai campus, whereas the green fruits of *C. cochinchinense* were collected in October 2007 at Kaun Kha Long District, Satun Province. Botanical identification was achieved through comparison with a voucher specimen No. SL-1 (PSU) in the herbarium of collection of Department of Biology, Faculty of Science, Prince of Songkla University, Thailand.

2.3 Extraction and Isolation

2.3.1 The extraction of the resin of *Cratoxylum cochinchinense*

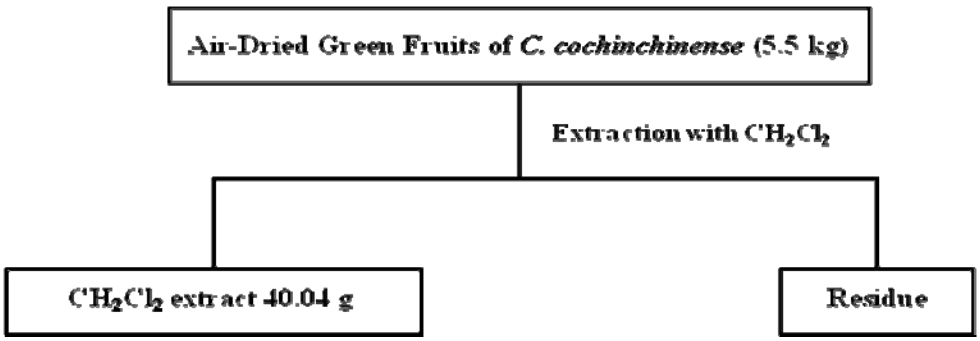
The resin of *C. cochinchinense* (87.75 g) was extracted with CH_2Cl_2 (2×2.0 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (47.04 g) (see **Scheme 1**).



Scheme 1 The extraction of the resin of *C. cochinchinense*

2.3.2 The extraction of the green fruits of *Cratoxylum cochinchinense*

Air-dried green fruits of *C. cochinchinense* (5.5 kg) were extracted with CH_2Cl_2 (2×20 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (40.04 g) (see **Scheme 2**).

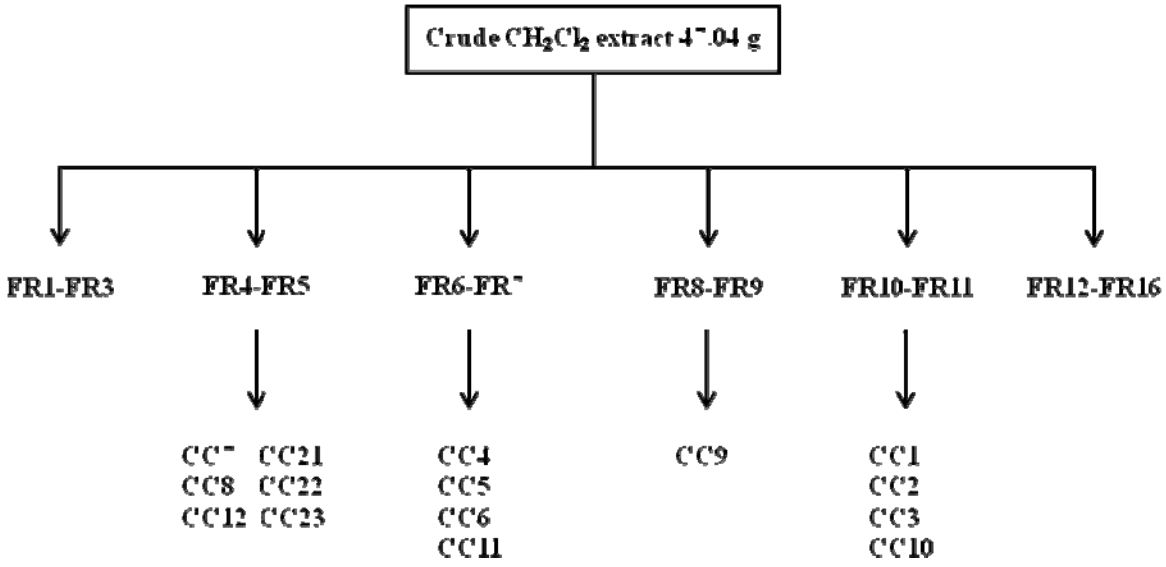


Scheme 2 The extraction of the green fruits of *C. cochinchinense*

2.4 *การสกัดและแยกสารจากยางและผลอ่อนต้นติ้วเกลี้ยง*

2.4.1 The CH_2Cl_2 extract of the resin of *Cratoxylum cochinchinense*

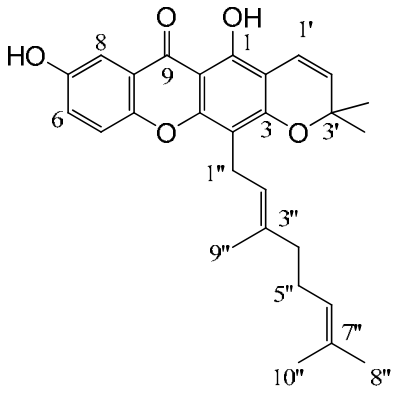
The crude CH_2Cl_2 extract (47.04 g) of the resin of *C. cochinchinense* was subjected to QCC (Quick column chromatography) on silica gel (Merck 60 F_{254}) using hexane as a first eluent and then increasing the polarity with acetone to give 16 fractions (FR1-FR16). Fractions FR4 and FR5 were separated by CC eluting with a gradient of acetone-hexane to give 8 subfractions (FR4A-FR4H) and **CC8** (150.2 mg). Subfraction FR4B was further purified by CC and eluted with a gradient of acetone-hexane to give **CC7** (31.4 mg), **CC12** (56.5 mg), **CC21** (24.5 mg) and a mixture of compounds **CC22** and **CC23** (78.5 mg), respectively. Fractions FR6 and FR7 were separated by QCC and eluted with a gradient of CH_2Cl_2-hexane to give 6 subfractions (FR6A-FR6F). Subfraction FR6B was further separated by QCC eluting with a gradient of acetone-hexane to give **CC6** (35.7 mg), **CC8** (83.2 mg) and **CC11** (1.8 mg). Subfraction FR6E was purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give **CC4** (849.4 mg) and **CC5** (1.25 g). Fraction FR8 and FR9 were separated by QCC and eluted with a gradient of CH_2Cl_2-hexane to afford **CC5** (551.3 mg), **CC7** (18.2 mg), **CC8** (116.6 mg) and **CC9** (148.7 mg). Fraction FR10 and FR11 were separated by QCC and eluted with a gradient of acetone-hexane to give 7 subfractions (FR10A-FR10G) and **CC7** (25.9 mg). Subfraction FR10B was further purified by CC on silica gel C-18 and eluted with MeOH to furnish **CC1** (8.5 mg). Subfraction FR10D was separated by QCC eluting with a gradient of acetone-hexane to give 6 subfractions (FR10D1-FR10D6). Subfraction FR10D2 was further separated by CC and eluted with a gradient of acetone-hexane to give 5 subfractions (FR10D2A-FR10D2E), **CC2** (1.8 mg), **CC4** (23.1 mg), **CC5** (34.2 mg) and an inseparable mixture of **CC2** and **CC3** (24.2 mg). The mixture was separated by acetylation with Ac_2O (0.1 mL) in pyridine (2.0 mL) and stirred over night at the room temperature to give yellow gum which was further purified by CC eluting with 70% $CHCl_3$-hexane to give acetylated derivatives **CC2a** (5.1 mg) and **CC3a** (18.9 mg), respectively. Subfraction FR10D2E was purified by CC and eluted with 80% CH_2Cl_2-hexane to give **CC8** (16.7 mg), **CC10** (5.0 mg) and **CC11** (1.5 mg), respectively (see **Scheme 3**).



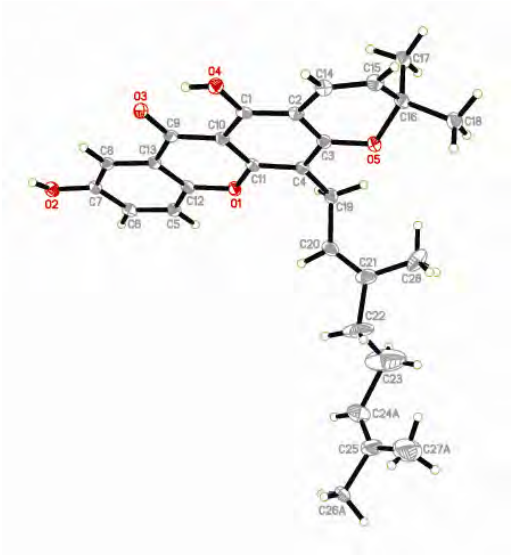
Scheme 3 Isolation of compounds **CC1**-**CC12** and **CC21**-**CC23**

Characterization of isolated compounds

***1. Compound* CC1**: *Cochinchinone I*



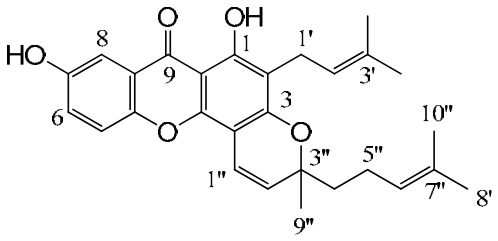
The structure of compound **1**



The X-ray structure of compound **1**

***Compound* CC1**: *Cochinchinone I.* Yellow needle single crystals, m.p. 160-162 $^{\circ}$C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 261 (4.01), 297 (4.31), 342 (3.69), 393 (3.46) nm; FT-IR (neat) ν_{max} 3406, 1650, 1612 cm^{-1}; HRMS *m/z* 446.2279 for $C_{28}H_{30}O_5$ (calcd. 446.2093). EIMS *m/z* (rel. int.): 446 $[M]^+$ (76), , 431 (100), 377 (73), 363 (76), 323 (26), 307 (15), 295 (13), 137 (5), 69 (6). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.1**. (Boonnak *et al*., 2009)

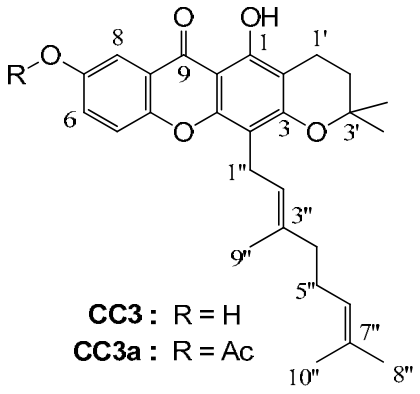
***2. Compound* CC2**: *Cochinchinone J*



The structure of compound **2**

***Compound* CC2**: *Cochinchinone J.* Yellow viscous oil, $[\alpha]^{25}_{D}$ = -69.8 (*c* 0.08, $CHCl_3$); UV-Vis ($CHCl_3$) λ_{max} (log ε) 243 (3.90), 289 (4.10), 298 (4.13), 320 (3.68), 351 (3.47), 391 (3.33) nm; FT-IR (neat) ν_{max} 3397, 1649, 1613 cm^{-1}; HRMS *m/z* 446.2092 for $C_{28}H_{30}O_5$ (calcd. 446.2093). EIMS *m/z* (rel. int.): 446 $[M]^+$ (11), 363 (100), 307 (18), 69 (5). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.2**. (Boonnak *et al*., 2009)

***3. Compound* CC3**: *Cochinchinone K.*

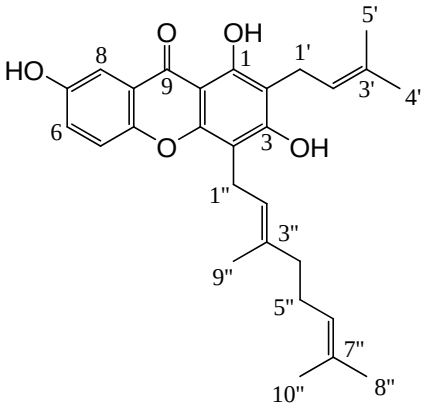


The structure of compound **3** and **3a**

***Compound* CC3**: *Cochinchinone K.* Compound **CC3** was isolated as a mono-acetylated form (**CC3a**) from an inseparable mixture with compound **CC2**, and the mixture was thus acetylated with Ac_2O in pyridine. The resulting product was further separated by CC eluting with 70% $CHCl_3$-hexane to give monoacetates **CC3a** (18.9 mg) and **CC2a** (5.1 mg), respectively. The latter was confirmed as an acetylated derivative of **CC2** (**CC2a**) by comparison of its spectral data with those of compound **CC2.** Yellow powder, m.p. 85-87 ^{o}C; UV ($CHCl_3$) λ_{max} (log ε) 243, 271, 303, 326 and 383 nm; IR (neat) ν_{max} 3392, 1709, 1648, 1612 cm^{-1}; HRMS *m/z* 490.2355 for $C_{30}H_{34}O_6$ (calcd. 490.2355). EIMS *m/z* (rel. int.): 490 $[M]^+$ (53), 473 (53), 419 (44), 405 (31), 365 (100), 323 (52), 311 (41), 267 (51), 69 (24). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.3**. (Boonnak *et al*., 2009)

***Compound* CC3a** is a yellow powder, m.p. 85-87 °C. The HREIMS spectrum showed a molecular ion peak at *m/z* 490.2355 $[M]^+$, corresponding to $C_{30}H_{34}O_6$. The UV spectrum showed absorption bands of a xanthone at 243, 271, 303, 326 and 383 nm (Boonnak *et al*., 2009), while the IR spectrum exhibited the hydroxyl and conjugated carbonyl functionalities at ν_{max} 3392 and 1648 cm^{-1} (Boonnak *et al*., 2009), respectively.

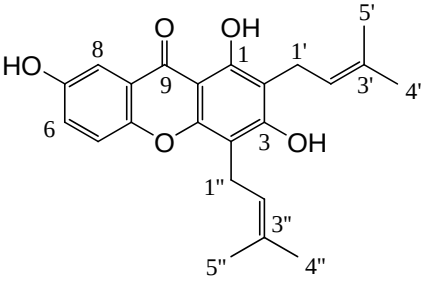
***4. Compound* CC4**: *Cochinchinone A*



The structure of compound **4**

***Compound* CC4**: *Cochinchinone A.* Pale-yellow powder, m.p. 119-120 °C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 232 (4.44), 268 (4.42), 316 (4.04), 384 (3.70) nm; FT-IR (neat) ν_{max} 3413, 1641 cm^{-1}; For ^1H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.4**. (Mahabussarakam *et al*., 2006)

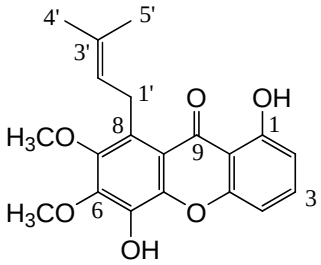
***5. Compound* CC5**



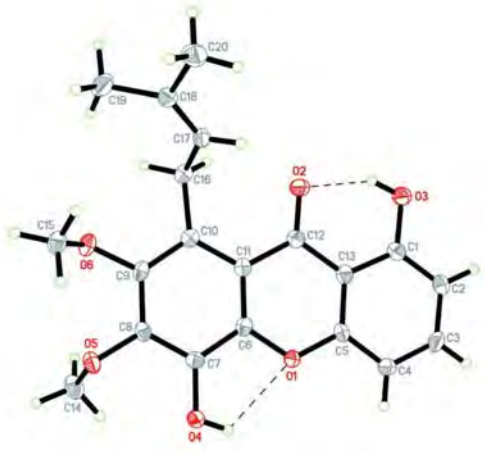
The structure of compound **5**

***Compound* CC5**: *1,3,7-trihydroxy-2,4-diisoprenylaxanthone.* Pale-yellow powder. UV-Vis ($CHCl_3$) λ_{max} (log ε) 234, 267, 317, 386 nm; FT-IR (neat) ν_{max} 3400, 1645 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.5**. (Iinuma *et al*., 1996; Nguyen and Harrison 1998)

6. Compound CC6



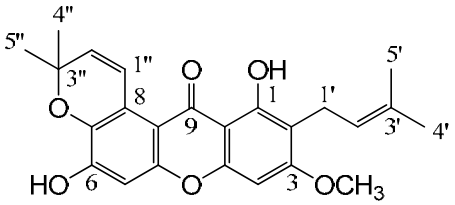
The structure of compound **6**



The X-ray structure of compound **6**

***Compound* CC6**: *Celebixanthone methyl ether.* Yellow needle-single crystals, m.p. 172-174 $^{\circ}C$; UV-Vis ($CHCl_3$) λ_{max} (log ε) 243 (3.90), 289 (4.10), 298 (4.13), 320 (3.68), 351 (3.47), 391 (3.33) nm; FT-IR (neat) ν_{max} 3397, 1649, 1613 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.6**. (Stout *et al*., 1963; Dechathai *et al*., 2006; Boonnak *et al*., 2007)

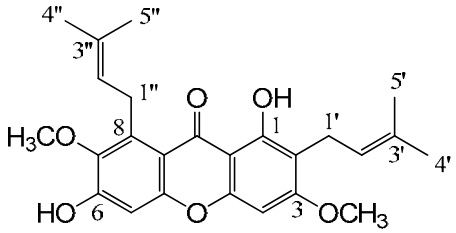
***7. Compound* CC7**: *Dulcisxanthone F*



The structure of compound **7**

***Compound* CC7**: *Dulcisxanthone F.* Yellow needle-single, m.p. 213-215 °C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 244 (4.54), 265 (4.53), 322 (4.43), 331 (4.44) nm; FT-IR (neat) ν_{max} 3479, 1675 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.7**. (Boonnak *et al*., 2006)

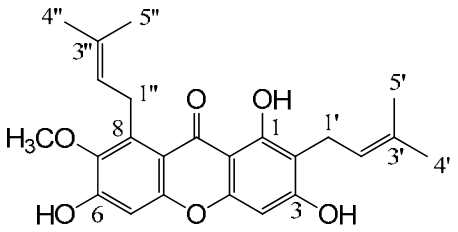
***8. Compound* CC8**: β*-Mangostin*



The structure of compound **8**

***Compound* CC8**: β*-Mangostin.* Yellow needle-single crystals, m.p. 172-174 °C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 243 (3.90), 289 (4.10), 298 (4.13), 320 (3.68), 351 (3.47), 391 (3.33) nm; FT-IR (neat) ν_{max} 3397, 1649, 1613 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.8**. (Mahabusarakam *et al*., 1987; Chantrapromma *et al*., 2006)

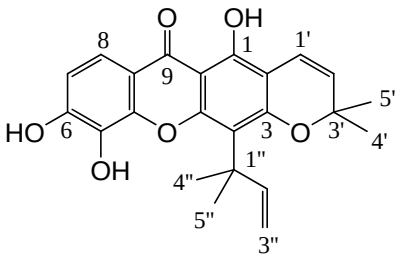
***9. Compound* CC9**: *α-Mangostin*



The structure of compound **9**

***Compound* CC9**: *α-Mangostin.* Deep-yellow powder, m.p. 180-182 °C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 243 (3.90), 289 (4.10), 298 (4.13), 320 (3.68), 351 (3.47), 391 (3.33) nm; FT-IR (neat) ν_{max} 3397, 1649, 1613 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.9**. (Mahabusarakam *et al*., 1987)

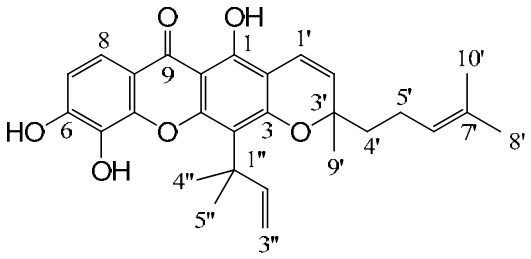
***10. Compound* CC10**: *Macluraxanthone*



The structure of compound **10**

***Compound* CC10**: *Macluraxanthone.* Brown-yellow solid. m.p. 183-184 °C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 240 (4.28), 283 (4.62), 338 (4.25) nm; FT-IR (neat) ν_{max} 3446, 1649 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.10**. (Delle Monache *et al*., 1981)

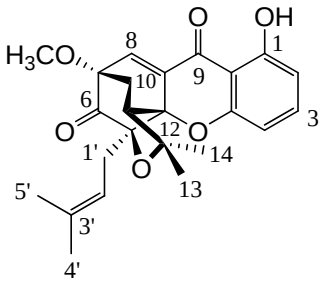
***11. Compound* CC11**: *Pruniflorone G*



The structure of compound **11**

***Compound* CC11**: *Pruniflorone G.* Brown powder, m.p. 143-145 °C; $[\alpha]^{27}_{D}$ = -7.4 (*c* 0.425, $CHCl_3$); UV-Vis ($CHCl_3$) λ_{max} (log ε) 243 (4.56), 288 (4.81), 335 (4.53) nm; FT-IR (neat) ν_{max} 3414, 1649, 1628, 1580 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.11**. (Boonnak *et al*., 2006)

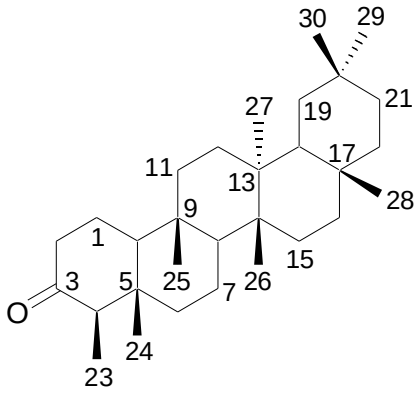
***12. Compound* CC12**: *Cochinchinone C*



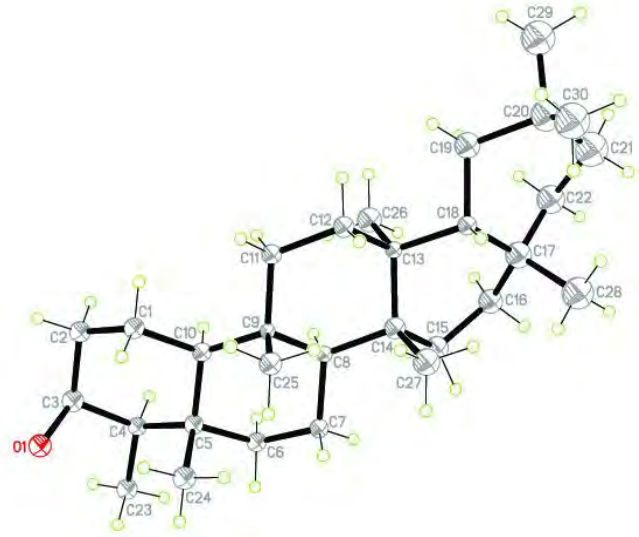
The structure of compound **12**

***Compound* CC12**: *Cochinchinone C.* Yellow needle crystals, m.p. 158-159 °C; $[\alpha]^{25}_{D}$ = +125.1 (*c* 0.14, $CHCl_3$), UV-Vis ($CHCl_3$) λ_{max} (log ε) 262 (3.28), 310 (4.06), 350 (3.82), 400 (3.35) nm; FT-IR (neat) ν_{max} 3428, 1746, 1644, 1604 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.12**. (Mahabussarakam *et al*., 2006).

***21. Compound* CC21**: *Friedelin*



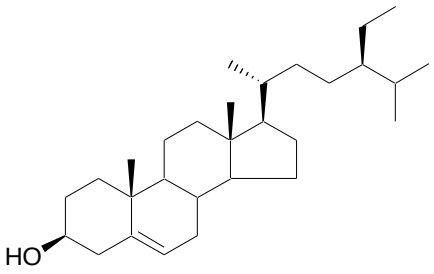
The structure of compound **21**



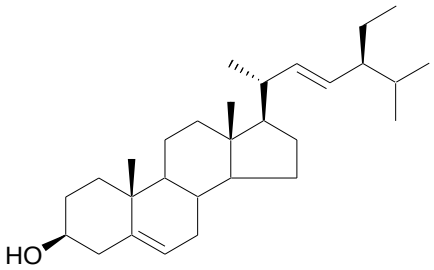
The X-ray structure of compound **21**

***Compound* CC21**: *Friedelin.* White crystal, m.p. 245-247 ^{O}C. FT-IR (neat) ν_{max} 1715 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.21**. (Ahad *et al*., 1991)

***22. Compounds* CC22** and **CC23**



The structure of compound **CC22**



The structure of compound **CC23**

***Compounds* CC22** and **CC23**: The mixture of compounds **CC22** and **CC23** was obtained as a white crystal. The ^{1}H NMR spectral data showed an oxymethine proton at δ 3.57-3.47 (*m*), three olefinic protons at δ 5.36-5.34 (*d*, 5.1 Hz), 5.16 (*dd*, 8.4, 15.1 Hz) and 5.01 (*dd*, 8.4, 15.1 Hz). The ^{1}H NMR data was corresponding to previous reported data, thus, the mixture was assigned as β-sitosterol (**CC22**) and stigmasterol (**CC23**) (Thongdeeying, 2005).

Table 2.1 NMR spectroscopic data of **CC1** in $CDCl_3$

Position	**Type of C**	**δ_H[a] (*J* in Herz)**	**δ_C[b]**	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.97, *s*	155.5	C-1, C-2, C-9a
2	C		104.2	
3	C		158.4	
4	C		107.4	
5	CH	7.31, *d*, 9.0	119.0	C-7, C-9, C-4b, C-8a
6	CH	7.23, *dd*, 9.0, 2.4	124.1	C-5, C-7, C-8, C-4b
7	C		150.5	
8	CH	7.56, *d*, 2.4	108.9	C-6, C-7, C-9, C-4b
9	C=O		180.9	
4a	C		154.5	
4b	C		152.4	
8a	C		120.6	
9a	C		103.3	
1′	CH	6.74, *d*, 9.9	115.8	C-1, C-2, C-3, C-3′, C-4′, C-5′
2′	CH	5.60, *d*, 9.9	127.3	C-2, C-3′, C-4′, C-5′
3′	C		78.1	
4′	CH_3	1.48, *s*	28.4	C-2′, C-3′
5′	CH_3	1.48, *s*	28.4	C-2′, C-3′
1″	CH_2	3.46, *d*, 7.5	21.3	C-3, C-4, C-2″
2″	CH	5.22, *br t*, 7.2	122.1	C-4, C-1″, C-4″, C-9″
3″	C		135.0	
4″	CH_2	1.99, *m*	39.7	C-2″, C-3″, C-5″
5″	CH_2	2.04, *m*	26.6	C-3″, C-4″, C-6″, C-7″
6″	CH	5.04, *br t*, 6.6	124.2	C-4″, C-5″, C-8″, C-10″
7″	C		131.3	
8″	CH_3	1.59, *s*	25.6	C-6″, C-7″, C-10″
9″	CH_3	1.86, *s*	16.3	C-2″, C-3″
10″	CH_3	1.53, *s*	17.6	C-6″, C-7″, C-8″

[a]Recorded in 300 MHz.; [b]Recorded in 75 MHz.

Table 2.2 NMR spectroscopic data of **CC2** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.16, *s*	160.2	C-1, C-2, C-9a
2	C		111.2	
3	C		158.9	
4	C		100.2	
5	CH	7.36, *d*, 8.7	118.9	C-7, C-8a
6	CH	7.25, *m*	123.8	C-7, C-4b
7	C		150.3	
8	CH	7.61, *br d*, 1.8	109.3	C-8
9	C=O		180.5	
4a	C		150.7	
4b	C		152.2	
8a	C		121.1	
9a	C		103.0	
1′	CH_2	3.36, *d*, 7.2	115.8	C-1, C-2, C-2′, C-3′
2′	CH	5.25, *br d*, 7.2	122.1	C-2′
3′	C		131.5	
4′	CH_3	1.68, *s*	25.8	C-2′, C-3′
5′	CH_3	1.81, *s*	17.9	C-2′, C-3′, C-5′
1″	CH	6.88, *d*, 10.2	115.9	C-3, C-4, C-3″, C-4a
2″	CH	5.54, *d*, 10.2	125.4	C-4, C-3″, C-4″, C-9″
3″	C		80.6	
4″	CH_2	1.89 (*m*); 1.68 (*m*)	41.8	C-2″, C-3″
5″	CH_2	2.12, *m*	22.8	C-6″, C-7″
6″	CH	5.10, *br t*, 7.2	123.8	C-6″
7″	C		131.9	
8″	CH_3	1.66, *s*	25.6	C-6″, C-7″, C-10″
9″	CH_3	1.44, *s*	27.1	C-2″, C-3″, C-4″
10″	CH_3	1.57, *s*	17.6	C-6″, C-7″, C-8″

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.3 NMR spectroscopic data of **CC3a** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.02, *s*	158.4	C-1, C-2, C-9a
2	C		104.0	
3	C		159.6	
4	C		107.3	
5	CH	7.46, *d*, 8.7	117.8	C-6, C-7, C-9, C-4b
6	CH	7.34, *dd*, 8.7, 2.7	128.6	C-5, C-7, C-4b
7	C		146.3	
8	CH	7.92, *d*, 2.7	118.7	C-7, C-9, C-4b, C-8a
9	C=O		180.3	
4a	C		152.4	
4b	C		153.7	
8a	C		121.2	
9a	C		102.4	
1′	CH_2	2.75, *t*, 6.9	16.2	C-1, C-2, C-3, C-2′, C-3′
2′	CH_2	1.81, *t*, 6.9	31.7	C-1, C-1′, C-3′, C-4′, C-5′
3′	C		76.3	
4′	CH_3	1.39, *s*	26.9	C-2′, C-3′
5′	CH_3	1.39, *s*	26.9	C-2′, C-3′
1″	CH_2	3.48, *d*, 7.5	21.5	C-3, C-4, C-2″, C-3″, C-4a
2″	CH	5.22, *br t*, 6.3	122.3	C-1″, C-3″, C-4″, C-9″
3″	C		134.9	
4″	CH_2	1.96, *m*	39.8	C-2″, C-3″, C-6″
5″	CH_2	2.03, *m*	26.7	C-3″, C-6″, C-7″
6″	CH	5.05, *br t*, 6.6	124.2	C-5″, C-8″, C-10″
7″	C		131.3	
8″	CH_3	1.60, *s*	25.6	C-6″, C-7″, C-10″
9″	CH_3	1.86, *s*	16.3	C-2″, C-3″
10″	CH_3	1.55, *s*	17.7	C-6″, C-7″, C-8″
7-OAc	CH_3	2.34, *s*	21.0	C-7, C-1‴
	C=O		169.4	

[a]Recorded in 300 MHz.; [b]Recorded in 75 MHz.

Table 2.4 NMR spectroscopic data of **CC4** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.79, *s*	158.2	C-1, C-2, C-9a
2	C		109.2	
3	C		161.1	
4	C		104.9	
5	CH	7.04, *br d*, 9.0	118.7	C-6, C-7, C-9, C-4b
6	CH	7.07, *br d*, 9.0	124.7	C-5, C-7, C-4b
7	C		150.1	
8	CH	7.44, *br s*	108.7	C-7, C-9, C-4b, C-8a
9	C=O		180.9	
4a	C		152.9	
4b	C		152.6	
8a	C		120.2	
9a	C		103.0	
1′	CH_2	3.32, *d*, 6.9	21.8	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.20, *br t*, 6.9	121.6	C-1, C-1′, C-3′, C-4′, C-5′
3′	C		134.8	
4′	CH_3	1.55, *s*	25.6	C-2′, C-3′
5′	CH_3	1.75, *s*	17.9	C-2′, C-3′
1″	CH_2	3.39, *d*, 6.9	21.6	C-3, C-4, C-2″, C-3″, C-4a
2″	CH	5.16, *br t*, 6.9	122.7	C-1″, C-3″, C-4″, C-9″
3″	C		137.9	
4″	CH_2	1.99, *m*	39.7	C-2″, C-3″, C-6″
5″	CH_2	1.97, *m*	26.4	C-3″, C-6″, C-7″
6″	CH	4.96, *br t*, 7.2	123.9	C-5″, C-8″, C-10″
7″	C		131.8	
8″	CH_3	1.67, *s*	25.8	C-6″, C-7″, C-10″
9″	CH_3	1.78, *s*	16.2	C-2″, C-3″
10″	CH_3	1.48, *s*	17.6	C-6″, C-7″, C-8″

[a]Recorded in 300 MHz.; [b]Recorded in 75 MHz.

Table 2.5 NMR spectroscopic data of **CC5** in $CDCl_3$

Position	**Type of C**	**δ_H[a] (*J* in Herz)**	**δ_C[b]**	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.89, *s*	158.2	C-1, C-2, C-3, C-9a
2	C		108.8	
3	C		161.1	
4	C		105.2	
5	CH	7.16, *d*, 8.7	118.8	C-7, C-9, C-4b, C-8a
6	CH	7.13, *br d*, 8.7	124.2	C-7, C-8, C-4b
7	C		150.2	
8	CH	7.55, *br s*	108.9	C-6, C-9, C-4b
9	C=O		180.9	
4a	C		153.0	
4b	C		152.5	
8a	C		120.3	
9a	C		103.1	
1′	CH_2	3.42, *d*, 6.9	21.6	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.28, *br t*, 7.2	121.5	C-2, C-1′, C-4′, C-5′
3′	C		135.2	
4′	CH_3	1.84, *s*	17.9	C-2, C-2′, C-3′
5′	CH_3	1.77, *s*	25.8	C-2, C-2′, C-3′
1″	CH_2	3.47, *d*, 6.9	21.8	C-3, C-4, C-2″, C-3″, C-4a
2″	CH	5.25, *br t*, 7.2	121.8	C-4, C-1″, C-4″, C-9″
3″	C		133.9	
4″	CH_3	1.87, *s*	17.9	C-4, C-2″, C-3″
5″	CH_3	1.74, *s*	25.8	C-4, C-2″, C-3″

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.6 NMR spectroscopic data of **CC6** in $CDCl_3$

Position	**Type of C**	$\delta_H{}^a$ **(*J* in Herz)**	$\delta_C{}^b$	**HMBC ($^1H \rightarrow {}^{13}C$)**
1-OH	C	13.08, *s*	162.1	C-1, C-2, C-3, C-9a
2	CH	6.75, *d*, 8.4	110.6	C-1, C-4, C-4a, C-9a
3	CH	7.52, *t*, 8.4	136.1	C-1, C-4, C-4a
4	CH	6.92, *d*, 8.4	106.2	C-1, C-2, C-4a, C-9a
5	C		135.9	
6	C		145.4	
7	C		147.3	
8	C		128.3	
9	C=O		183.6	
4a	C		155.2	
4b	C		143.4	
8a	C		114.5	
9a	C		109.1	
1′	CH_2	4.04, *d*, 6.6	25.4	C-7, C-8, C-8a, C-2′, C-3′
2′	CH	5.21, *br t*, 6.6	123.5	C-8
3′	C		131.6	
4′	CH_3	1.85, *s*	18.1	C-2′, C-3′
5′	CH_3	1.70, *s*	25.9	C-2′, C-3′
6-OCH_3	CH_3	4.07, *s*	61.1	C-6
7-OCH_3	CH_3	3.82, *s*	61.1	C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.7 NMR spectroscopic data of **CC7** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.35, *s*	159.6	C-1, C-2, C-9a
2	C		111.5	
3	C		163.1	
4	CH	6.36, *s*	88.9	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.82, *s*	102.2	C-6, C-7, C-9, C-4b, C-8a
6	C		150.8	
7	C		137.5	
8	C		120.5	
9	C=O		182.4	
4a	C		155.3	
4b	C		153.3	
8a	C		108.4	
9a	C		104.6	
1′	CH_2	3.35, *d*, 6.9	21.4	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.23, *br t*, 6.9	122.3	-
3′	C		132.0	
4′	CH_3	1.68, *s*	25.8	C-2′, C-3′, C-5′
5′	CH_3	1.80, *s*	17.8	C-2′, C-3′, C-4′
1″	CH	8.04, *d*, 10.2	121.0	C-7, C-8, C-8a, C-3″
2″	CH	5.82, *d*, 10.2	132.2	C-8, C-3″, C-4″, C-5″
3″	C		77.2	
4″	CH_3	1.50, *s*	27.4	C-2′, C-3′
5″	CH_3	1.50, *s*	27.4	C-2′, C-3′
3-OCH_3	CH_3	3.91, *s*	61.1	C-3

[a]Recorded in 300 MHz.

[b]Recorded in 125 MHz.

Table 2.8 NMR spectroscopic data of **CC8** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.41, *s*	159.8	C-1, C-2, C-9a
2	C		111.5	
3	C		163.5	
4	CH	6.32, *s*	88.8	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.82, *s*	101.5	C-6, C-7, C-9, C-4b, C-8a
6	C		154.4	
7	C		142.6	
8	C		137.0	
9	C=O		181.9	
4a	C		155.7	
4b	C		155.2	
8a	C		112.4	
9a	C		103.8	
1′	CH_2	3.35, *d*, 7.2	21.4	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.23, *m*	122.4	C-1′, C-3′
3′	C		131.7	
4′	CH_3	1.68, *s*	25.8	C-2′, C-3′
5′	CH_3	1.80, *s*	17.8	C-2′, C-3′
1″	CH	4.09, *d*, 7.2	26.6	C-7, C-8, C-8a, C-2″, C-3″
2″	CH_2	5.26, *m*	123.2	C-1″, C-3″
3″	C		132.0	
4″	CH_3	1.68, *s*	25.8	C-2″, C-3″
5″	CH_3	1.83, *s*	18.2	C-2″, C-3″
3-OCH_3	CH_3	3.90, *s*	62.0	C-3
7-OCH_3	CH_3	3.81, *s*	55.8	C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.9 NMR spectroscopic data of **CC9** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.77, *s*	160.2	C-1, C-2, C-9a
2	C		109.9	
3	C		161.7	
4	CH	6.29, *s*	92.5	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.82, *s*	101.7	C-6, C-7, C-9, C-4b, C-8a
6	C		155.4	
7	C		142.9	
8	C		137.3	
9	C=O		181.9	
4a	C		154.8	
4b	C		155.5	
8a	C		111.7	
9a	C		103.7	
1′	CH_2	3.45, *d*, 7.2	21.3	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.29, *m*	122.3	-
3′	C		132.3	
4′	CH_3	1.77, *s*	25.7	C-2′, C-3′
5′	CH_3	1.84, *s*	17.7	C-2′, C-3′
1″	CH_2	4.09, *d*, 6.0	26.3	C-7, C-8, C-8a, C-2″, C-3″
2″	CH	5.26, *m*	123.5	-
3″	C		131.6	
4″	CH_3	1.69, *s*	25.7	C-2″, C-3″
5″	CH_3	1.84, *s*	18.0	C-2″, C-3″
7-OCH_3	CH_3	3.81, *s*	61.2	C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.10 NMR spectroscopic data of **CC10** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.53, *s*	156.8	C-1, C-2, C-9a
2	C		105.6	
3	C		158.9	
4	C		113.1	
5	C		131.1	
6	C		149.0	
7	CH	6.94, *d*, 9.0	112.8	C-5, C-6, C-8a
8	CH	7.68, *d*, 9.0	117.5	C-5, C-6, C-9, C-4b
9	C=O		180.8	
4a	C		154.1	
4b	C		144.5	
8a	C		113.7	
9a	C		103.8	
1′	CH	6.76, *d*, 9.9	116.1	C-1, C-2, C-3, C-9a, C-3′
2′	CH	5.61, *d*, 9.9	127.2	C-2, C-3′, C-4′, C-5′
3′	C		78.3	
4′	CH_3	1.52, *s*	27.9	C-2′, C-3′
5′	CH_3	1.52, *s*	27.9	C-2′, C-3′
1″	C		41.4	
2″	CH	6.76, *dd*, 17.7, 10.5	156.8	C-3, C-1″, C-3″, C-4″, C-5″
3″	CH_2	5.22, *dd*, 17.7, 1.5	103.3	C-1″, C-2″
		5.05, *dd*, 10.5, 1.5		C-1″
4″	CH_3	1.65, *s*	28.2	C-4, C-1″, C-2″
5″	CH_3	1.65, *s*	28.2	C-4, C-1″, C-2″
5-OH	-	6.27, *brs*	-	C-5, C-6, C-4b

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.11 NMR spectroscopic data of **CC11** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC (^{1}H→^{13}C)**
1-OH	C	13.53, *s*	156.8	C-1, C-2, C-9, C-9a
2	C		105.2	
3	C		159.2	
4	C		112.8	
5	C		131.0	
6	C		149.0	
7	CH	6.94, *d*, 7.8	112.8	-
8	CH	7.67, *d*, 8.4	117.5	C-6, C-9
9	C=O		180.7	
4a	C		154.1	
4b	C		144.5	
8a	C		113.7	
9a	C		102.9	
1′	CH	6.81, *d*, 9.9	116.7	C-1, C-2, C-3, C-3′
2′	CH	5.57, *d*, 9.9	125.6	C-2, C-3′, C-4′, C-9′
3′	C		81.1	
4′	CH_2	1.90, *m*	41.8	C-2′, C-4′, C-5′, C-6′
		1.71, *m*		-
5′	CH_2	2.13, *m*	23.2	C-6′, C-7′
6′	CH	5.12, *brt*, 6.9	123.7	C-5′, C-8′, C-10′
7′	C		132.1	
8′	CH_3	1.68, *s*	25.7	C-6′, C-7′
9′	CH_3	1.46, *s*	26.9	C-2′, C-3′, C-4′
10′	CH_3	1.59, *s*	17.6	C-6′, C-7′
1′′	C		41.4	
2′′	CH	6.73, *dd*, 17.7, 10.8	156.7	C-4, C-1′′, C-4′′, C-5′′
3′′	CH_2	5.21, *dd*, 17.4, 1.2	103.3	C-1′′, C-2′′
		5.04, *dd*, 10.5, 1.2		C-1′′
4′′	CH_3	1.65, *s*	28.0	C-4, C-1′′, C-2′′
5′′	CH_3	1.65, *s*	28.4	C-4, C-1′′, C-2′′

[a]Recorded in 300 MHz.

[b]Recorded in 125 MHz.

Table 2.12 NMR spectroscopic data of **CC12** in $CDCl_3$

Position	**Type of C**	**δ_H[a] (*J* in Herz)**	**δ_C[b]**	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.10, *s*	162.9	C-1, C-2, C-9a
2	CH	6.55, *dd*, 8.4, 0.9	109.5	C-1, C-4, C-9a
3	CH	7.41, *t*, 8.4	138.9	C-1, C-4a
4	CH	6.52, *dd*, 8.1, 0.9	107.4	C-2, C-9, C-4a, C-9a
5	C		84.2	
6	C=O		201.1	
7	C		84.9	
8	CH	7.51, *d*, 1.2	135.3	C-6, C-7, C-9, C-11, C-4b, C-8a
9	C=O		180.7	
4a	C		159.4	
4b	C		88.8	
8a	C		132.1	
9a	C		106.1	
10	CH_2	2.39, *br d*, 13.2	29.7	C-6, C-7, C-8, C-11, C-4b
		1.59, *dd*, 13.2, 9.9		C-6, C-7, C-8
11	CH	2.53, *d*, 9.6	49.4	C-7, C-4b
12	C		83.9	
13	CH_3	1.68, *s*	30.4	C-11, C-12, C-14
14	CH_3	1.33, *s*	29.0	C-11, C-12, C-13
1′	CH_2	2.64, *d*, 8.4	29.2	C-5, C-6, C-4b, C-16, C-17
2′	CH	4.41, *br t*, 7.8	118.4	C-15, C-18, C-19
3′	C		135.7	
4′	CH_3	1.37, *s*	25.5	C-16, C-17, C-19
5′	CH_3	1.01, *s*	16.7	C-16, C-17, C-18
7-OCH_3	CH_3	3.65, *s*	54.1	C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.21 NMR spectroscopic data of **CC21** in $CDCl_3$

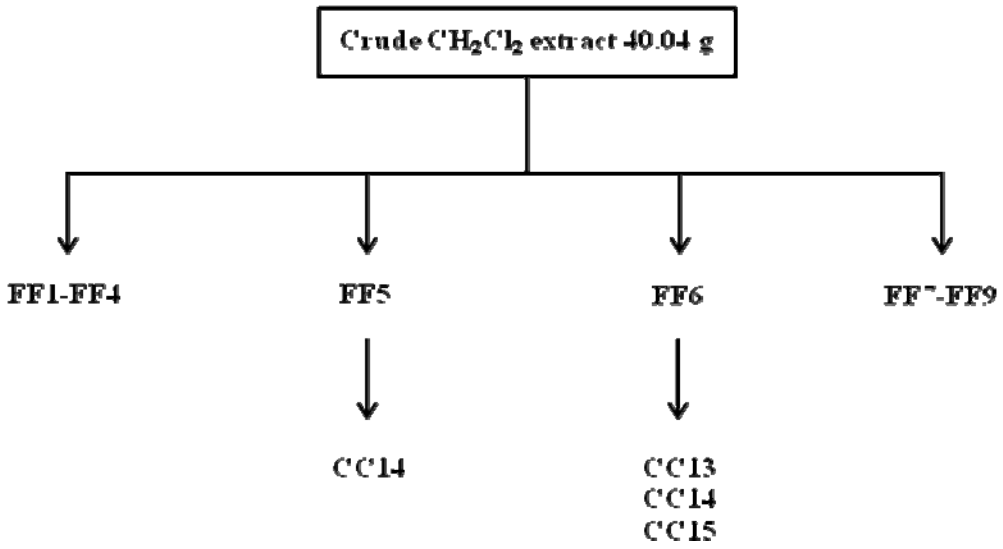
Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1	CH_2	1.64, *m*, 1.69, *m*	22.3	-
2	CH_2	2.36, *m*, 2.23, *m*	41.5	-
3	C	-	213.3	-
4	CH	2.24, *m*	58.2	
5	C	-	42.2	-
6	CH_2	2.44, *m*, 1.78, *m*	41.3	-
7	CH_2	1.52, *m*, 1.39, *m*	18.2	-
8	CH	1.42, *m*	53.1	-
9	C	-	37.4	-
10	CH_2	1.61, *m*, 1.43, *m*	35.6	-
11	CH_2	1.46, *m*, 1.34, *m*	30.5	-
12	C	-	39.7	-
13	C	-	38.3	-
14	CH_2	1.51, *m*, 1.29, *m*	32.4	-
15	CH_2	1.61, *m*, 1.36, *m*	36.0	-
16	C	-	30.0	-
17	CH	1.53, *m*	42.8	-
18	CH_2	1.64, *m*, 1.69, *m*	22.3	-
19	CH_2	1.62, *m*, 1.49, *m*	35.3	-
20	C	-	28.2	-
21	CH_2	1.48, *m*, 0.93, *m*	39.3	-
22	CH_2	1.50, *m*, 1.26, *m*	32.8	-
23	CH_3	0.89, *d*, 6.3 Hz	6.8	C-3, C-4, C-5
24	CH_3	0.72, *s*	14.7	C-4, C-5, C-6, C-10
25	CH_3	0.87, *s*	17.9	C-8, C-9, C-10, C-11
26	CH_3	1.01, *s*	20.3	C-8, C-13, C-14, C-15
27	CH_3	1.05, *s*	18.5	C-12, C-13, C-14, C-18
28	CH_3	1.18, *s*	32.1	C-16, C-17, C-18, C-22
29	CH_3	1.00, *s*	31.8	C-19, C- 20, C-21
30	CH_3	0.95, *s*	35.0	C-19, C-20, C-21

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

2.4.2 The CH_2Cl_2 extract of the green fruits of *Cratoxylum cochinchinense*

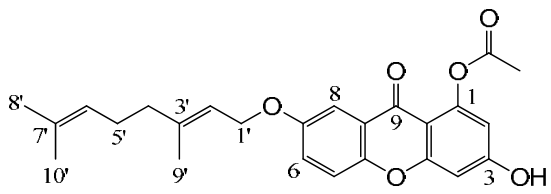
The crude CH_2Cl_2 extract (40.04 g) of the green fruits of *C. cochinchinense* was subjected to QCC on silica gel using hexane as a first eluent and increasing polarity with EtOAc to give 9 fractions (FF1-FF9). Fraction FS5 was purified by CC eluting with pure $CHCl_3$ to give **CC14** (1.88 g). Fraction FF6 was further separated by CC eluting with pure $CHCl_3$ to furnish 6 subfractions (FF6A-FF6F), **CC14** (2.10 g) and **CC15** (490.2 mg)., respectively. Subfraction FF6B was further purified by CC eluting with a gradient of acetone-hexane to give **CC13** (53.3 mg) (see **Scheme 4**).



Scheme 4 Isolation of compounds **CC13-CC15**

Characterization of isolated compounds

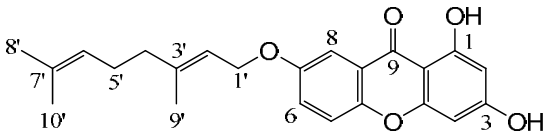
***13. Compound* CC13**: *Cochinchinone L*



The structure of compound **13**

***Compound* CC13**: *Cochinchinone L*. Yellow powder, m.p. 114-116 $^{\circ}$C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 248 (4.59), 273 (4.03), 305 (4.13), 354 (3.80) nm; FT-IR (neat) ν_{max} 3237, 1774, 1728, 1628 cm^{-1}; HRMS *m/z* 422.1718 for $C_{25}H_{26}O_6$ (calcd. 422.1729). EIMS *m/z* (rel. int.): 422 $[M]^+$ (1), 286 (40), 244 (100), 187 (4), 81 (9), 69 (28). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.13**. (Boonnak *et al*., 2009)

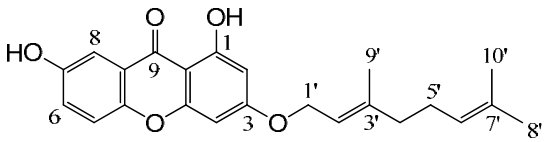
***14. Compound* CC14**



The structure of compound **14**

***Compound* CC14**: *7-geranyloxy-1,3-dihydroxyxanthone.* Yellow powder, m.p. 138-140 $^{\circ}$C; UV-Vis λ_{max} (log ε) 206 (4.25), 236 (4.48), 260 (4.39), 316 (3.89), 364 (3.94) nm; FT-IR (KBr) ν_{max} 3162, 1652 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.14**. (Nguyen and Harrison, 1998)

***15. Compound* CC15**: *Cochinchinone G*



The structure of compound **15**

***Compound* CC15**: *Cochinchinone G*. Yellow powder, m.p. 147-148 $^{\circ}$C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 203 (4.49), 229 (4.30), 259 (4.31), 307 (3.99), 374 (3.63) nm; FT-IR (KBr) ν_{max} 3288, 1647 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.15**. (Mahabusarakam *et al*., 2008)

Table 2.13 NMR spectroscopic data for **CC13** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1	C		151.2	
2	CH	6.43, *d*, 2.1	108.3	C-1, C-3, C-4, C-9, C9a
3	C		162.6	
4	CH	6.57, *d*, 2.1	101.3	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.08, *br d*, 9.0	118.8	C-7, C-9, C-8a
6	CH	7.10, *br d*, 9.3	125.1	C-5, C-8, C-4b
7	C		155.4	
8	CH	7.45, *br s*	106.4	C-6, C-7, C-9, C-4b, C-8a
9	C=O		175.7	
4a	C		158.8	
4b	C		150.0	
8a	C		121.9	
9a	C		107.8	
1′	CH_2	4.42, *d*, 6.3	65.5	C-7, C-2′, C-3′, C-4′, C-5′, C-9′
2′	CH	5.73, *br t*, 6.3	118.8	C-1′, C-4′, C-9′
3′	C		141.9	
4′	CH_2	1.99, *m*	39.5	C-2′, C-3′, C-6′
5′	CH_2	1.98, *m*	26.3	C-3′, C-6′, C-7′
6′	CH	4.98, *br t*, 6.6	123.8	C-4′, C-5′, C-8′, C-10′
7′	C		131.8	
8′	CH_3	1.60, *s*	25.7	C-6′, C-7′, C-10′
9′	CH_3	1.57, *s*	16.7	C-2′, C-3′, C-4′
10′	CH_3	1.50, *s*	17.7	C-6′, C-7′, C-8′
1-OAc	CH_3	2.38, *s*	21.3	C-1, C-1′′
	C=O		170.9	

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.14 NMR spectroscopic data for **CC14** in $CDCl_3$

Position	**Type of C**	**δ_H[a] (*J* in Herz)**	**δ_C[b]**	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.85, *s*	163.8	C-1, C-2, C-9, C-9a
2	CH	6.22, *d*, 1.8	98.5	C-1, C-3, C-4
3	C		164.0	
4	CH	6.27, *d*, 1.8	94.4	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.14, *br d*, 9.0	118.9	C-6, C-7, C-9, C-4b
6	CH	7.18, *br d*, 9.6	125.6	C-7, C-8, C-4b
7	C		155.2	
8	CH	7.40, *br d*, 1.5	105.9	C-6, C-7, C-9, C-4b, C-8a
9	C=O		180.5	
4a	C		157.8	
4b	C		150.7	
8a	C		120.4	
9a	C		103.3	
1′	CH_2	4.45, *d*, 6.3	65.6	C-7, C-2′, C-3′, C-4′, C-5′, C-9′
2′	CH	5.39, *br t*, 6.3	118.6	C-1′, C-4′, C-5′, C-9′
3′	C		141.2	
4′	CH_2	2.01, *m*	39.5	C-2′, C-3′, C-6′
5′	CH_2	1.95, *m*	26.3	C-3′, C-6′, C-7′
6′	CH	4.99, *br t*, 5.7	123.8	C-4′, C-5′, C-8′, C-10′
7′	C		131.8	
8′	CH_3	1.57, *s*	26.3	C-6′, C-7′, C-10′
9′	CH_3	1.64, *s*	16.4	C-2′, C-3′, C-4′
10′	CH_3	1.50, *s*	17.7	C-6′, C-7′, C-8′
3-OH		7.86, *br s*		-

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

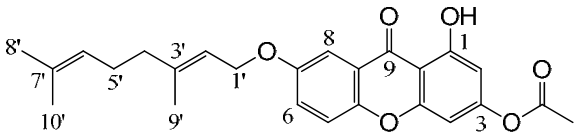
Table 2.15 NMR spectroscopic data of **CC15** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.73, *s*	163.2	C-1, C-2, C-9a
2	CH	6.34, *d*, 2.4	97.6	C-1, C-3, C-4, C-9a
3	C		166.1	
4	CH	6.40, *d*, 2.4	93.2	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.30, *br d*, 9.3	119.0	C-6, C-7, C-9, C-4b, C-8a
6	CH	7.26, *br d*, 9.3	124.2	C-5, C-7, C-4b
7	C		152.5	
8	CH	7.40, *br s*	109.0	C-6, C-7, C-9, C-4b
9	C=O		180.6	
4a	C		157.8	
4b	C		150.5	
8a	C		120.9	
9a	C		103.5	
1′	CH_2	4.63, *d*, 6.6	65.6	C-3, C-2′, C-3′
2′	CH	5.50, *br t*, 6.6	118.4	C-4′, C-9′
3′	C		142.4	
4′	CH_2	2.14, *m*	39.5	C-2′, C-3′, C-6′
5′	CH_2	2.10, *m*	26.2	C-3′, C-6′, C-7′
6′	CH	5.11, *br t*, 5.7	123.6	-
7′	C		131.9	
8′	CH_3	1.69, *s*	25.6	C-6′, C-7′, C-10′
9′	CH_3	1.78, *s*	16.8	C-2′, C-3′, C-4′
10′	CH_3	1.63, *s*	17.7	C-6′, C-7′, C-8′
7-OH		7.03, *br s*		-

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

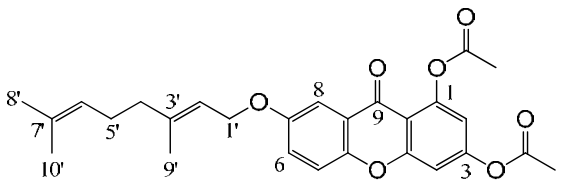
***16. Compound* CC16**



The structure of compound **16**

***Compound* CC16**: *Mono-acetylation of **CC14**.* Compound **CC14** (82.5 mg) was treated with Ac_2O (2.5 mL) in pyridine (2.0 mL) and stirred for 6 hr at room temperature. The reaction mixture was diluted with water, extracted with CH_2Cl_2. The combined organic extract was washed with 10% HCl and then washed with water again. After the organic solvent was removed, the resulting residue was dried over anhydrous Na_2SO_4. Chromatography over silica gel yielded a pale yellow powder of **16** (80.6 mg). ***Compound* CC16**: *3-Acetoxy-7-geranyloxy-1-hydroxyxanthone*. Yellow powder, m.p. 94-95 $^{\circ}$C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 239 (4.40), 262 (4.66), 289 (3.97), 379 (3.93) nm; FT-IR (neat) ν_{max} 3429, 1768, 1649, 1612 cm^{-1}; HRMS *m/z* 422.1725 for $C_{25}H_{26}O_6$ (calcd. 422.1729). EIMS *m/z* (rel. int.): 422 $[M]^+$ (1), 286 (43), 244 (100), 187 (4), 81 (9), 69 (24). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.16**. (Boonnak *et al*., 2009)

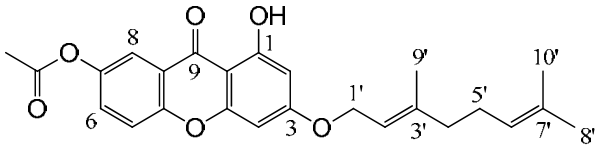
***17. Compound* CC17**



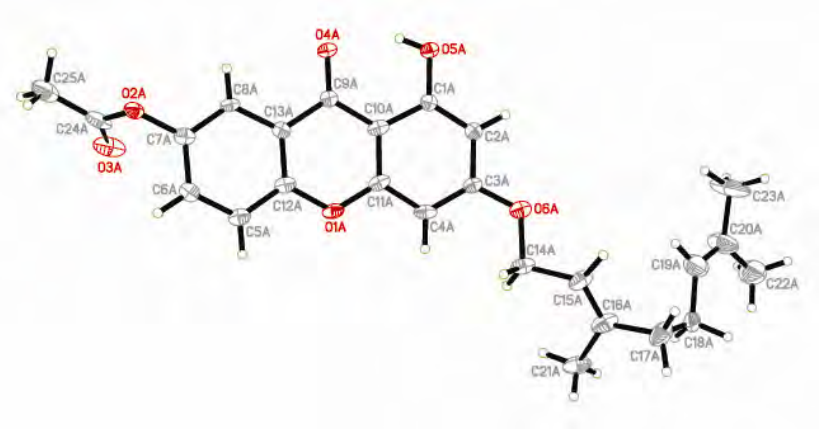
The structure of compound **17**

Compound* CC17**: *Di-acetylation of* ***CC14*.* Compound **CC14** (200.5 mg) was treated with Ac_2O (6.0 mL) in pyridine (3.0 mL) and stirred overnight at room temperature. Chromatography over silica gel yielded a pale yellow powder of **CC16** (10.6 mg) and **CC17** (177.8 mg), respectively. ***Compound* CC17**: *1,3-Diacetoxy-7-geranyloxyxanthone.* Yellow powder, m.p. 96-97 ^{o}C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 253 (4.59), 300 (3.45), 361 (3.86) nm; FT-IR (neat) ν_{max} 3429, 1776, 1656, 1624 cm^{-1}; HRMS *m/z* 464.1838 for $C_{27}H_{28}O_7$ (calcd. 464.1835). EIMS *m/z* (rel. int.): 464 $[M]^+$ (2), 328 (3), 286 (58), 244 (100), 187 (5), 81 (17), 69 (36). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.17**. (Boonnak *et al*., 2009)

18. Compound CC18



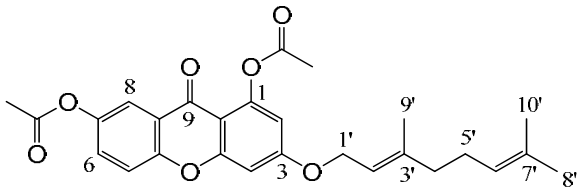
The structure of compound **18**



The X-ray structure of compound **18**

***Compound* CC18**: *Mono-acetylation of **CC15**.* Compound **CC15** (85.5 mg) was treated with Ac_2O (2.5 mL) in pyridine (2.0 mL) and stirred for 6 hr at room temperature. Chromatography over silica gel yielded a pale yellow powder of **CC18** (83.7 mg). ***Compound* CC18**: *7-Acetoxy-3-geranyloxy-1-hydroxyxanthone.* Yellow powder, m.p. 104-106 $^{\circ}$C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 243 (4.44), 257 (4.52), 310 (4.29), 358 (3.82) nm; FT-IR (neat) ν_{max} 3429, 1758, 1665, 1607 cm^{-1}; HRMS *m/z* 422.1726 for $C_{25}H_{26}O_6$ (calcd. 422.1729). EIMS *m/z* (rel. int.): 422 $[M]^+$ (5), 286 (16), 244 (100), 187 (2), 81 (16), 69 (56). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.18**. (Boonnak *et al*., 2009)

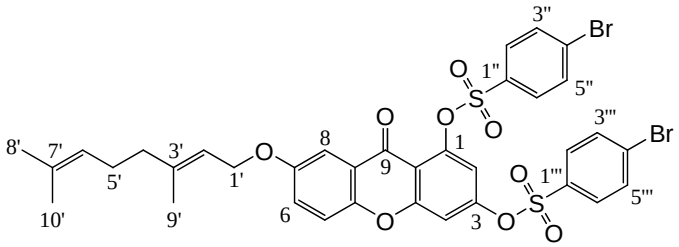
19. Compound CC19



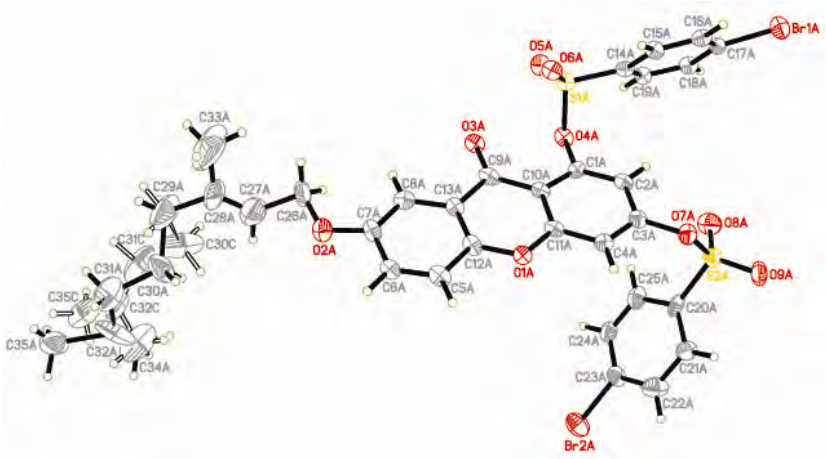
The structure of compound CC**19**

***Compound* CC19**: *Di-acetylation of **CC15**.* Compound **CC15** (190.0 mg) was treated with Ac_2O (6.0 mL) in pyridine (3.0 mL) and stirred overnight at room temperature. Chromatography over silica gel yielded a pale yellow powder of **CC18** (8.7 mg) and **CC19** (170.0 mg), respectively. ***Compound* CC19**: *1,7-Diacetoxy-3-geranyloxyxanthone.* Yellow powder, m.p. 85-87 $^{\circ}$C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 246 (4.61), 275 (4.01), 302 (4.28), 334 (3.86) nm; FT-IR (neat) ν_{max} 3453, 1770, 1655, 1629 cm^{-1}; HRMS *m/z* 464.1834 for $C_{27}H_{28}O_7$ (calcd. 464.1835). EIMS *m/z* (rel. int.): 464 $[M]^+$ (4), 328 (4), 286 (32), 244 (100), 187 (2), 81 (24), 69(73). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 2.19**. (Boonnak *et al*., 2009)

***20. Compound* CC20**



The structure of compound **20**



The X-ray structure of compound **20**

Compound **CC20** was isolated as yellow needle crystal, m.p. 106-108 $^{\circ}$C. The ^{1}H and ^{13}C NMR data of **CC20** (**Table 2.20**) were similar to those of **CC14**, except for the presence of two aromatic signals of the *p*-bromobenzenesulfonyl groups at δ 7.81 (*dd*, J = 9.0, 2.1 Hz, H-2$''$ and H-6$''$), 7.65 (*dd*, J = 8.7, 2.1 Hz, H-3$''$, H-5$''$, H-2$'''$ and H-6$'''$) and 7.63 (*dd*, J = 8.7, 2.1 Hz, H-3$'''$ and H-5$'''$) instead of chelated- and hydroxyl groups at C-1 and C-3 as in **CC14**. Compound **CC20** was assigned as 7-geranyloxy-1,3-dibrosylatedxyxanthone (Boonnak *et al*., 2009).

Table 2.16 NMR spectroscopic data of **CC16** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.71, *s*	162.9	C-1, C-2, C-3, C-9, C-4a, C-9a
2	CH	6.42, *br d*, 2.1	104.0	C-1, C-3, C-4, C-9a
3	C		156.8	
4	CH	6.59, *br d*, 2.1	100.7	C-3, C-9, C-4a, C-9a
5	CH	7.21, *br d*, 8.1	119.0	C-7, C-9, C-4b, C-8a
6	CH	7.20, *br d*, 8.1	126.0	C-7, C-8, C-4b
7	C		155.5	
8	CH	7.44, *br s*	106.0	C-6, C-7, C-9, C-4b
9	C=O		181.2	
4a	C		156.7	
4b	C		150.8	
8a	C		120.7	
9a	C		106.6	
1′	CH_2	4.51, *d*, 6.6	65.6	C-7, C-2′, C-3′, C-9′
2′	CH	5.40, *br t*, 6.6	118.8	C-1′, C-3′, C-4′, C-9′
3′	C		141.9	
4′	CH_2	2.04, *m*	39.6	C-2′, C-3′
5′	CH_2	2.02, *m*	26.3	C-4′, C-6′, C-7′
6′	CH	5.00, *br t*, 5.7	123.7	C-4′, C-5′, C-10′
7′	C		131.8	
8′	CH_3	1.58, *s*	25.6	C-6′, C-7′, C-10′
9′	CH_3	1.68, *s*	16.7	C-2′, C-3′, C-4′
10′	CH_3	1.51, *s*	17.7	C-6′, C-7′, C-8′
3-OAc	CH_3	2.23, *s*	21.2	C-3
	C=O		168.2	

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.17 NMR spectroscopic data of **CC17** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1	C		150.9	
2	CH	7.16, *d*, 2.1	108.7	C-1, C-3, C-4, C-4a
3	C		154.5	
4	CH	6.80, *d*, 2.1	112.4	C-1, C-2, C-3, C-4a, C-9a
5	CH	7.23, *br d*, 9.3	118.9	C-7, C-9, C-4b, C-8a
6	CH	7.28, *dd*, 9.3, 2.4	125.3	C-7, C-8, C-4b
7	C		155.5	
8	CH	7.44, *br d*, 2.1	106.6	C-6, C-7, C-9, C-4b, C-8a
9	C=O		174.7	
4a	C		157.7	
4b	C		149.9	
8a	C		122.3	
9a	C		112.2	
1′	CH_2	4.56, *d*, 6.3	65.5	C-7, C-2′, C-3′, C-9′
2′	CH	5.49, *br t*, 6.3	118.8	C-1′, C-3′, C-4′
3′	C		141.6	
4′	CH_2	2.11, *m*	39.5	C-2′, C-3′, C-5′, C-9′
5′	CH_2	2.09, *m*	26.3	C-3′, C-6′, C-7′
6′	CH	5.09, *br t*, 6.3	123.8	C-4′, C-5′, C-8′, C-10′
7′	C		131.7	
8′	CH_3	1.66, *s*	25.6	C-6′, C-7′, C-10′
9′	CH_3	1.74, *s*	16.7	C-2′, C-3′, C-4′
10′	CH_3	1.60, *s*	17.7	C-6′, C-7′, C-8′
1-OAc	CH_3	2.47, *s*	21.1	C-1
	C=O		169.2	
3-OAc	CH_3	2.27, *s*	21.0	C-3
	C=O		167.8	

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.18 NMR spectroscopic data of **CC18** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.55, *s*	163.3	C-1, C-2, C-9, C-9a
2	CH	6.19, *br d*, 2.1	97.8	C-3, C-4, C-9a
3	C		166.2	
4	CH	6.24, *br d*, 2.1	93.4	C-2, C-3, C-4a, C-9a
5	CH	7.25, *d*, 9.3	118.7	C-6, C-7, C-9, C-4b, C-8a
6	CH	7.28, *br d*, 9.0	128.9	C-7, C-8, C-4b
7	C		146.5	
8	CH	7.76, *br s*	117.8	C-6, C-7, C-9, C-4b, C-8a
9	C=O		179.8	
4a	C		157.5	
4b	C		153.4	
8a	C		121.1	
9a	C		103.4	
1′	CH_2	4.49, *d*, 6.3	65.6	C-3, C-2′, C-3′, C-5′
2′	CH	5.37, *br t*, 6.3	118.4	C-1′, C-3′, C-4′, C-9′
3′	C		142.2	
4′	CH_2	2.03, *m*	39.5	C-2′, C-3′, C-5′, C-6′
5′	CH_2	1.97, *m*	26.2	C-3′, C-6′, C-7′
6′	CH	4.99, *br t*, 6.9	123.9	C-4′, C-5′, C-8′, C-10′
7′	C		131.9	
8′	CH_3	1.58, *s*	25.6	C-6′, C-7′, C-10′
9′	CH_3	1.66, *s*	16.7	C-2′, C-3′, C-4′
10′	CH_3	1.51, *s*	17.7	C-6′, C-7′, C-8′
7-OAc	CH_3	2.22, *s*	20.9	C-7
	C=O		169.2	

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 2.19 NMR spectroscopic data of **CC19** in $CDCl_3$

Position	**Type of C**	δ_H[a] **(*J* in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1	C		158.7	
2	CH	6.64, *d*, 2.1	99.5	C-1, C-3, C-4, C-9, C-9a
3	C		163.6	
4	CH	6.47, *d*, 2.1	108.1	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.24, *d*, 8.1	118.6	C-6, C-7, C-9, C-4b, C-8a
6	CH	7.27, *br d*, 8.1	128.3	C-7, C-8, C-4b
7	C		146.2	
8	CH	7.76, *br d*, 1.5	118.4	C-6, C-7, C-9, C-4b, C-8a
9	C=O		174.1	
4a	C		151.4	
4b	C		152.7	
8a	C		123.6	
9a	C		108.6	
1′	CH_2	4.51, *d*, 6.3	65.8	C-3, C-2′, C-3′, C-4′, C-5′
2′	CH	5.37, *br t*, 6.3	118.0	C-1′, C-4′, C-9′
3′	C		142.6	
4′	CH_2	2.03, *m*	39.5	C-2′, C-3′, C-6′
5′	CH_2	2.01, *m*	26.2	C-3′, C-6′, C-7′
6′	CH	4.99, *br t*, 6.6	123.6	C-4′, C-8′, C-10′
7′	C		131.9	
8′	CH_3	1.57, *s*	25.7	C-6′, C-7′, C-10′
9′	CH_3	1.65, *s*	16.7	C-2′, C-3′, C-4′
10′	CH_3	1.51, *s*	17.7	C-6′, C-7′, C-8′
1-OAc	CH_3	2.40, *s*	21.2	C-1
	C=O		169.5	
7-OAc	CH_3	2.19, *s*	20.9	C-7
	C=O		169.2	

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

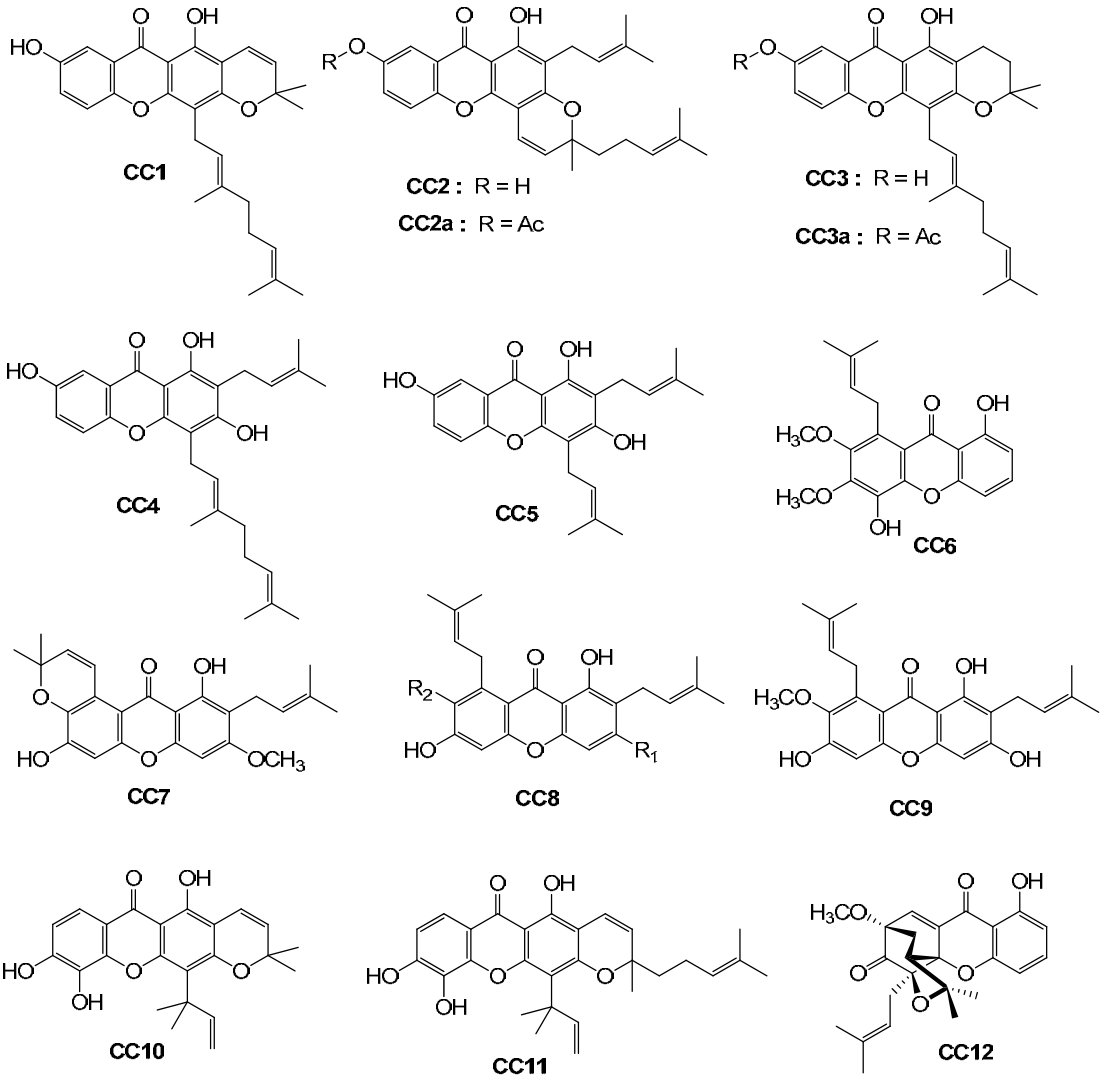
Table 2.20 NMR spectroscopic data of **CC20** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1	C		157.4	
2	CH	7.32, *d*, 2.4	111.1	C-1, C-3, C-4, C-4a
3	C		152.0	
4	CH	7.68, *d*, 2.4	112.8	C-2, C-3, C-4a, C-9a
5	CH	7.26, *d*, 9.0	118.8	C-4b, C-8a
6	CH	7.19, *dd*, 9.6, 2.4	124.9	-
7	C		155.9	
8	CH	7.50, *dd*, 2.4	106.8	C-7, C-4b, C-8a
9	C=O		181.6	
4a	C		148.6	
4b	C		149.7	
8a	C		122.4	
9a	C		114.2	
1′	CH_2	4.56, *d*, 6.6	65.6	C-7, C-2′, C-3′
2′	CH	5.44, *br t*, 6.6	118.6	C-4′, C-9′
3′	C		142.1	
4′	CH_2	2.05, *m*	39.5	C-2′, C-5′, C-6′
5′	CH_2	2.07, *m*	26.2	C-3′, C-4′, C-7′
6′	CH	5.04, *br t*, 6.3	123.7	-
7′	C		131.8	
8′	CH_3	1.60, *s*	25.5	C-6′, C-7′, C-10′
9′	CH_3	1.71, *s*	16.6	C-2′, C-3′, C-4′
10′	CH_3	1.54, *s*	17.5	C-6′, C-7′, C-8′
1″	C		134.3	
2″/6″	CH	7.81, *dd*, 9.0, 2.1	130.3	C-1″, C-4″
3″/5″	CH	7.65, *dd*, 8.7, 2.1	133.0	C-1″, C-4″
4″	C		130.6	
1‴	C		133.4	
2‴/6‴	CH	7.65, *dd*, 8.7, 2.1	129.8	C-1‴, C-4‴
3‴/5‴	CH	7.63, *dd*, 8.7, 2.1	132.5	C-1‴, C-4‴
4‴	C		130.1	

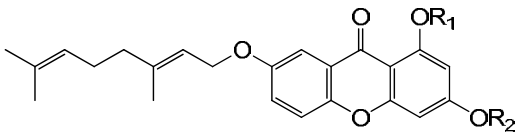
[a]Recorded in 300 MHz.; [b]Recorded in 75 MHz.

2.5 การทดสอบการออกฤทธิ์ต้านเชื้อแบคทีเรียและเชื้อราของสาร CC1-CC20

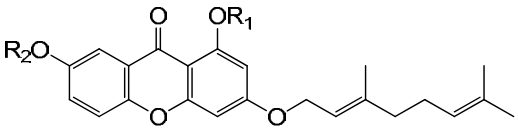
The isolated compounds (**CC1-CC20**) were evaluated for their antibacterial activities against both Gram-positive bacteria: *Bacillus subtilis*, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR 459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC 43300, Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299 and Gram-negative bacteria: *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*. All compounds were also submitted to antifungal assay against *Candida albicans.*



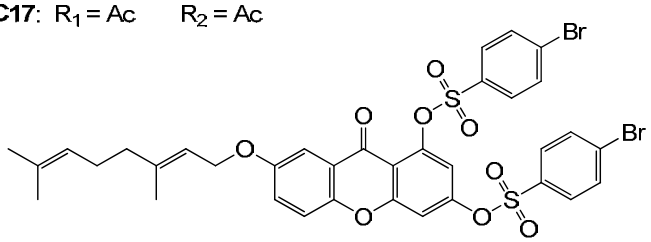
The structures of **CC1-CC12**



CC13: R_1 = Ac R_2 = H
CC14: R_1 = H R_2 = H
CC16: R_1 = H R_2 = Ac
CC17: R_1 = Ac R_2 = Ac



CC15: R_1 = H R_2 = H
CC18: R_1 = H R_2 = Ac
CC19: R_1 = Ac R_2 = Ac



CC20

The structures of **CC13-CC20**

ผลการทดสอบการออกฤทธิ์ต้านเชื้อแบคทีเรียและเชื้อราของสาร **CC1-CC20** แสดงดังตารางที่ 3

Table 3 Antimicrobial activity of compounds **CC1-CC20**

No	Antibacterial activity								Antifungal activity
	Gram-positive bacteria[a]					Gram-positive bacteria[b]			*C. albicans*[c]
	BS	*SA*	*EF*	*MRSA*	*VRE*	*ST*	*SS*	*PA*	
CC1	>300	>300	>300	>300	>300	>300	>300	>300	>300
CC2	300	300	>300	300	300	>300	>300	300	75
CC3	75	>300	300	>300	>300	>300	>300	>300	300
CC4	150	150	150	9.37	150	>150	>150	4.67	75
CC5	150	75	150	37.5	75	>150	>150	4.67	37.5
CC4: CC5[e]	75	150	75	9.37	150	>150	>150	4.67	75
CC6	>150	>150	>150	150	150	>150	>150	4.67	150
CC7	150	300	300	150	150	>300	>300	150	300
CC8	300	75	150	75	150	>300	>300	300	150
CC9	9.37	9.37	9.37	9.37	9.37	>300	>300	18.7	2.34
CC10	18.7	37.5	37.5	37.5	37.5	>300	>300	37.5	4.67
CC11	>300	300	>300	150	150	>300	>300	>300	300
CC12	75	>300	300	150	150	>300	>300	>300	>300
CC13	150	>150	150	37.5	150	>150	>150	4.67	18.7
CC14	150	>150	150	18.7	150	>150	>150	4.67	75
CC15	150	150	150	9.37	150	>150	>150	4.67	37.5
CC14: CC15[e]	75	37.5	37.5	4.67	37.5	>150	>150	4.67	37.5
CC16	150	>150	150	18.7	150	>150	>150	4.67	150
CC17	75	>150	75	37.5	150	>150	>150	4.67	18.7
CC18	>150	>150	150	18.7	150	>150	>150	4.67	150
CC19	>150	>150	>150	37.5	150	>150	>150	4.67	150
CC20	>150	>150	150	300	>300	>150	>150	4.67	>300
STD[d]	37.5	75	>300	150	300	>300	>300	300	300

[a] *Bacillus subtilis*, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR 459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC 43300, Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299.; [b] *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*.; [c] *Candida albicans*; [d] *1,3,7-trihydroxyxanthone*;
[e] a mixture in 1:1 ratio

The results showed that most of the isolated compounds **CC4**-**CC6** and **CC13**-**CC15** exhibited strong antibacterial activity specifically against *P. aeruginosa* (**Table 3**) (Boonnak *et al*., 2009). Interestingly, only compounds **CC9** and **CC10** exhibited strong activity against *Candida albicans*. It is important to note that compound **CC9** also exhibited broad spectrum antimicrobial activity against all Gram positive bacteria. Most of the the compounds which are active against *P. aeruginosa* are the 1,3,7-trihydroxyxanthones (**CC4** and **CC5**) or 1,3,7-trioxygenated xanthones with an oxygeranyl side-chain either at C-3 or C-7 and dihydroxyl groups (**CC14**-**CC15**) whose indicated structures might contribute to the strong antibacterial activity specifically against *P. aeruginosa*. However, when the free hydroxyl group of the 1,3,7-trihydroxyxanthone was cyclized onto the isoprenyl or geranyl side chain to form a chromane or chromene ring, the antibacterial activity against *P. aeruginosa* decreased drastically as shown in compounds **CC1**, **CC2** and **CC3a**.

Compounds **CC14** and **CC15** are the major components obtained from this plant, this prompts us to modify their structures for the structure-activity relationships (SARs). To investigate whether the free hydroxyl group was responsible for antibacterial activity, the acetylation with acetic anhydride in pyridine was therefore applied to **CC14** and **CC15**. Four acetylated geranyloxy xanthone derivatives: 3-acetoxy-7-geranyloxy-1-hydroxyxanthone (**CC16**) and 1,3-diacetoxy-7-geranyloxyxanthone (**CC17**) were obtained from **CC14**, whereas 7-acetoxy-3-geranyloxy-1-hydroxyxanthone (**CC18**) and 1,7-diacetoxy-3-geranyloxyxanthone (**CC19**) were obtained from **CC15** (Boonnak *et al*., 2009).

The antibacterial activity of acetylated geranyloxy xanthone derivatives **CC16-CC19** and dibrosylate **CC20** were evaluated. All of them showed strong anti-*P. aeruginosa* (**Table 3**). This implied that the free hydroxyl groups should not be responsible for the inhibition of *P. aeruginosa.* For further investigation of the role of an oxygeranyl side-chain, a 1,3,7-trihydroxyxanthone (**CC21**) obtained from the dried fruits of *Cratoxylum cochinchinense*, was tested against *P. aeruginosa*. It was inactive against Gram-negative bacteria as shown in **Table 3**. From these results, it can be suggested that the geranyl side-chain is necessary for anti-*P. aeruginosa* activity. Moreover, mixtures of compound **CC4** with compound CC**5**, and compound **CC14** with compound **CC15** were subjected to antimicrobial assay. Interestingly, the mixture of compounds **CC14** and **CC15** significantly increased antibacterial activity against MRSA compared with the pure forms as indicated by the lower of MIC values shown in **Table 3**. The mixture of compounds **CC4** and **CC5**, on the other hand, did not show any significant differences for antibacterial activity compared to the pure

forms as also shown in **Table 3**. Therefore, it may be proposed that the 1,3,7-trihydroxyxanthone with the isoprenyl or geranyl side chain at C-2 and C-4 in (**CC4**) and (**CC5**), respectively and 1,3,7-trioxygenated xanthone with the geranyl side chain at C-3 or C-7 in (**CC13**-**CC19**) are essential for their antibacterial activity against *P. aeruginosa*. Therefore, 1,3,7-trihydroxyxanthones (**CC4** and **CC5**) and 1,3,7-trioxy-genated xanthone with geranyl side-chain (**CC13**-**CC19**) should be considered as potent candidates as anti-*P. aeruginosa* (Boonnak *et al*., 2009).

3. ผลการวิจัยและอภิปรายผล (ติ้วขน)

การสกัดและแยกสารจากต้นติ้วขน

3.1 General experimental procedures

Melting points were determined on the Fisher-John melting point apparatus. Optical rotations were measured on a JASCO P-1020 digital polarimeter. UV and IR spectra were recorded on SPECORD S 100 (Analytikjena) and Perkin-Elmer FTS FT-IR spectrophotometer, respectively. The ^{1}H and ^{13}C NMR spectra were recorded on a 500 MHz Varian UNITY INOVA and/or 300 MHz Bruker FTNMR Ultra Shield^{TM} spectrometers in $CDCl_3$ or CD_3OD with TMS as the internal standard. Chemical shifts are reported in δ (ppm) and coupling constants (*J*) are expressed in hertz. EI and HREI mass spectra were measured on a Kratos MS 25 RFA spectrometer. Quick column chromatography (QCC) and column chromatography (CC) were carried out on silica gel 60 F_{254} (Merck) and silica gel 100 (Merck), respectively.

3.2 Plant materials

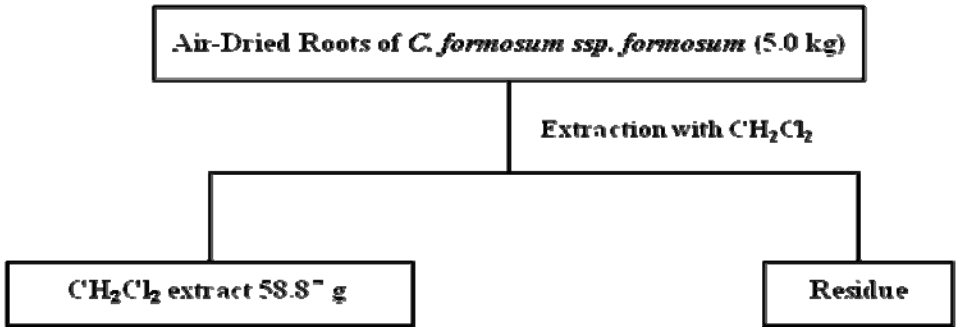
The roots and green fruits of *Cratoxylum formosum* ssp. *pruniflorum*

The roots of *C. formosum* ssp. *pruniflorum* were collected in May 2004 from Nong Khai Province, whereas the green fruits of *C. formosum* ssp. *pruniflorum* were collected in August 2008 from Pha Yao Province. Botanical identification was carried out by comparison with a voucher specimen number 0012677 in the herbarium collection of Department of Biology, Faculty of Science, Prince of Songkla University, Thailand.

3.3 Extraction and Isolation

3.3.1 The extraction of the roots of *Cratoxylum formosum* ssp. *pruniflorum*

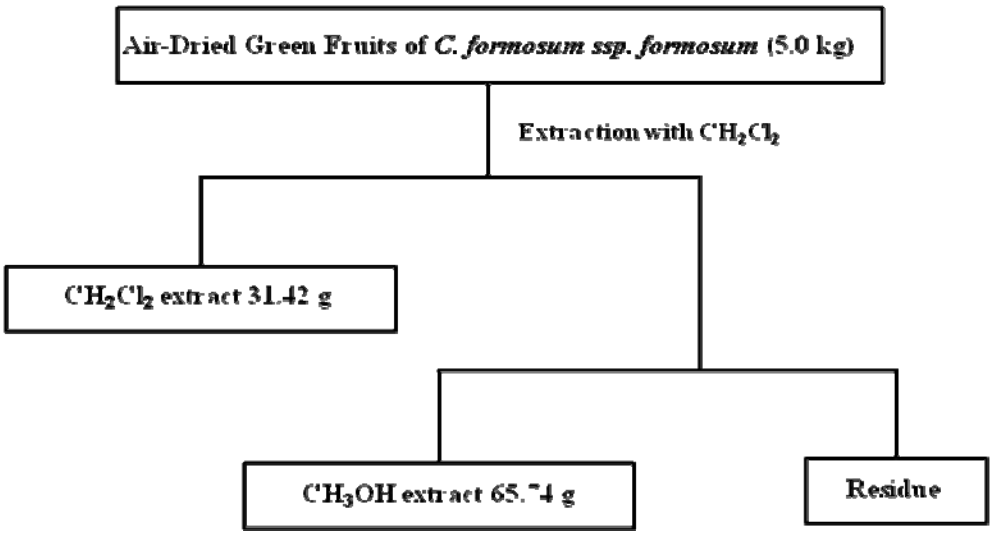
The air-dried roots of *C. formosum* ssp. *pruniflorum* (5.0 kg) was extracted with CH_2Cl_2 (2×20 L, for a week) at room temperature and was further evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (58.87 g) (see **Scheme 5**).



Scheme 5 The extraction of the roots of *C. formosum* ssp. *pruniflorum*

3.3.2 The extraction of the green fruits of *Cratoxylum formosum* ssp. *pruniflorum*

The green fruits of *C. formosum* ssp. *pruniflorum* (5.0 kg) was extracted with CH_2Cl_2 and CH_3OH (each 2×20 L, for a week) successively at room temperature and were further evaporated under reduced pressure to afford the crude extracts of CH_2Cl_2 (31.42 g) and CH_3OH (65.74 g) respectively (see **Scheme 6**).



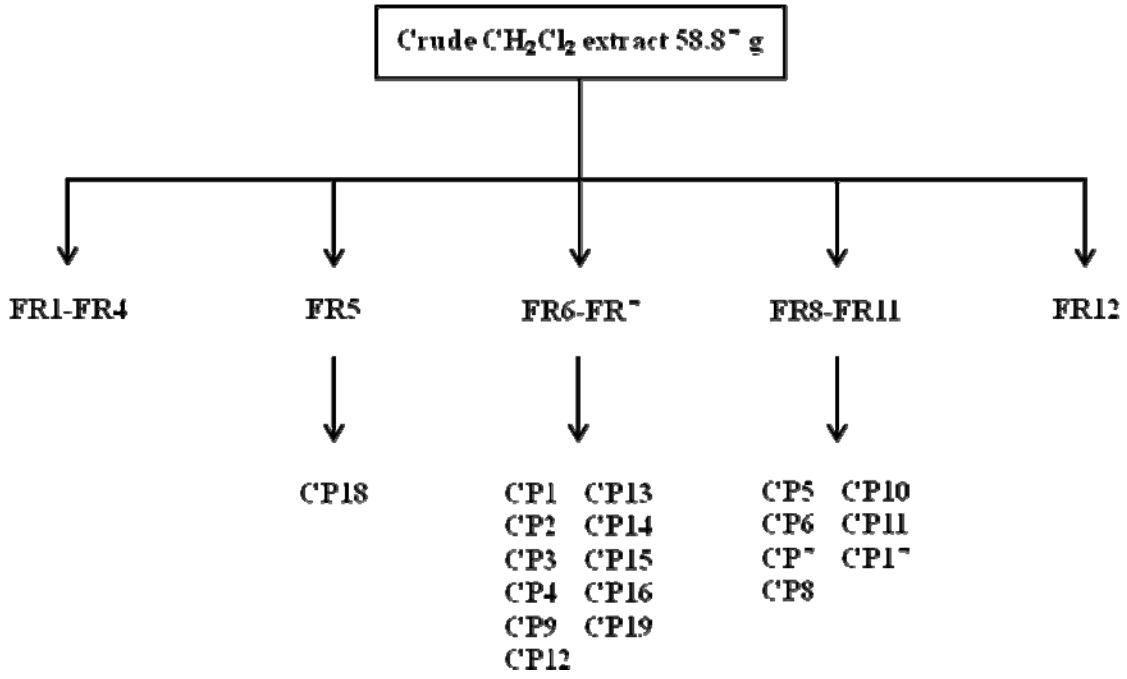
Scheme 6 The extraction of the green fruits of *C. formosum* ssp. *pruniflorum*

3.4 *การสกัดและแยกสารจากรากและผลอ่อนของต้นติ้วขน*

3.4.1 The CH_2Cl_2 extract of the roots of *Cratoxylum formosum* ssp. *pruniflorum*

The crude CH_2Cl_2 extract (58.87 g) of the roots of *C. formosum* spp. *pruniflorum* was subjected to QCC on silica gel using hexane as a first eluent and then increasing the polarity with acetone to give 12 fractions (FR1-FR12). Fraction FR5 was separated by QCC eluting with a gradient of CH_2Cl_2-hexane to give 9 subfractions (FR5A-FR5I), **CP18** (>350 mg) and caged prenylated xanthone (>150 mg). Fractions FR6 and FR7 were separated by QCC eluting with a gradient of acetone-hexane to give 10 subfractions (FR6A-FR6J). Subfraction FR6B was separated by QCC and eluted with a gradient of acetone-hexane to give 11 subfractions (FR6B1-FR6B11), **CP15** (1.5 mg) and **CP16** (2.5 mg). Subfractions FR6B2 were separated by CC and eluted with 10% acetone-hexane to give 4 subfractions (FR6B2A-FR6B2D) and **CP3** (25.5 mg). Subfractions FR6B6 and FR6B7 were separated by CC and eluted with 30% CH_2Cl_2-hexane to give 9 subfractions (FR6B6A-FR6B6I), **CP1** (5.7 mg), **CP19** (3.5 mg), caged prenylated xanthone (25.3 mg) and a mixture of β-sitosterol and stigmasterol (>55.4 mg), respectively. Subfraction FR6B6E was further purified by CC

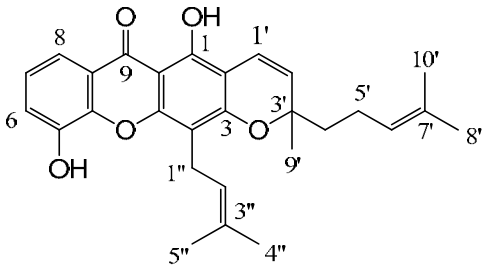
on silica gel C-18 and eluted with MeOH to furnish **CP9** (7.0 mg). Subfractions FR6B8 and FR6B9 were separated by CC and eluted with 30% CH_2Cl_2-n-hexane to give 10 subfractions (FR6B8A-FR6B8J), **CP4** (1.5 mg), dulxisxanthone F (23.2 mg), pruniflorone G (3.5 mg) and pruniflorone H (7.5 mg). Subfraction FR6H was further separated by QCC eluting with a gradient of acetone-n-hexane to give 11 subfractions (FR6H1-FR6H11), **CP2** (9.7 mg), cochinchinone A (80.7 mg) and 1,3,7-trihydroxy-2,4-diisoprenylxanthone (150.2 mg). Subfraction FR6H5 was further purified by CC using 10% acetone-n-hexane as a mobile phase to give cochinchinone I (5.6 mg). Subfraction FR6I was separated by QCC eluting with a gradient of acetone-hexane to give 7 subfractions (FR6I1-FR6I7), **CP12** (3.5 mg), **CP13** (5.6 mg) and **CP14** (4.5 mg), respectively. Fractions FR8-FR11 was separated by QCC eluting with 30% EtOAc-n-hexane to give 8 subfractions (FR8A-FR8H). Subfractions FR8E and FR8F were separated by QCC and eluted with 30% EtOAc-n-hexane to obtain 20 subfractions (FR8E1-FR8E20). Subfraction FR8E10-FR8E12 were separated by QCC and eluted with a gradient of CH_2Cl_2-n-hexane to give 12 subfractions (FR8E10A-FR8E10L). Subfraction FR8E10B was further purified by CC and eluted with 5% acetone-n-hexane to give **CP11** (4.5 mg) and **CP17** (5.6 mg). Subfraction FR8E10D was separated by CC eluting with 10% acetone-n-hexane to give 8 subfractions (FR8E10D1-FR8E10D8). Subfraction FR8E10D5 was further purified by CC and eluted with a gradient of CH_2Cl_2-n-hexane to give celebixanthone methyl ether (15.3 mg) and **CP6** (3.5 mg). Subfraction FR8E10E was separated by CC and eluted with a gradient of acetone-n-hexane to give 7 subfractions (FR8E10E1- FR8E10E7) and **CP8** (2.5 mg). Subfraction FR8E10F was separated by CC and eluted with a gradient of acetone-n-hexane to give 8 subfractions (FR8E10F1- FR8E10F8). Subfraction FR8E10F6 was further separated by CC and eluted with 60% $CHCl_3$-n-hexane to give 4 subfractions (FR8E10F6A- FR8E10F6D) and a mixture of macluraxanthone and **CP10** (35.5 mg) which was further purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give macluraxanthone (21.2 mg) and **CP10** (12.0 mg). Subfraction FR8E8 was separated by CC eluting with acetone-n-hexane to give **CP5** (7.5 mg). Subfraction FR8E9- FR8E11 were separated by CC and eluted with a gradient of acetone-n-hexane to give **CP7** (15.6 mg) (see **Scheme 7**).



Scheme 7 Isolation of compounds **CP1-CP19**

Characterization of isolated compounds

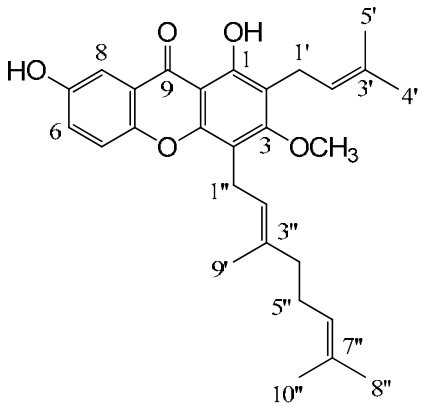
1. *Compound* CP1: *Pruniflorone K*



The structure of compound CP**1**

***Compound* CP1**: *Pruniflorone K*. Yellow viscous oil, $[\alpha]^{28}_{D}$ = -18.2 (*c* 0.285, $CHCl_3$); UV-Vis ($CHCl_3$) λ_{max} (log ε) 245 (4.63), 260 (4.56), 317 (4.37), 367 (3.73) nm; FT-IR (KBr) ν_{max} 3338, 1647, 1617 cm^{-1}; HREIMS *m/z* $[M]^+$ 446.2092 (calcd for $C_{28}H_{30}O_5$: 446.2093). EIMS *m/z* (rel. int.): 446 $[M]^+$ (13), 363 (100), 295 (8), 149 (14), 83 (8), 69 (13). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.1** (Boonnak *et al*., 2010)

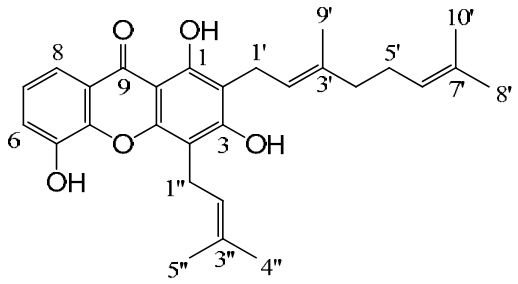
2. *Compound* CP2: *Pruniflorone L*



The structure of compound CP**2**

***Compound* CP2**: *Pruniflorone L.* Pale yellow powder, mp 259-260 ^{O}C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 245 (4.05), 268 (4.19), 317 (3.81), 388 (3.39) nm; FT-IR (KBr) v_{max} 3421, 1637 cm^{-1}; HREIMS *m/z* $[M]^+$ 462.2408 (calcd for $C_{29}H_{34}O_5$: 462.2406). EIMS *m/z* (rel. int.): 462 $[M]^+$ (100), 419 (83), 407 (74), 393 (29), 337 (100), 323 (16), 305 (23) 369 (22), 137 (10), 69 (16). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.2**. (Boonnak *et al*., 2010)

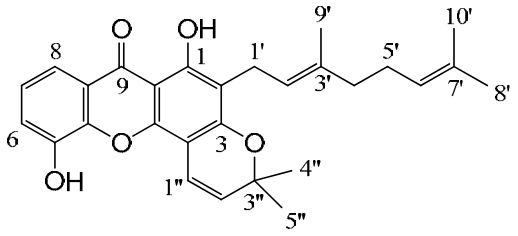
3. *Compound* CP3: *Formoxanthone A*



The structure of compound CP**3**

***Compound* CP3**: *Formoxanthone A*. Yellow powder, mp 111-113 ^{O}C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 245 (4.39), 269 (411), 332 (3.71), 377 (3.18) nm; FT-IR (KBr) v_{max} 3373, 1650 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.3**. (Boonsri *et al*., 2006)

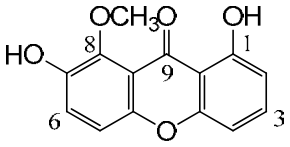
4. *Compound* CP4: *Formoxanthone B*



The structure of compound CP**4**

***Compound* CP4**: *Formoxanthone B.* Yellow powder, mp 144-146 ^{O}C; UV-Vis ($CHCl_3$) λ_{max} (log ε) 253 (4.15), 260 (4.29), 319 (4.08), 367 (3.50) nm; FT-IR (KBr) ν_{max} 3476, 1646 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.4**. (Boonsri *et al*., 2006)

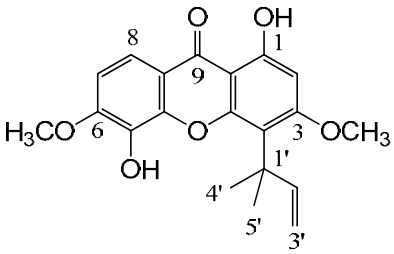
5. *Compound* CP5



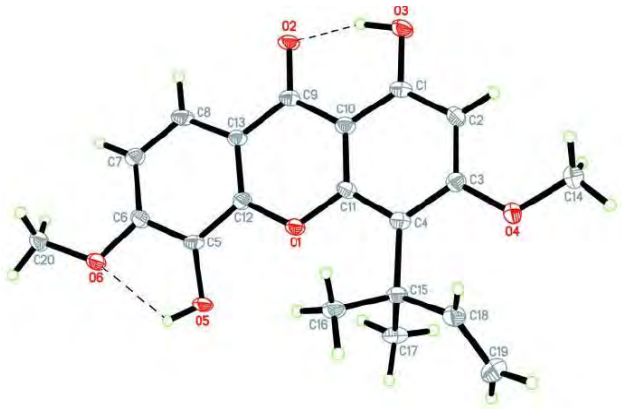
The structure of compound CP**5**

***Compound* CP5**: *1,7-dihydroxy-8-methoxyxanthone*. Yellow solid, mp 197-199 ^{O}C; UV-Vis (NaOH) λ_{max} 254, 275, 350 nm; FT-IR (KBr) ν_{max} 3330, 1647 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.5**. (Gottilieb *et al*., 1966; Kijjoa *et al*., 1997)

6. *Compound* CP6: *Vieillardiixanthone B*



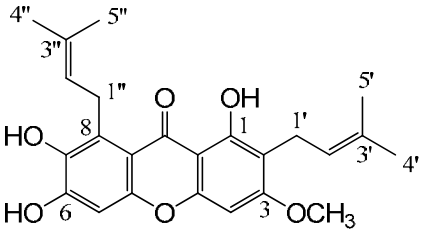
The structure of compound CP**6**



The X-ray structure of compound CP**6**

***Compound* CP6**: *Vieillardiixanthone B.* Yellow powder, mp 213-215 ^{O}C; UV-Vis (CH_3OH) λ_{max} (log ε) 217 (1.87), 253 (2.57), 286 (0.80), 327 (1.34) nm; FT-IR (neat) (CH_2Cl_2) ν_{max} 3304, 1643 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.6**. (Hay *et al*., 2008; Boonnak *et al*., 2010)

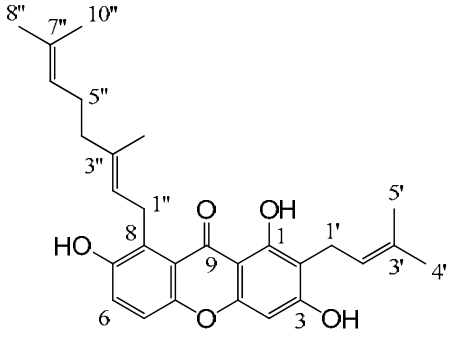
7. *Compound* CP7: *Dulcisxanthone B*



The structure of compound CP**7**

***Compound* CP7**: *Dulcisxanthone B.* Yellow powder, mp 170-172 ^{O}C; UV-Vis (CH_3OH) λ_{max} (log ε) 209 (4.25), 244 (4.50), 261 (4.49), 317 (4.25) 368 (4.04) nm; FT-IR (KBr) ν_{max} 3306, 1642 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.7**. (Dechathai *et al*., 2005)

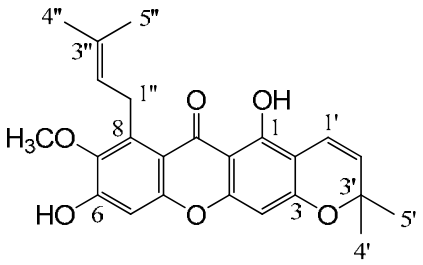
8. *Compound* CP8: *Cochinxanthone E*



The structure of compound CP**8**

***Compound* CP8**: *Cochinxanthone E.* Yellow oil; UV-Vis (CH_3OH) λ_{max} (log ε) 241 (4.20), 265 (4.18), 314 (3.92), 382 (3.43) nm; FT-IR (neat) ν_{max} 3437, 1638 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.8**. (Laphookhieo *et al*., 2009)

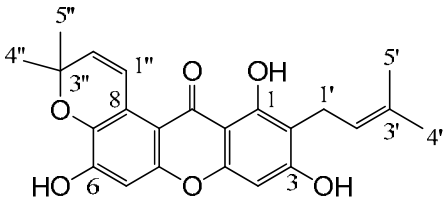
9. *Compound* CP9



The structure of compound CP**9**

Compound **CP9**: *5,9-dihydroxy-8-methoxy-2,2-dimethyl-7-(3-methyl-but-2-enyl)-2H,6H-pyrano[3,2b]xanthone.* Yellow powder, mp 156-157 ^{O}C. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.9**. (Sen *et al*., 1980)

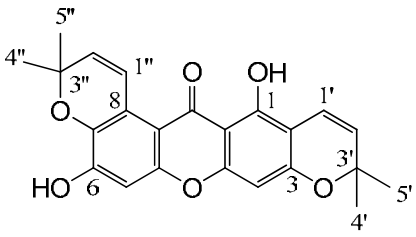
10. *Compound* CP10



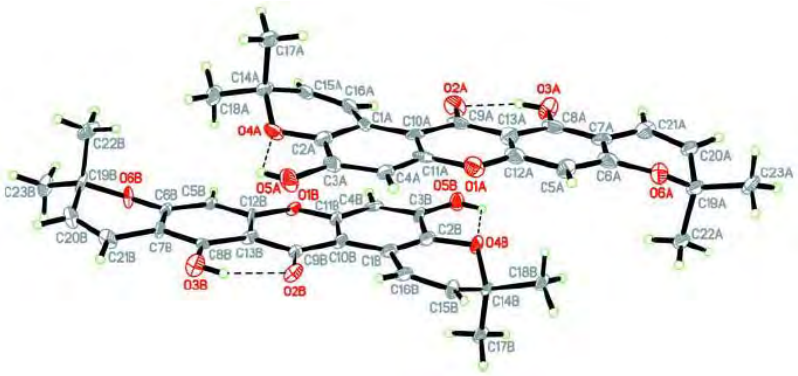
The structure of compound CP**10**

Compound **CP10**: *Garcinone B.* Yellow powder, mp 190-192 ^{O}C; UV-Vis (EtOH) λ_{max} (log ε) 247 (4.40), 267 (4.40), 339 (4.10), 390 (4.00) nm; FT-IR (KBr) v_{max} 3480, 1650 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.10**. (Sen *et al*., 1982)

11. *Compound* CP11



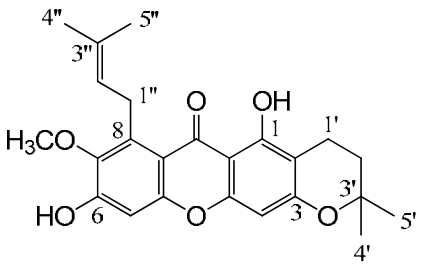
The structure of compound CP**11**



The X-ray structure of compound CP**11**

***Compound* CP11**: *Brasilixanthone.* Yellow powder, mp 205-207 ^{o}C; UV-Vis (CH_3OH) λ_{max} (log ε) 287 (3.95), 290 (3.94), 310 (3.81), 385 (3.33) nm; FT-IR (KBr) ν_{max} 3491, 3355, 1621 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.11**. (Marques *et al*., 2000; Chantrapromm *et al*., 2010)

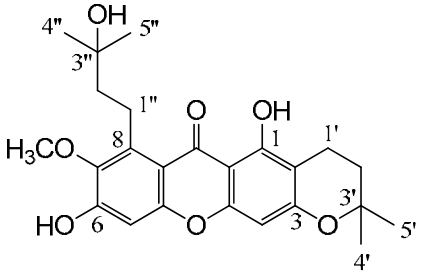
12. *Compound* CP12



The structure of compound CP**12**

***Compound* CP12**: *3-Isomangostin.* Yellow powder, mp 154-155 ^{O}C. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.12**. (Mahabusarakam *et al*., 1987)

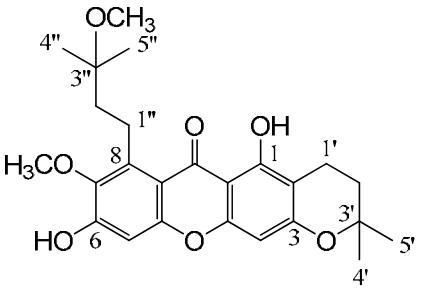
13. *Compound* CP13



The structure of compound CP**13**

***Compound* CP13**: *3,4-Dihydro-5,9-dihydroxy-7-(3-hydroxy-3-methylbutyl)-8-metho-xy-2,2-dimethyl-2H,6H-pyrano[3,2b]xanthone.* Yellow powder; mp 180-182 ^{O}C. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.13**. (Dutta *et al*., 1987)

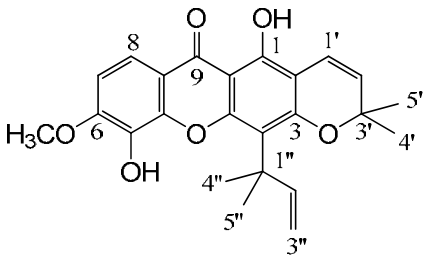
14. *Compound* CP14



The structure of compound CP**14**

***Compound* CP14**: *3,4-Dihydro-5,9-dihydroxy-8-methoxy-7-(3-methoxy-3-methyl-butyl)-2,2-dimethyl-2H,6H-pyrano[3,2b]xanthone.* Yellow oil. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.14**. (Dutta *et al*., 1987)

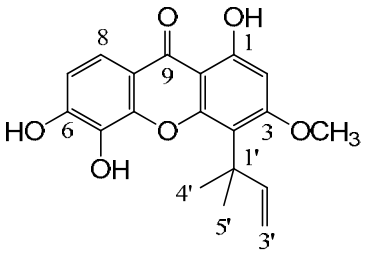
15. *Compound* CP15



The structure of compound CP**15**

***Compound* CP15**: *10-O-methylmacluraxanthone.* Yellow solid, mp 157-158 ^{o}C; UV-Vis (EtOH) λ_{max} (log ε) 242 (4.31), 281 (4.55), 290 (4.57), 334 (4.27) nm; FT-IR (nujol) ν_{max} 3520, 1652 cm^{-1}. For ^{1}H NMR (300 MHz, $CDCl_3$) spectroscopic data see **Table 3.15**. (Gunasekera *et al*., 1975)

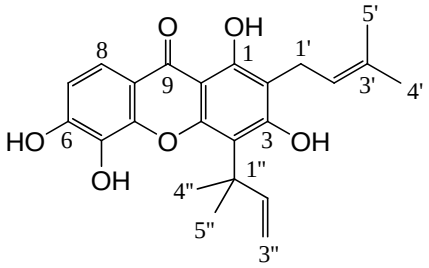
16. *Compound* CP16



The structure of compound CP**16**

***Compound* CP16**: *Isocudraniaxanthone B.* Yellow powder, UV-Vis (CH_3OH) λ_{max} (log ε) 246, 275, 293 (*sh*), 323 (*sh*), 399 nm; FT-IR (KBr) v_{max} 3370, 1650 cm^{-1}. For ^{1}H NMR (300 MHz, $CDCl_3$) spectroscopic data see **Table 3.16**. (Kobayashi *et al*., 1997)

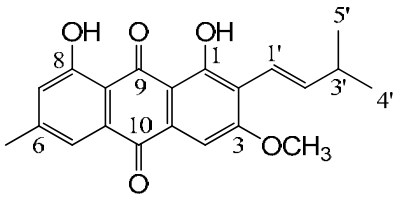
17. *Compound* CP17



The structure of compound CP**17**

***Compound* CP17**: *Gerontoxanthone I.* Yellow solid, mp 180-181 ^{O}C; UV-Vis (CH_3OH) λ_{max} (log ε) 203 (4.26), 253 (4.42), 287 (3.92), 328 (4.09) nm; FT-IR (KBr) v_{max} 3380, 1621 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.17**. (Chang *et al*., 1989)

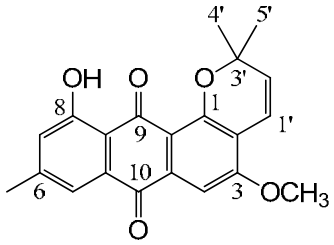
18. *Compound* CP18



The structure of compound CP**18**

***Compound* CP18:** *Vismiaquinone A.* Red-orange powder, mp 201-203 ^{O}C; UV-Vis (CH_3OH) λ_{max} (log ε) 220 (3.59), 278 (3.39), 425 (3.04) nm; FT-IR (KBr) v_{max} 3425, 1624 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.18**. (Goncalves and Mors, 1981)

19. *Compound* CP19



The structure of compound CP**19**

Compound **CP19**: *11-Hydroxy-5-methoxy-2,2,9-trimethyl-2H-anthra-[1,2b]pyran-7,12-dione.* Orange solid, mp 224-226 ^{O}C; UV-Vis (CH_3OH) λ_{max} (log ε) 208 (3.05), 224 (3.59), 265 (3.37), 285 (3.39), 424 (3.04) nm; FT-IR (KBr) ν_{max} 3446, 1646 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (125 MHz) NMR ($CDCl_3$) spectroscopic data see **Table 3.19**. (Delle Monache *et al*., 1979)

Table 3.1 NMR spectroscopic data of **CP1** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.08, *s*	156.1	C-1, C-2, C-3, C-9a
2	C		104.5	
3	C		158.5	
4	C		106.7	
5-OH	C	5.73, *br s*	144.5	C-5, C-6, C-4b
6	CH	7.30, *dd*, 7.8, 1.8	119.8	C-8, C-4b
7	CH	7.23, *t*, 7.8	123.9	C-5, C-8a
8	CH	7.75, *dd*, 7.5, 1.8	116.8	C-9, C-4b
9	C=O		181.5	
4a	C		154.7	
4b	C		144.5	
8a	C		120.9	
9a	C		103.3	
1′	CH	6.79, *d*, 10.2	116.2	C-1, C-2, C-3, C-3′
2′	CH	5.56, *d*, 10.2	126.2	C-2, C-3′, C-4′, C-9′
3′	C		80.9	
4′	CH_2	1.78, *m*	41.8	C-2′, C-3′, C-4′, C-5′, C-9′
5′	CH_2	2.12, *m*	22.7	C-4′, C-6′, C-7′
6′	CH	5.09, *brt*, 6.9	123.7	C-5′, C-8′, C-10′
7′	C		131.9	
8′	CH_3	1.68, *s*	25.6	C-6′, C-7′
9′	CH_3	1.45, *s*	27.2	C-2′, C-3′, C-4′
10′	CH_3	1.45, *s*	17.6	C-6′, C-7′
1′′	CH_2	3.50, *d*, 6.9	21.7	C-3, C-4, C-4a, C-2′′, C-3′′
2′′	CH	5.23, *br t*, 6.9	122.7	C-1′′, C-5′′
3′′	C		131.7	
4′′	CH_3	1.72, *s*	25.5	C-2′′, C-3′′
5′′	CH_3	1.84, *s*	17.9	C-2′′, C-3′′

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.2 NMR spectroscopic data of **CP2** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.78, *s*	158.8	C-1, C-2, C-3, C-9, C-9a
2	C		116.9	
3	C		163.8	
4	C		113.1	
5	CH	7.28, *d*, 8.7	118.2	C-6, C-7, C-8, C-9, C-4b, C-8a
6	CH	7.50, *dd*, 8.7, 1.8	124.4	C-7, C-8, C-4b, C-8a
7-OH	C	6.55, *br s*	152.3	-
8	CH	7.54, *d*, 1.8	108.9	C-6, C-7, C-9, C-4b
9	C=O		181.6	
4a	C		153.4	
4b	C		150.7	
8a	C		120.7	
9a	C		105.8	
1′	CH_2	3.34, *d*, 6.6	22.6	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.20, *br t*, 6.6	122.6	C-2, C-1′, C-4′, C-5′
3′	C		131.9	
4′	CH_3	1.63, *s*	25.7	C-2′, C-3′
5′	CH_3	1.74, *s*	17.9	C-2′, C-3′
1″	CH_2	3.36, *d*, 6.9	22.7	C-3, C-4, C-4a, C-2″, C-3″, C-9
2″	CH	5.15, *br t*, 6.9	122.8	C-4, C-1″, C-4″, C-9″
3″	C		135.3	
4″	CH_2	1.94, *m*	39.6	C-2″, C-3″, C-6″
5″	CH_2	1.98, *m*	26.6	C-3″, C-6″, C-7″
6″	CH	4.95, *br t*, 6.6	124.1	C-4″, C-5″, C-8″, C-10″
7″	C		131.4	
8″	CH_3	1.50, *s*	25.6	C-6″, C-7″
9″	CH_3	1.80, *s*	16.3	C-2″, C-3″
10″	CH_3	1.46, *s*	17.6	C-6″, C-7″
3-OCH_3	CH_3	3.74, *s*	61.9	C-3

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.3 NMR spectroscopic data of **CP3** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.20, *s*	158.6	C-1, C-2, C-9a
2	C		108.9	
3-OH	C	6.57, *s*	161.1	C-2, C-3, C-4
4	C		105.7	
5-OH	C	5.86, *br s*	144.5	-
6	CH	7.29, *dd*, 7.8, 0.9	119.8	C-8, C-4b
7	CH	7.22, *t*, 7.8	123.8	C-5, C-8a
8	CH	7.75, *dd*, 7.8, 0.9	116.9	C-9, C-4b
9	C=O		181.1	
4a	C		152.5	
4b	C		144.3	
8a	C		120.9	
9a	C		103.3	
1′	CH_2	3.50, *d*, 7.2	21.6	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.29, *br t*, 7.2	121.1	C-1′, C-4′, C-9′
3′	C		140.1	
4′	CH_2	2.11, *m*	39.7	C-2′, C-3′, C-6′
5′	CH_2	2.11, *m*	26.3	C-3′, C-7′
6′	CH	5.06, *m*	123.7	-
7′	C		132.2	
8′	CH_3	1.68, s	25.7	C-6′, C-7′
9′	CH_3	1.85, *s*	16.3	C-2′, C-3′
10′	CH_3	1.60, s	17.7	C-6′, C-7′
1″	CH_2	3.54, *d*, 6.9	22.0	C-3, C-4, C-4a, C-2″, C-3″
2″	CH	5.26, *br t*, 6.9	122.4	C-5″
3″	C		133.1	
4″	CH_3	1.74, *s*	25.6	C-2″, C-3″
5″	CH_3	1.86, *s*	17.9	C-2″, C-3″

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.4 NMR spectroscopic data of **CP4** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.20, *s*	160.6	C-1, C-2, C-9a
2	C		112.3	
3	C		158.7	
4	C		100.7	
5	C		144.3	
6	CH	7.31, *dd*, 7.8, 1.8	120.1	C-5, C-8
7	CH	7.25, *t*, 7.8	123.9	C-5, C-8a
8	CH	7.79, *dd*, 7.8, 1.8	117.2	C-6, C-4b, C-9
9	C=O		180.8	
4a	C		149.2	
4b	C		144.1	
8a	C		121.2	
9a	C		103.2	
1′	CH_2	3.38, *d*, 7.2	21.1	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.26, *br t*, 7.2	121.7	C-1′, C-4′, C-9′
3′	C		135.2	
4′	CH_2	2.02, *m*	39.8	C-5′, C-9′
5′	CH_2	2.02, *m*	26.7	C-4′
6′	CH	5.09, *br t*, 7.2	124.4	-
7′	C		131.3	
8′	CH_3	1.64, s	25.7	C-6′, C-7′, C-10′
9′	CH_3	1.82, *s*	16.3	C-2′, C-3′, C-4′
10′	CH_3	1.58, s	17.7	C-6′, C-7′, C10′
1″	CH	6.80, *d*, 9.9	114.9	C-3, C-4, C-4a, C-3″
2″	CH	5.65, *d*, 9.9	127.4	C-4, C-3″, C-4″, C-5″
3″	C		78.1	
4″	CH_3	1.50, *s*	28.2	C-2″, C-3″
5″	CH_3	1.50, *s*	28.2	C-2″, C-3″

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.5 NMR spectroscopic data of **CP5** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.82, *s*	162.0	C-1, C-2, C-3, C-9a
2	CH	6.70, *br d*, 8.4	110.2	C-1, C-4, C-9a
3	CH	7.49, *t*, 8.4	136.6	C-1, C-4a
4	CH	6.80, *br d*, 8.4	106.5	C-2, C-9, C-4a, C-9a
5	CH	7.14, *d*, 9.3	114.2	C-6, C-7, C-9, C-4b, C-8a
6	CH	7.36, *d*, 9.3	123.3	C-5, C-7, C-8, C-4b
7	C		145.5	
8	C		144.2	
9	C=O		182.0	
4a	C		155.8	
4b	C		150.9	
8a	C		114.8	
9a	C		109.0	
8-OCH_3	CH_3	3.97, *s*	62.8	C-8

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.6 NMR spectroscopic data of **CP6** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.82, *s*	162.5	C-1, C-2, C-9a
2	CH	6.33, *s*	95.6	C-1, C-3, C-4, C-9a
3	C		165.4	
4	C		113.6	
5-OH	C	6.18, *br s*	133.6	C-5, C-6, C-4b
6	C		151.6	
7	CH	6.90, *d*, 8.8	108.3	C-5, C-6, C-8a
8	CH	7.68, *d*, 9.2	116.9	C-6, C-9, C-4b
9	C=O		181.1	
4a	C		154.0	
4b	C		144.6	
8a	C		114.2	
9a	C		103.1	
1′	C		41.5	
2′	CH	6.58, *dd*, 17.6, 10.4	155.1	C-4, C-1′, C-4′, C-5′
3′	CH_2	5.10, *d*, 17.6	104.5	C-1′, C-2′
		4.97, *d*, 10.4		C-1′
4′	CH_3	1.56, *s*	28.2	C-4, C-1′, C-2′
5′	CH_3	1.56, *s*	28.1	C-4, C-1′, C-2′
3-OCH_3	CH_3	3.82, *s*		C-3
6-OCH_3	CH_3	3.96, *s*	62.8	C-6

[a]Recorded in 300 MHz.; [b]Recorded in 75 MHz.

Table 3.7 NMR spectroscopic data of **CP7** in $CDCl_3$

Position	**Type of C**	$\delta_H{}^a$ **(*J* in Herz)**	$\delta_C{}^b$	**HMBC ($^1H \rightarrow {}^{13}C$)**
1-OH	C	13.44, *s*	159.7	C-1, C-2, C-9a
2	C		111.4	
3	C		163.5	
4	CH	6.32, *s*	88.8	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.81, *s*	101.1	C-6, C-7, C-4b, C-8a
6	C		150.7	
7	C		139.6	
8	C		127.4	
9	C=O		182.6	
4a	C		155.3	
4b	C		153.5	
8a	C		111.7	
9a	C		103.9	
1′	CH_2	3.35, *d*, 7.2	21.4	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.23, *br t*, 7.2	122.4	C-2
3′	C		131.7	
4′	CH_3	1.68, *s*	25.8	C-2′, C-3′, C-5′
5′	CH_3	1.80, *s*	17.8	C-2′, C-3′, C-4′
1″	CH_2	4.33, *d*, 6.9	26.0	C-7, C-8, C-8a, C-2″, C-3″
2″	CH	5.31, *br t*, 6.9	121.5	C-8
3″	C		135.6	
4″	CH_3	1.79, *s*	25.8	C-2″, C-3″, C-5″
5″	CH_3	1.89, *s*	18.1	C-2″, C-3″, C-4″
3-OCH_3	CH_3	3.90, *s*	55.8	C-3

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.8 NMR spectroscopic data of **CP8** in $CDCl_3$

Position	**Type of C**	$\delta_H{}^a$ **(*J* in Herz)**	$\delta_C{}^b$	**HMBC (^{1}H→^{13}C)**
1-OH	C	13.44, *s*	160.7	C-1, C-2, C-9a
2	C		108.4	
3	C		162.2	
4	CH	6.24, *s*	93.2	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.13, *s*	116.7	C-7, C-9, C-4b, C-8a
6	CH	7.13, *s*	123.7	C-7, C-8, C-4b
7	C		151.3	
8	C		127.1	
9	C=O		183.5	
4a	C		155.3	
4b	C		152.0	
8a	C		118.5	
9a	C		104.1	
1′	CH_2	3.45, *d*, 7.2	21.5	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.23, *br t*, 7.2	121.4	C-4′, C-5′
3′	C		135.7	
4′	CH_3	1.70, *s*	25.8	C-2′, C-3′
5′	CH_3	1.78, *s*	17.9	C-2′, C-3′
1″	CH_2	4.25, *d*, 7.2	25.7	C-7, C-8, C-8a, C-2″, C-3″
2″	CH	5.20, *br t*, 7.2	121.4	C-4″, C-9″
3″	C		138.7	
4″	CH_2	2.02, *m*	39.7	C-9″
5″	CH_2	2.02, *m*	26.4	C-4″
6″	CH	4.97, *br t*, 6.0	123.7	-
7″	C		131.9	
8″	CH_3	1.59, *s*	25.8	C-6″, C-7″
9″	CH_3	1.80, *s*	16.4	C-2″, C-3″, C-4″
10″	CH_3	1.51, *s*	17.7	C-6″, C-7″

[a]Recorded in 300 MHz.; [b]Recorded in 75 MHz.

Table 3.9 NMR spectroscopic data of **CP9** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.63, *s*	157.9	C-1, C-2, C-9, C-9a
2	C		104.5	
3	C		159.8	
4	CH	6.17, *s*	94.1	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.81, *s*	101.7	C-6, C-7, C-8, C-4b, C-8a
6	C		155.7	
7	C		142.7	
8	C		137.0	
9	C=O		182.0	
4a	C		156.2	
4b	C		154.8	
8a	C		112.1	
9a	C		103.7	
1′	CH	6.66, *d*, 9.9	115.7	C-1, C-2, C-3, C-3′
2′	CH	5.50, *d*, 9.9	127.1	C-2, C-3′, C-4′, C-5′
3′	C		77.9	
4′	CH_3	1.39, *s*	28.3	C-2′, C-3′
5′	CH_3	1.39, *s*	28.3	C-2′, C-3′
1″	CH_2	4.01, *d*, 6.3	26.5	C-7, C-8, C-8a, C-2″, C-3″
2″	CH	5.19, *br t*, 6.6	123.2	C-8, C-4″, C-5″
3″	C		132.1	
4″	CH_3	1.62, *s*	25.8	C-2″, C-3″, C-5″
5″	CH_3	1.76, *s*	18.2	C-2″, C-3″, C-4″
7-OCH_3	CH_3	3.73, *s*	61.9	C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.10 NMR spectroscopic data of **CP10** in $CDCl_3$

Position	**Type of C**	$\delta_H{}^a$ **(*J* in Herz)**	$\delta_C{}^b$	**HMBC ($^1H \rightarrow {}^{13}C$)**
1-OH	C	13.62, *s*	160.5	C-1, C-2, C-9a
2	C		108.5	
3	C		161.8	
4	CH	6.25, *s*	93.4	-
5	CH	6.74, *s*	102.3	-
6	C		153.1	
7	C		136.8	
8	C		119.8	
9	C=O		180.3	
4a	C		155.2	
4b	C		151.7	
8a	C		108.6	
9a	C		103.8	
1′	CH_2	3.38, *d*, 6.6	21.4	C-2, C-3, C-2′, C-3′
2′	CH	5.23, *br t*, 6.6	121.5	C-1′, C-4′
3′	C		135.6	
4′	CH_3	1.70, *s*	25.8	C-2′, C-3′, C-5′
5′	CH_3	1.77 *s*	17.9	C-1′, C-3′, C-4′
1″	CH	7.95, *d*, 10.2	121.0	C-7, C-8, C-8a, C-3″
2″	CH	5.75, *d*, 10.2	132.3	C-7, C-8, C-3″, C-4″, C-5″
3″	C		76.7	
4″	CH_3	1.43, *s*	27.3	C-2″, C-3″
5″	CH_3	1.43, *s*	27.3	C-2″, C-3″

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.11 NMR spectroscopic data of **CP11** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.55, *s*	157.8	C-1, C-2, C-3, C-9, C-9a
2	C		104.4	
3	C		159.9	
4	CH	6.19, *s*	94.3	C-2, C-3, C-9, C-4a, C-8a
5	CH	6.75, *s*	102.4	C-6, C-7, C-9, C-4b, C-8a
6	C		153.1	
7	C		136.8	
8	C		119.7	
9	C=O		182.4	
4a	C		156.6	
4b	C		150.9	
8a	C		108.6	
9a	C		103.9	
1′	CH	6.65, *d*, 10.2	115.7	C-1, C-2, C-3, C-3′
2′	CH	5.50, *d*, 10.2	127.2	C-2, C-3′, C-4′, C-5′
3′	C		78.0	
4′	CH_3	1.40, *s*	28.3	C-1′, C-2′, C-3′
5′	CH_3	1.40, *s*	28.3	C-1′, C-2′, C-3′
1″	CH	7.94, *d*, 10.2	120.9	C-7, C-8, C-8a, C-3″
2″	CH	5.75, *d*, 10.2	132.3	C-7, C-8, C-4″, C-5″
3″	C		76.8	
4″	CH_3	1.42, *s*	27.3	C-7, C-1″, C-2″, C-3″
5″	CH_3	1.42, *s*	27.3	C-7, C-1″, C-2″, C-3″

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.12 NMR spectroscopic data of **CP12** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.73, *s*	160.6	C-1, C-2, C-9a
2	C		103.8	
3	C		160.7	
4	CH	6.23, *s*	94.0	C-3, C-9, C-4a, C-8a
5	CH	6.82, *s*	101.6	C-6, C-7, C-9, C-4b, C-8a
6	C		155.9	
7	C		142.4	
8	C		136.9	
9	C=O		182.0	
4a	C		152.5	
4b	C		154.7	
8a	C		112.1	
9a	C		102.9	
1′	CH_2	2.71, *t*, 6.6	16.1	C-1, C-2, C-3, C-2′, C-3′
2′	CH_2	1.83, *t*, 6.6	31.9	C-2, C-1′, C-3′
3′	C		76.0	
4′	CH_3	1.37, *s*	26.7	C-4′, C-5′
5′	CH_3	1.37, *s*	26.7	C-4′, C-5′
1″	CH_2	4.10, *d*, 6.3	26.5	C-7, C-8, C-8a, C-2″, C-3″
2″	CH	5.27, *br t*, 6.3	123.3	-
3″	C		132.2	
4″	CH_3	1.69, *s*	25.8	C-2″, C-3″, C-5″
5″	CH_3	1.83, *s*	18.2	C-2″, C-3″, C-4″
7-OCH_3	CH_3	3.80, *s*	62.0	C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.13 NMR spectroscopic data of **CP13** in $CDCl_3$

Position	**Type of C**	δ_H[a] (***J* in Herz**)	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.60, *s*	160.5	-
2	C		103.9	
3	C		160.9	
4	CH	6.22, *s*	94.1	C-3, C-9, C-4a
5	CH	6.83, *s*	101.8	C-6, C-7, C-9, C-4b, C-8a
6	C		154.7	
7	C		142.5	
8	C		138.4	
9	C=O		182.0	
4a	C		154.8	
4b	C		156.1	
8a	C		111.8	
9a	C		102.8	
1′	CH_2	2.70, *t*, 6.9	16.1	C-1, C-2, C-3, C-2′, C-3′
2′	CH_2	1.83, *t*, 6.6	31.9	C-2, C-3′, C-4′, C-5′
3′	C		76.1	
4′	CH_3	1.37, *s*	26.8	C-2′, C-3′
5′	CH_3	1.37, *s*	26.8	C-2′, C-3′
1″	CH_2	3.42, *br t*, 8.1	22.1	C-7, C-8, C-3″
2″	CH_2	1.79, *br t*, 8.1	44.4	C-8, C-1″, C-3″, C-4″, C-5″
3″	C		70.8	
4″	CH_3	1.33, *s*	29.2	C-2″, C-3″
5″	CH_3	1.33, *s*	29.2	C-2″, C-3″
7-OCH_3	CH_3	3.86, *s*	62.2	C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.14 NMR spectroscopic data of **CP14** in $CDCl_3$

Position	**Type of C**	δ_H[a] (*J* **in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.90, *s*	160.7	-
2	C		103.7	
3	C		160.7	
4	CH	6.23, *s*	93.9	C-3, C-4a, C-9a
5	CH	6.81, *s*	101.5	C-6, C-7, C-4b, C-8a
6	C		156.0	
7	C		142.4	
8	C		138.7	
9	C=O		182.0	
4a	C		154.5	
4b	C		154.7	
8a	C		111.9	
9a	C		102.8	
1′	CH_2	2.71, *t*, 6.9	16.1	C-1, C-2, C-3, C-2′, C-3′
2′	CH_2	1.84, *t*, 6.9	31.9	C-2, C-1′, C-3′, C-4′, C-5′
3′	C		76.0	
4′	CH_3	1.37, *s*	26.8	C-3′
5′	CH_3	1.37, *s*	26.8	C-3′
1″	CH_2	3.39, *br t*, 8.1	22.1	C-7, C-8, C-8a, C-2″
2″	CH_2	1.75, *br t*, 8.1	39.8	C-8, C-1″, C-3″, C-4″, C-5″
3″	C		74.9	
4″	CH_3	1.30, *s*	25.2	C-3″
5″	CH_3	1.30, *s*	25.2	C-3″
3″-OCH_3	CH_3	3.32, *s*	49.2	C-3″
7-OCH_3	CH_3	3.86, *s*	62.1	C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.15 NMR spectroscopic data of **CP15** in $CDCl_3$

Position	CP15		CC10		
	Type of C	**δ_H[a] (*J* in Herz)**	**Type of C**	**δ_H[a] (*J* in Herz)**	**δ_C[b]**
1-OH	C	13.52, *s*	C	13.53, *s*	156.8
2	C		C		105.6
3	C		C		158.9
4	C		C		113.1
5	C		C		131.1
6	C		C		149.0
7	CH	6.97, *d*, 8.7	CH	6.94, *d*, 9.0	112.8
8	CH	7.74, *d*, 8.7	CH	7.68, *d*, 9.0	117.5
9	C=O		C=O		180.8
4a	C		C		154.1
4b	C		C		144.5
8a	C		C		113.7
9a	C		C		103.8
1′	CH	6.77, *d*, 9.9	CH	6.76, *d*, 9.9	116.1
2′	CH	5.61, *d*, 9.9	CH	5.61, *d*, 9.9	127.2
3′	C		C		78.3
4′	CH_3	1.51, *s*	CH_3	1.52, *s*	27.9
5′	CH_3	1.51, *s*	CH_3	1.52, *s*	27.9
1″	C		C		41.4
2″	CH	6.66, *dd*, 17.7, 10.8	CH	6.76, *dd*, 17.7, 10.5	156.8
3″	CH_2	5.18, *br d*, 17.7	CH_2	5.22, *dd*, 17.7, 1.5	103.3
		5.04, *br d*, 10.8		5.05, *dd*, 10.5, 1.5	
4″	CH_3	1.66, *s*	CH_3	1.65, *s*	28.2
5″	CH_3	1.66, *s*	CH_3	1.65, *s*	28.2
6-OCH_3	CH_3	3.98, *s*	-	-	-

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.16 NMR spectroscopic data of **CP16** in $CDCl_3$

Position	CP16			CP6	
	Type of C	**δ_H[a] (*J* in Herz)**	**NOESY**	**δ_H[a] (*J* in Herz)**	**δ_C[b]**
1-OH	C	13.38, *s*		12.82, *s*	162.5
2	CH	6.40, *s*	3-OCH_3	6.33,	95.6
3	C				165.4
4	C				113.6
5-OH	C			6.18, *br s*	133.6
6	C				151.6
7	CH	6.95, *d*, 8.4	H-8	6.90, *d*, 8.8	108.3
8	CH	7.69, *d*, 8.7	H-7	7.68, *d*, 9.2	116.9
9	C=O				181.1
4a	C				154.0
4b	C				144.6
8a	C				114.2
9a	C				103.1
1′	C				41.5
2′	CH	7.73, *dd*, 17.4, 10.8	H-3′	6.58, *dd*, 17.6, 10.4	155.1
3′	CH_2	5.21, *br d*, 17.4	H-2′	5.10, *d*, 17.6	104.5
		5.04, *br d*, 10.5		4.97, *d*, 10.4	
4′	CH_3	1.58, *s*		1.56, *s*	28.2
5′	CH_3	1.58, *s*		1.56, *s*	28.1
3-OCH_3	CH_3	3.90, *s*	H-2	3.82, *s*	
6-OCH_3	CH_3	-		3.96, *s*	62.8

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.17 NMR spectroscopic data of **CP17** in $CDCl_3$

Position	**Type of C**	δ_H[a] **(J in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	13.61, *s*	158.9	C-1, C-2, C-9a
2	C		110.2	
3	C		161.4	
4	C		111.1	
5	C		131.0	
6	C		149.0	
7	CH	6.94, *d*, .87	112.6	C-5, C-6, C-8a
8	CH	7.70, *d*, 8.7	117.6	C-6, C-9, C-4b
9	C		180.8	
4a	C		153.3	
4b	C		144.8	
8a	C		113.8	
9a	C		103.0	
1′	CH_2	3.47, *d*, 6.9	21.6	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.24, *br t*, 6.9	121.2	C-1′, C-4′, C-5′
3′	C		135.9	
4′	CH_3	1.79, *s*	25.9	C-2′, C-3′
5′	CH_3	1.86, *s*	17.9	C-2′, C-3′
1″	C		41.6	
2″	CH	6.68, *dd*, 17.7, 10.5	154.6	C-1″, C-4″, C-5″
3″	CH_2	5.30, *dd*, 17.7, 0.9	106.6	C-1″, C-2″
		5.15, *dd*, 10.5, 0.9		-
4″	CH_3		28.0	C-4, C-1″, C-2″
5″	CH_3	1.69, *s*	28.0	C-4, C-1″, C-2″

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.18 NMR spectroscopic data of **CP18** in $CDCl_3$

Position	**Type of C**	**δ_H[a] (*J* in Herz)**	**δ_C[b]**	**HMBC ($^1H \rightarrow ^{13}C$)**
1-OH	C	12.84, *s*	162.5	C-1, C-2, C-9, C-9a
2	C		120.0	
3	C		163.0	
4	CH	7.40, *s*	103.4	C-2, C-3, C-9, C-10, C-4a, C-9a
5	CH	7.61, *br s*	121.1	C-6, C-7, C-8, C-9, C-10, C-8a
6	C		148.4	
7	CH	7.07, *br s*	124.4	C-5, C-8, 6-CH_3
8-OH	C	12.02, *s*	162.1	C-7, C-8, C-9, 6-CH_3, C-8a
9	C=O		191.4	
10	C=O		181.9	
4a	C		132.1	
4b	C		133.2	
8a	C		113.7	
9a	C		110.5	
1′	CH	6.66, *dd*, 16.2, 1.2	115.8	C-1, C-2, C-3
2′	CH	6.92, *dd*, 16.2, 7.2	146.8	C-2
3′	CH	2.50, *m*	33.4	C-1′, C-2′, C-4′, C-5′
4′	CH_3	1.14, *d*, 6.9	22.5	C-1′, C-3′
5′	CH_3	1.14, *d*, 6.9	22.5	C-1′, C-3′
3-OCH_3	CH_3	4.05, *s*	56.3	C-3
6-CH_3	CH_3	2.45, *s*	22.2	C-6, C-7, C-4b

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.19 NMR spectroscopic data of **CP19** in $CDCl_3$

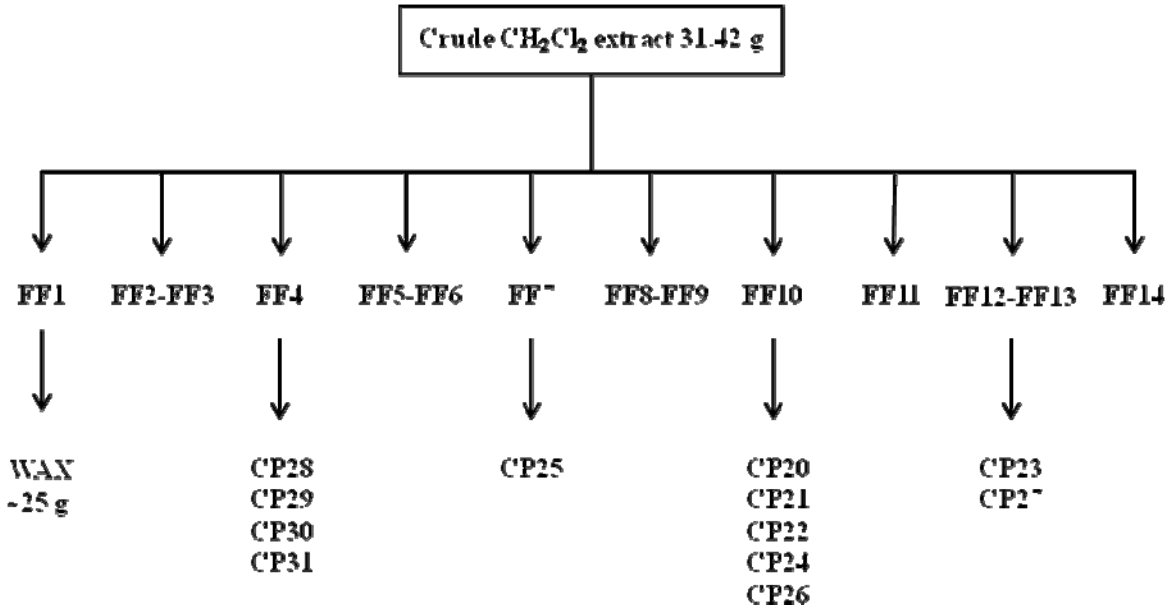
Position	**Type of C**	δ_H[a] **(J in Herz)**	δ_C[b]	**HMBC ($^1H \rightarrow ^{13}C$)**
1	C		156.3	
2	C		114.9	
3	C		158.8	
4	CH	7.43, *s*	102.8	C-3, C-9, C-10, C-4a, C-9a
5	CH	7.56, *dd*, 1.5, 0.6	119.8	C-7, C-10, C-8a, 6-CH_3
6	C		146.7	
7	CH	7.67, *dd*, 1.8, 0.9	124.5	C-5, C-8, C-8a, 6-CH_3
8-OH	C	13.18, *s*	162.6	C-7, C-8, C-8a, 6-CH_3
9	C=O		187.2	
10	C=O		182.7	
4a	C		135.4	
4b	C		132.6	
8a	C		115.4	
9a	C		116.3	
1′	CH	6.73, *d*, 10.2	116.1	C-1, C-3
2′	CH	5.84, *d*, 10.2	132.2	C-1′, C-3′, C-4′, C-5′
3′	C		77.8	
4′	CH_3	1.57, *s*	27.9	C-2′, C-3′
5′	CH_3	1.57, *s*	27.9	C-2′, C-3′
3-OCH_3	CH_3	4.03, *s*	56.2	C-3
6-CH_3	CH_3	2.42, *s*	22.0	C-5, C-6, C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

3.4.2 The CH_2Cl_2 extract of the green fruits of *Cratoxylum formosum* ssp. *pruniflorum*

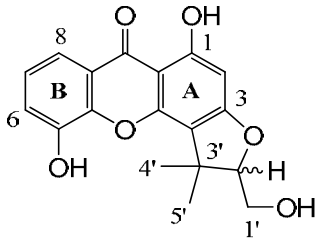
The crude CH_2Cl_2 extract (31.42 g) of the green fruits *C. formosum* spp. *pruniflorum* was further subjected to QCC on silica gel using hexane as a first eluent and then increasing the polarity with acetone to give 14 fractions (FF1-FF14). Fraction FF7 was separated by CC eluting with a gradient of acetone-hexane to give 10 subfractions (FF7A-FF7J). Subfraction FF7G was separated by CC and eluted with 20% acetone-hexane to give 7 subfractions (FF7G1-FF7G7). Subfraction FF7G3 was further purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give **CP25** (1.2 mg). Fraction FF10 was separated by QCC eluting with a gradient of acetone-hexane to give 17 subfractions (FF10A-FF10Q). Subfractions FF10N and FF10O were separated by CC and eluted with a gradient of EtOAc-hexane to give 8 subfractions (FF10N1-FF10N8). Subfractions FF10N1 was further purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give **CP22** (1.2 mg). Subfraction FF10N2 was separated by CC and eluted with $CHCl_3$ to give **CP21** (28.0 mg) and **CP24** (1.5 mg). Subfraction FF10N6 was separated by CC and eluted with $CHCl_3$ to give **CP20** (5.3 mg) and **CP26** (15.2 mg). Fractions FF12 and FF13 were separated by QCC eluting with a gradient of acetone-hexane to give 13 subfractions (FF12A-FF12J). Subfraction FF12H was separated by CC and eluted with a gradient of acetone-hexane to give 10 subfractions (FF12H1-FF12H10). Subfractions FF12H4 and FF12H5 were further purified by CC eluting with a gradient of EtOAc-hexane to give **CP23** (3.7 mg). Subfraction FF12I was further separated by CC eluting with a gradient of acetone-hexane to give **CP27** (3.0 mg) (see **Scheme 8**).



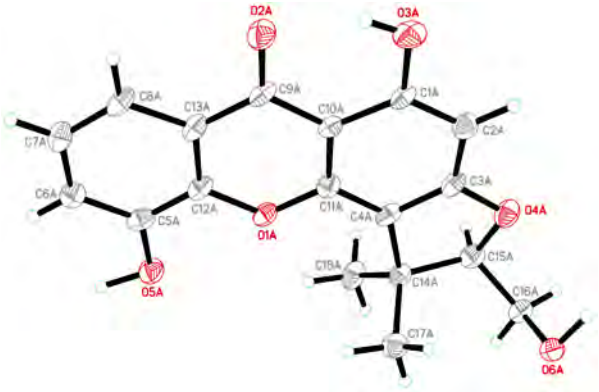
Scheme 8 Isolation of compounds **CP20-CP27**

Characterization of isolated compounds

20. *Compound* CP20



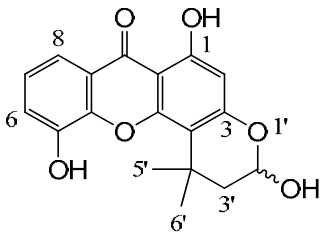
The structure of compound CP**20**



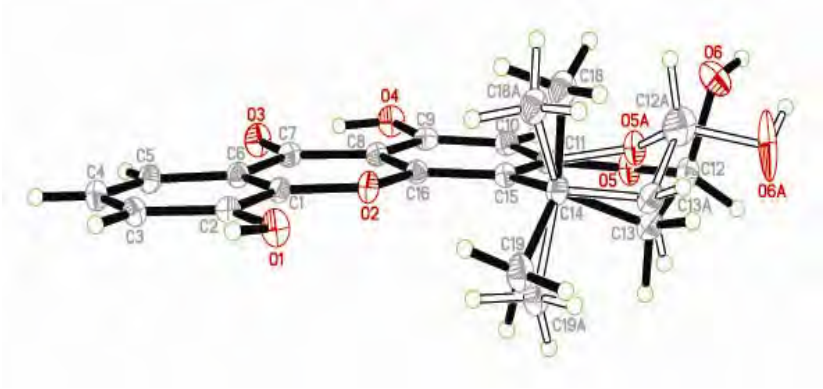
The X-ray structure of compound CP**20**

***Compound* CP20**: *Pruniflorone M*. Yellow single crystal, mp 235-237 °C; $[\alpha]^{25}_{D}$ = +64.6 (*c* 0.04, $CHCl_3$); UV-Vis ($CHCl_3$) λ_{max} (log ε) 246 (4.45), 257 (4.34), 315 (4.14), 357 (3.58) nm; FT-IR (neat) ν_{max} 3368, 1648, 1587 cm^{-1}; HRMS *m/z* 328.0947 for $C_{18}H_{16}O_6$ (calcd. 328.0947). EIMS *m/z* (rel. int.): 328 $[M]^+$ (38), 313 (93), 283 (100), 255(25), 141 (5). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 3.20**. (Boonnak *et al*., 2010)

21. *Compound* CP21

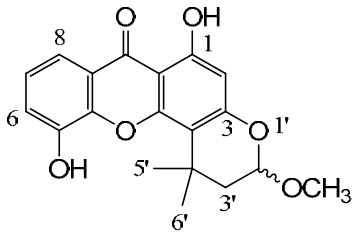


The structure of compound CP**21**



The X-ray structure of compound CP**21**

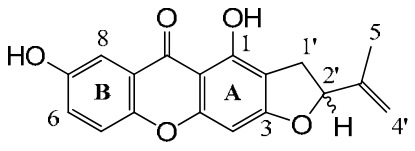
***Compound* CP21**: *Pruniflorone N*. Yellow powder, mp 250-252 °C; $[\alpha]^{25}_{D}$ = +5.2 (*c* 0.42, acetone); UV-Vis ($CHCl_3$) λ_{max} (log ε) 246 (4.32), 259 (4.21), 316 (4.02), 356 (3.39) nm; FR-IR (neat) ν_{max} 3411, 1651, 1622, 1578 cm^{-1}; HRMS *m/z* 328.0948 for $C_{18}H_{16}O_6$ (calcd. 328.0947). EIMS *m/z* (rel. int.): 328 $[M]^+$ (40), 313 (100), 285 (31), 257 (16), 243 (12), 149 (6). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 3.21**. (Boonnak *et al*., 2010)



The structure of compound CP**21a**

Compound* CP21a**: *Hydrolysis of **CP21. A solution of **CP21** (7.8 mg) in 20% HCl–CH_3OH (2.0 mL) was left to stand for 4 days at room temperature. The solution was evaporated in vacuum to give a residue, which was purified by CC on silica gel and eluted with 25% acetone-hexane to give compounds **CP21** (3.5 mg) and **CP21a** (3.5 mg). Compound **CP21a** was yellow powder. mp 208-210 °C; $[\alpha]^{28}_{D}$ = +40.8 (*c* 0.18, acetone). HRMS *m/z* 342.1091 for $C_{19}H_{18}O_6$ (calcd. 342.1103). EIMS *m/z* (rel. int.): 342 $[M]^+$ (42), 327 (100), 295 (68), 259 (7), 83 (4). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 3.21a**. (Boonnak *et al*., 2010)

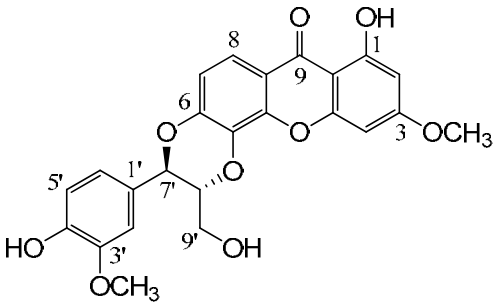
22. *Compound* CP22



The structure of compound CP**22**

***Compound* CP22**: *Pruniflorone O.* Yellow viscous oil, $[\alpha]^{26}_{D}$ = +15.1 (*c* 0.04, acetone); UV-Vis ($CHCl_3$) λ_{max} (log ε) 243 (4.38), 269 (4.07), 280 (3.99), 315 (3.78), 352 (3.61) nm; Ft-IR (neat) ν_{max} 3378, 1630 cm^{-1}; HRMS *m/z* 310.0845 for $C_{18}H_{14}O_5$ (calcd. 310.0841). EIMS *m/z* (rel. int.): 310 $[M]^+$ (3), 295 (4), 257 (100), 229 (4). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 3.22**. (Boonnak *et al*., 2010)

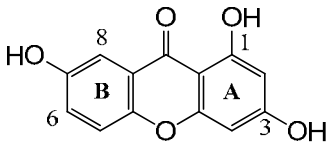
23. *Compound* CP23



The structure of compound CP**23**

***Compound* CP23**: *3-Methoxy-5′-demethoxycadensin G.* Yellow powder, $[\alpha]^{26}_{D}$ = +53.4 (*c* 0.06, acetone); UV ($CHCl_3$) λ_{max} (log ε) 253 (4.37), 281 (3.83), 318 (3.97) nm; IR (neat) ν_{max} 3431, 1646 cm^{-1}; HRMS *m/z* 452.1119 for $C_{24}H_{20}O_9$ (calcd. 452.1107). EIMS *m/z* (rel. int.): 452 $[M]^+$ (100), 434 (2), 420 (13), 393 (12), 315 (18), 285 (17), 274 (35), 245 (23), 180 (73), 162 (15), 137 (66), 124 (40), 119 (13), 101 (13), 77 (5). For ^1H and ^{13}C NMR spectroscopic data, see **Table 3.23.** (Boonnak *et al*., 2010)

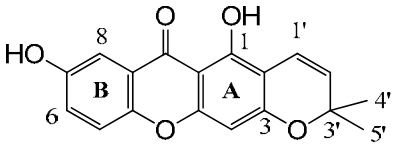
24. *Compound* CP24



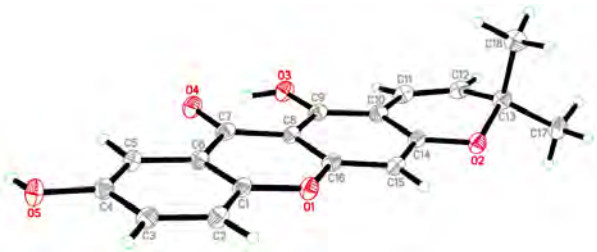
The structure of compound CP**24**

***Compound* CP24**: *1,3,7-Trihydroxyxanthone.* Yellow powder, mp = 318-319 °C. For ^{1}H NMR spectroscopic data, see **Table 3.24**. (Noro *et al*., 1984; Mondal *et al*., 2006)

25. *Compound* CP25



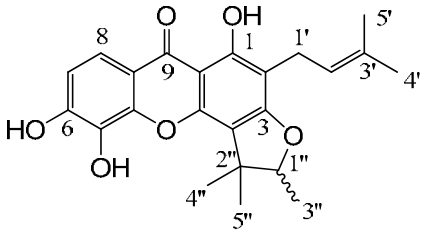
The structure of compound CP**25**



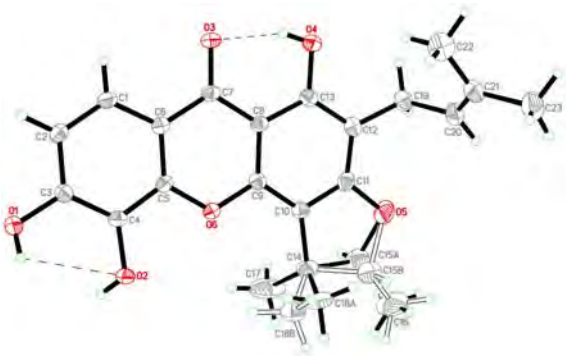
The X-ray structure of compound CP**25**

***Compound* CP25**: *Osajaxanthone.* Yellow powder, mp = 266-268 °C. (Mondal *et al*., 2006)

26. *Compound* CP26



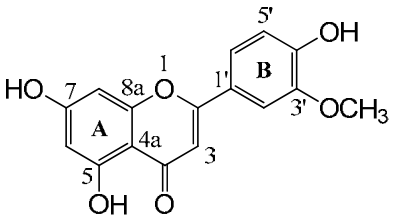
The structure of compound CP**26**



The X-ray structure of compound CP**26**

***Compound* CP26**: *Formoxanthone C*. Yellow solid, mp 152-154 $^{\circ}$C; $[\alpha]^{25}_{D}$ = -44.8 (*c* 0.05, $CHCl_3$); UV-Vis (CH_3OH) λ_{max} (log ε) 258 (4.51), 276 (4.44), 392 (3.85) nm; FT-IR (KBr) ν_{max} 3440, 1646, 1624 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CD_3OD+$CDCl_3$) spectroscopic data see **Table 3.26**. (Boonsri *et al*., 2006)

27. *Compound* CP27



The structure of compound CP**27**

***Compound* CP27**: *Chrysoeriol.* Pale-yellow solid. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 3.27**. (Wagner *et al*., 1976; Nakasuki *et al*., 2006)

Table 3.20 NMR spectroscopic data of **CP20** in d_6-acetone

Position	**Type of C**	$\delta_H{}^a$ **(*J* in Herz)**	$\delta_C{}^b$	**HMBC (^{1}H→^{13}C)**
1-OH	C	13.27, *s*	164.4	C-1, C-2, C-3, C-9a
2	CH	6.21, *s*	93.2	C-1, C-3, C-4, C-4a, C-9a
3	C		166.4	
4	C		113.1	
5	C		146.3	
6	CH	7.39, *dd*, 7.8, 1.8	120.4	C-5, C-8, C-4b
7	CH	7.26, *t*, 7.8	123.9	C-5, C-8, C-4b, C-8a
8	CH	7.68, *dd*, 7.8, 1.5	115.4	C-6, C-9, C-4b
9	C=O		180.7	
4a	C		152.6	
4b	C		145.2	
8a	C		121.5	
9a	C		103.4	
1′	CH_2	3.93, *d*, 5.7	60.4	C-2′, C-3′
2′	CH	4.54, *t*, 5.7	94.8	C-3, C-4, C-1′, C-3′, C-4′, C-5′
3′	C		43.1	
4′	CH_3	1.48, *s*	20.2	C-4, C-1′, C-2′, C-3′, C-5′
5′	CH_3	1.71, *s*	26.3	C-4, C-2′, C-3′, C-4′

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.21 NMR spectroscopic data of **CP21** in d_6-acetone

Position	**Type of C**	$\delta_H{}^a$ (***J*** **in Herz)**	$\delta_C{}^b$	**HMBC (^{1}H→^{13}C)**
1-OH	C	12.82, *s*	161.1	C-1, C-2, C-3, C-9a
2	CH	6.03, *s*	99.0	C-1, C-4, C-9, C-4a, C-9a, C-4′
3	C		160.7	
4	C		109.6	
5	C		146.8	
6	CH	7.26, *br d*, 7.5	119.9	C-5, C-8, C-4b
7	CH	7.10, *t*, 7.8	123.9	C-5, C-6, C-8, C-4b, C-8a
8	CH	7.49, *d*, 8.1	114.8	C-5, C-6, C-7, C-9, C-4b
9	C=O		181.2	
4a	C		155.7	
4b	C		145.3	
8a	C		121.2	
9a	C		104.1	
1′	-	-	-	-
2′	CH	5.39, *dd*, 7.8, 2.1	93.1	C-3, C-3′, C-4′
3′	CH_2	1.87, *dd*, 13.8, 2.1	45.9	C-4, C-2′, C-4′, C-5′, C-6′
		1.78, *dd*, 13.8, 7.8		C-4, C-2′, C-4′
4′	C		31.9	
5′	CH_3	1.59, *s*	28.1	C-4, C-2′, C-4′, C-6′
6′	CH_3	1.48, *s*	28.0	C-4, C-4′, C-6′

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.21a NMR spectroscopic data of **CP21a** in d_6-acetone

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC (^{1}H→^{13}C)**
1-OH	C	12.84, *s*	161.2	C-1, C-2, C-9a
2	CH	6.12, *s*	99.2	C-1, C-3, C-4, C-9a
3	C		159.6	
4	C		110.0	
5	C		146.5	
6	CH	7.28, *dd*, 7.8, 1.2	120.0	C-5, C-8, C-4b
7	CH	7.14, *t*, 7.8	124.0	C-5, C-8a
8	CH	7.55, *d*, 7.8, 1.5	115.1	C-6, C-9, C-4b
9	C=O		181.3	
4a	C		155.7	
4b	C		145.4	
8a	C		121.3	
9a	C		104.4	
1′	-	-	-	-
2′	CH	5.09, *dd*, 6.0, 3.0	99.6	C-3, C-3′, C-4′, 2′-OCH_3
3′	CH_2	1.89, *dd*, 13.8, 6.3	43.8	C-4, C-2′, C-4′, C-5′, C-6′
		1.82, *dd*, 13.8, 2.7		C-4, C-2′, C-4′, C-5′, C-6′
4′	C		31.1	
5′	CH_3	1.59, *s*	28.0	C-4, C-3′, C-4′, C-6′
6′	CH_3	1.51, *s*	28.4	C-4, C-3′, C-4′, C-5′
2′-OCH_3	CH_3	3.41, *s*	55.6	C-2′

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.22 NMR spectroscopic data of **CP22** in d_6-acetone

Position	**Type of C**	δ_H[a] (***J*** **in Herz)**	δ_C[b]	**HMBC (^{1}H→^{13}C)**
1-OH	C	13.42, *s*	160.9	C-1, C-2, C-9a
2	C		108.3	
3	C		165.6	
4	CH	6.44, *s*	94.1	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.42, *d*, 8.7	118.7	C-7, C-4b, C-8a
6	CH	7.33, *dd*, 8.7, 3.0	124.1	C-7, C-4b
7	C		154.1	
8	CH	7.57, *d*, 3.0	108.4	C-6, C-7, C-9, C-4b
9	C=O		180.2	
4a	C		156.4	
4b	C		149.7	
8a	C		121.0	
9a	C		103.0	
1′	CH_2	3.05, *dd*, 14.1, 2.4	29.0	C-1, C-2, C-3, C-3′
		2.92, *dd*, 14.1, 7.5		C-1, C-2, C-3, C-3′
2′	CH	4.42, *m*	75.3	C-2, C-4′
3′	C		147.6	
4′	CH_2	4.89, *br s*	109.4	C-3′, C-5′
		4.72, *br s*		C-3′, C-5′
5′	CH_3	1.83, *s*	17.3	C-3′, C-4′

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

Table 3.23 NMR spectroscopic data of **CP23** in d_6-acetone

Position	**Type of C**	δ_H[a] **(*J* in Herz)**	δ_C[b]	**HMBC (^{1}H→^{13}C)**
1-OH	C	-	163.3	-
2	CH	6.28, *d*, 2.4	97.5	C-1, C-3, C-4, C-9a
3	C		166.7	
4	CH	6.46, *d*, 2.4	93.2	C-2, C-3, C-4a, C-9a
5	C		131.9	
6	C		146.5	
7	CH	6.92, *d*, 8.8	114.2	C-5, C-6, C-8a
8	CH	7.67, *d*, 8.8	117.8	C-6, C-8, C-9, C-4b
9	C=O		180.6	
4a	C		157.9	
4b	C		149.5	
8a	C		115.3	
9a	C		103.6	
1′	C		127.1	
2′	CH	6.89, *d*, 1.6	110.5	C-4′, C-6′, C-7′
3′	C		147.9	
4′	C		147.2	
5′	CH	6.86, *d*, 8.8	115.5	C-1′, C-3′
6′	CH	6.89, *dd*, 8.8, 1.6	120.9	C-2′, C-4′, C-7′
7′	CH	5.06, *d*, 8.0	77.2	C-1′, C-2′, C-6′, C-8′
8′	CH	4.07, *ddd*, 8.0, 3.6, 2.8	79.0	-
9′	CH_2	3.89, *m*	61.0	C-8′
		3.54, *dd*, 12.8, 3.6		-
3-OCH_3	CH_3	3.83, *s*	56.0	C-3
3′-OCH_3	CH	3.85, *s*	56.2	C-3′

[a]Recorded in 400 MHz.

[b]Recorded in 100 MHz.

Table 3.24 ^{1}H NMR spectroscopic data of **CP24** in $CD_3OD+CDCl_3$

Position	**CP24**		**1,3,7-trihydroxyxanthone**[b]	
	Type of C	**δ_a[a] (*J* in Herz)**	**δ_C[c]**	**δ_H[d] (*J* in Herz)**
1-OH	C	12.92, *s*	162.7	12.88, *s*
2	CH	6.25, *d*, 2.1	98.0	6.18, *d*, 2.1
3	C		163.0	
4	CH	6.37, *d*, 2.1	93.9	6.35, *d*, 1.9
5	CH	7.33, *d*, 9.0	119.1	7.45, *d*, 9.0
6	CH	7.25, *dd*, 9.0, 2.7	124.6	7.27, *dd*, 9.0, 2.9
7	C		154.1	
8	CH	7.51, *d*, 2.7	108.2	7.40, *d*, 2.7
9	C=O		179.9	
4a	C		157.7	
4b	C		149.2	
8a	C		120.6	
9a	C		102.1	

[a] Recorded at 300 MHz in $CDCl_3+CD_3OD$

[b] It was previously reported by Mondal *et al*., 2006.

[c] Recorded at 200 MHz in DMSO

[d] Recorded at 50 MHz in DMSO

Table 3.26 NMR spectroscopic data of **CP26** in $CD_3OD+CDCl_3$

Position	**Type of C**	$\delta_H{}^a$ **(*J* in Herz)**	$\delta_C{}^b$	**HMBC ($^1H \rightarrow {}^{13}C$)**
1-OH	C	13.40, *s*	164.5	C-1, C-2, C-9a
2	C		110.6	
3	C		168.0	
4	C		115.9	
5	C		135.9	
6	C		149.7	
7	CH	6.85, *br s*	115.9	C-5, C-8a
8	CH	7.63, *d*, 8.1	121.1	C-6, C-9, C-4b
9	C		184.5	
4a	C		154.9	
4b	C		154.1	
8a	C		118.2	
9a	C		106.8	
1′	CH_2	3.29, *d*, 7.2	25.6	C-1, C-2, C-3, C-2′, C-3′, C-5′
2′	CH	5.28, *br t*, 7.2	125.6	C-2, C-1′, C-4′, C-5′
3′	C		135.9	
4′	CH_3	1.68, *s*	29.6	C-2′, C-3′
5′	CH_3	1.77, *s*	21.6	C-2′, C-3′
1″	CH	4.52, *q*, 6.6	44.4	C-3, C-4, C-2″, C-4″, C-5″
2″	C		47.9	
3″	CH_3	1.41, *d*, 6.6	18.2	C-1″, C-2″
4″	CH_3	1.31, *s*	25.1	C-4, C-1″, C-2″
5″	CH_3	1.59, *s*	29.6	C-4, C-1″, C-2″

[a]Recorded in 300 MHz.; [b]Recorded in 75 MHz.

Table 3.27 NMR spectroscopic data of **CP27** in d_6-acetone

Position	**Type of C**	$\delta_H{}^a$ **(*J* in Herz)**	$\delta_C{}^b$	**HMBC (^{1}H→^{13}C)**
1	-		-	
2	C		164.2	
3	CH	6.71, *s*	103.5	C-2, C-4, C-4a, C-1′
4	C=O		182.2	
5-OH	C	13.08, *s*	162.4	C-5, C-6, C-7, C-4a
6	CH	6.26, *d*, 1.8	98.9	
7	C		164.2	C-5, C-7, C-8, C-4b
8	CH	6.56, *d*, 1.8	93.9	C-6, C-7, C-4b, C-8a
4a	C		104.4	
8a	C		157.9	
1′	C		122.7	
2′	CH	7.64, *brs*	109.7	C-2, C-3′, C-4′, C-6′
3′	C		148.1	
4′	C		150.7	
5′	CH	7.02, *d*, 8.4	115.5	C-1′, C-3′, C-4′
6′	CH	7.62, *dd*, 8.4, 2.1	120.5	C-2, C-2′, C-4′
3′-OCH_3	CH_3	4.00, *s*	55.7	C-3′

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

3.5 การทดสอบการออกฤทธิ์ทางชีวภาพของสาร CP1-CP27

3.5.1 การทดสอบการออกฤทธิ์ต้านเชื้อแบคทีเรีย

Only sufficient quantity isolated compounds were further tested against both Gram positive and Gram negative bacteria: *Bacillus subtilis*, *Staphylococcus aureus,* TISTR517, *Enterococcus faecalis* TISTR459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC43300, Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299, *Streptococcus faecalis*, *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*. The microrganisms were obtained from the culture collections, Department of Industrial Biotechnology and Department of Pharmacognosy and Botany, PSU, except for the TISTR and ATCC strains, which were obtained from Microbial Research Center (MIRCEN), Bangkok, Thailand. The antibacterial assay employed was the same as described in Boonsri (Boonsri *et al*., 2006). Vancomycin, which was used as a standard, showed antibacterial activity against Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299 at 75.0 μg/mL.

3.5.2 การทดสอบการออกฤทธิ์ต้านเชื้อรา

Candida albicans was obtained from Department of Pharmacognosy and Botany, PSU. The antifungal assay employed was the same as described in Boonsri and co-worker (Boonsri *et al*., 2006) by using amphotencin B as a positive control.

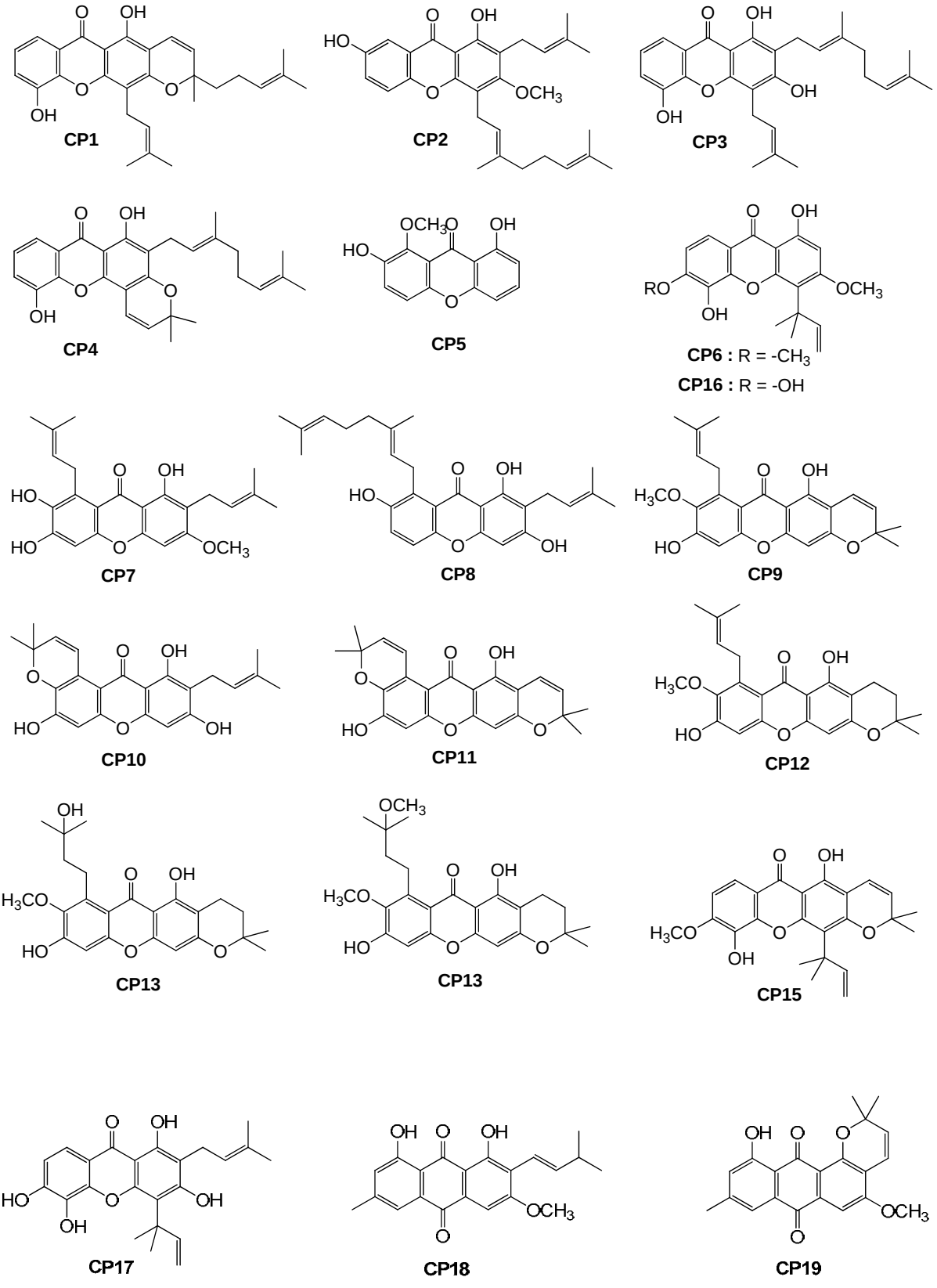
3.5.3 การทดสอบการออกฤทธิ์ต้านการอักเสบ (Nitric oxide inhibitory activity assay)

Inhibitory effect on NO production by murine macrophage-like RAW264.7 cells was evaluated using a modified method from that previously reported (Tewtrakul *et al*., 2009). Briefly, the RAW264.7 cell line (purchased from Cell Lines Services) was cultured in RPMI medium supplemented with 0.1% sodium bicarbonate and 2 mM glutamine, penicillin G (100 units/mL), streptomycin (100 μg/mL) and 10% FCS. The cells were harvested with trypsin-EDTA and diluted to a suspension in a fresh medium. The cells were seeded in 96-well

plates with 1×10^5 cells/well and allowed to adhere for 1 h at 37°C in a humidified atmosphere containing 5% CO_2. After that the medium was replaced with a fresh medium containing 50 μg/mL of LPS together with the test samples at various concentrations (3-100 μg/mL for crude extract and 3-100 μM for pure compounds) and was then incubated for 24 h. NO production was determined by measuring the accumulation of nitrite in the culture supernatant using the Griess reagent. Cytotoxicity was determined using the MTT colorimetric method. Briefly, after 24 h incubation with the test samples, MTT solution (10 μL, 5 mg/mL in PBS) was added to the wells. After 4 h incubation, the medium was removed, and isopropanol containing 0.04 M HCl was then added to dissolve the formazan production in the cells. The optical density of the formazan solution was measured with a microplate reader at 570 nm. The test compounds were considered to be cytotoxic when the optical density of the sample-treated group was less than 80% of that in the control (vehicle-treated) group. L-NA, CAPE and indomethacin were used as positive controls. The stock solution of each test sample was dissolved in DMSO, and the solution was added to the medium RPMI (final DMSO is 1%). Inhibition (%) was calculated using the following equation and IC_{50} values were determined graphically (n = 4):

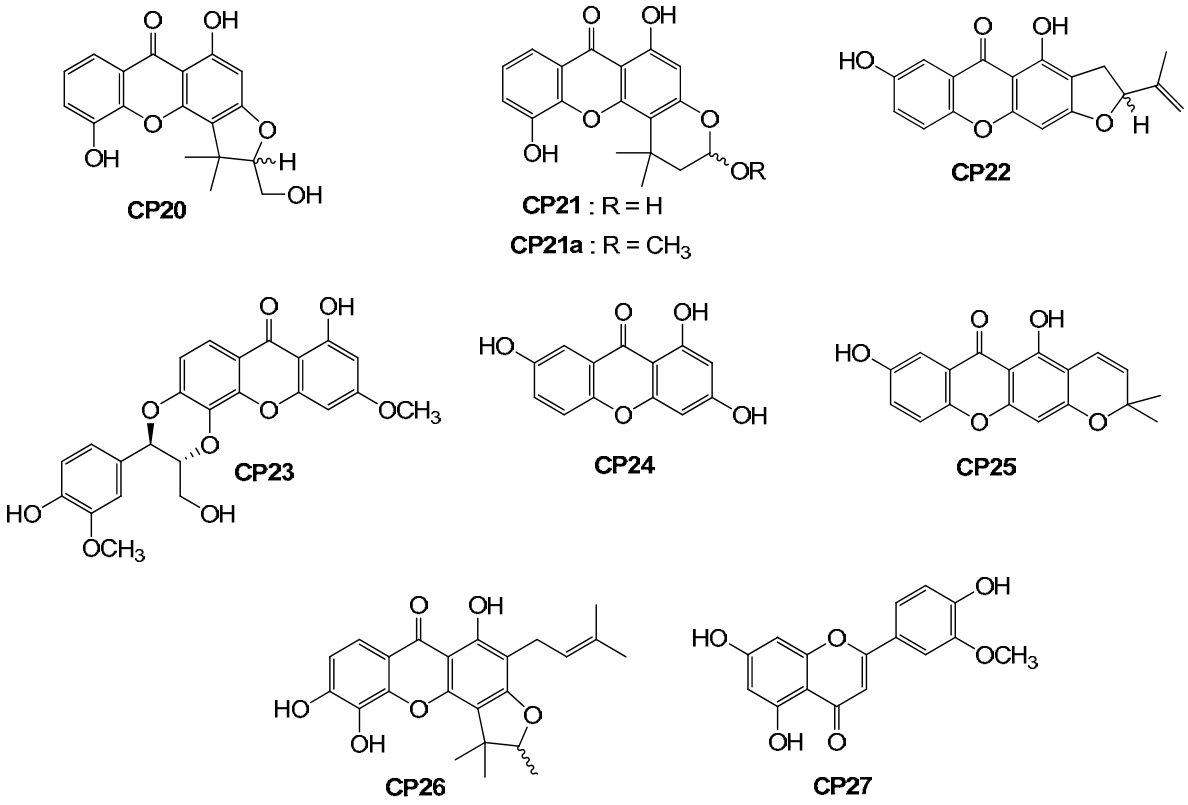
$$\text{Inhibition (\%)} = \frac{A - B}{A - C} \times 100$$

A-*C* : NO_2^- concentration (μM) [*A* : LPS (+), sample (-); *B* : LPS (+), sample(+); *C* : LPS (-), sample (-)].



The structures of **CP1-CP19**

The isolated compounds the roots of *C. formosum* spp. *pruniflorum*



The structures of **CP20-CP27**

The isolated compounds the green fruits of *C. formosum* spp. *pruniflorum*

ผลการทดสอบการออกฤทธิ์ต้านเชื้อแบคทีเรียและเชื้อราของสาร **CP1-CP27** แสดงดัง Table 4 และผลต้านการอักเสบแสดงดัง Table 5

Table 4 Antimicrobial activity of compounds **CP1-CP3, CP5-CP14**, **CP17**, **CP18**, **CP21**, **CP21a**, **CP23**, **CP26** and **CP27**

No	Antibacterial activity								Antifungal activity
	Gram-positive bacteria[a]					Gram-positive bacteria[b]			*C. albicans*[c]
	BS	*SA*	*EF*	*MRSA*	*VRE*	*ST*	*SS*	*PA*	
CP1	300	>300	>300	>300	>300	>300	>300	>300	>300
CP2	>300	>300	>300	>300	>300	>300	>300	>300	>300
CP3	18.7	37.5	-	NT[d]	NT[d]	-	-	-	NT[d]
CP5	37.5	37.5	300	75	300	>300	>300	300	300
CP6	300	300	300	300	300	>300	>300	>300	>300
CP7	18.7	18.7	300	9.37	9.37	18.7	>300	300	>300
CP8	4.67	9.37	9.37	18.7	75	>300	>300	300	300
CP9	18.7	18.7	75	37.5	75	>300	>300	300	300
CP10	75	75	75	150	75	>300	>300	>300	>300
CP10: CC10[e]	4.67	2.34	9.37	2.34	4.67	>300	>300	>300	300
CP11	300	300	>300	300	300	>300	>300	>300	>300
CP12	-	-	-	NT[d]	NT[d]	-	-	-	NT[d]
CP13	9.37	<1.1	-	NT[d]	NT[d]	-	-	-	NT[d]
CP14	-	-	-	NT[d]	NT[d]	-	-	-	NT[d]
CP17	<1.1	<1.1	4.67	NT[d]	NT[d]	37.5	<1.1	<1.1	NT[d]
CP18	-	-	-	NT[d]	NT[d]	-	-	-	NT[d]
CP20	>300	>300	>300	>300	>300	>300	>300	>300	>300
CP21	300	>300	>300	9.37	300	150	>300	>300	>300
CP21a	18.7	300	300	37.5	75	300	300	37.5	300
CP23	>300	>300	>300	>300	>300	>300	>300	>300	>300
CP26	4.67	9.37	4.67	4.67	4.67	4.67	>300	37.5	>300
CP27	>300	>300	>300	37.5	37.5	300	>300	>300	>300

[a] *Bacillus subtilis, Staphylococcus aureus, Enterococcus faecalis* TISTR 459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC 43300, Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299.; [b] *Salmonella typhi, Shigella sonnei* and *Pseudomonas aeruginosa*.;
[c] *Candida albicans*; [d] NT = not tested; [e] a mixture in 1:1 ratio

From the antibacterial results (**Table 3**), it showed that 1,3,7-trihydroxyxanthone with the isoprenyl or geranyl side chain at C-2 and C-4 is essential for its antibacterial activity against *P. aeruginosa*, we can further added that the hydroxyl group at C-3 is also essential for the activity as compared pruniflorone L (**CP2**, MIC > 300 µg/mL) with cochinchinone A (**CC3**, MIC 4.7 µg/mL) (**Table 3**). During the chromatographic separation, we have observed that compounds **CP10** and **CC10** came as a mixture. Therefore, these two compounds were tested as a mixture for anti-microbial activity whose result was good. This prompted us to purify the mixture of which was successfully separated by reversed phase RP-18 CC eluting with MeOH. We then tested each of the compounds **CP10**, **CC10** and their 1:1 mixture. Interestingly, the 1:1 mixture of compounds **CP10** and **CC10** significantly increased the antibacterial activities against *B. subtilis*, *S. aureus*, *E. faecalis*, MRSA and VRE compared with the pure forms (**CP10** and **CC10**) (**Table 4**). From this result, it may be possible that a 1:1 mixture of compounds **CP10** and **CC10** showed synergistic effect for antibacterial activity against all Gram-positive bacteria tested.

Moreover, compounds **CP17** and **CP26** exhibited strong antibacterial activity against both Gram-positive and Gram-negative (*S. typhi*) bacteria with MIC value of <1.1 and 4.67 µg/mL, whereas compound **CP21** showed moderate antibacterial activity specifically against MRSA with MIC value of 9.37 µg/mL (**Table 4**). The methylated product **CP21a** was not significant for antibacterial activity but showed better activity against *B. subtilis* when compared with compound **CP21**.

Only isolated compounds from the green fruits of *C. formosum* spp. *pruniflorum* with sufficient amount were further evaluated for their nitric oxide inhibitory activity using RAW264.7 cells (Boonnak *et al*., 2010). As shown in **Table 5**, compounds **CP21** and **CP26** possessed potent NO inhibitory activity against lipopolysaccharide (LPS)-induced nitric oxide release with IC_{50} values of 4.4 and 4.3 µM, respectively better than that of the positive control, indomethacin, which is a non-steroidal anti-inflammatory drug (IC_{50} = 20.1 µM). Compound **CP20** (IC_{50} = 20.6 µM) exhibited moderate NO inhibitory activity when compared with that of compound **CP21** (IC_{50} = 4.4 µM), whereas compound **CP23** was inactive (IC_{50} >100 µM). From the result, it might be proposed that the pyran ring of 1,3,5-trioxygenatedxanthone (**CP21**) was essential for NO inhibitory activity than the furan ring (**CP20**), whereas catechol unit of compound **CP26** might play an important role for NO inhibition. Moreover, compound **CP21** has a part of hemiacetal

moiety in its pyran ring, which would be possible to open to an aldehyde side chain in acidic condition. The experiment has been set up in 20% HCl-CH_3OH. It was found that an aldehyde product was not observed, instead the methylated product **CP21a** was formed. Compound **CP21a** was further tested for NO inhibition which showed less NO inhibitory activity than **CP21** with an IC_{50} value of 9.5 μM. From this result, it could be suggested that the methoxyl group might decrease the NO inhibitory activity (Boonnak *et al*., 2010).

Table 5 Nitric oxide inhibitory activity of compounds **CP20**, **CP21**, **CP21a**, **CP23** and **CP26**

No	% Inhibition of various concentrations [μM]					IC_{50} [μM]
	0	3	10	30	100	
CP20	0.0±4.5	15.5±0.9**	29.6±0.9**	44.0±1.6**	95.3±0.6[a]**	20.6
CP21	0.0±4.5	34.8±1.4*	72.7±1.1**	93.1±1.7**	99.5±0.6[a]**	4.4
CP21a	0.0±2.4	15.7±5.3	44.4±1.9**	91.0±1.0**	101.3±3.0[a]**	9.5
CP23	0.0±5.4	–	21.7±1.0*	23.5±2.0**	47.4±3.1**	>100
CP26	0.0±5.4	30.1±1.6**	94.3±0.7[a]**	102.1±2.4[a]**	102.9±0.7[a]**	4.3
Indomethacin	0.0±4.2	16.6±2.9	32.7±2.6**	53.4±3.0**	85.6±1.8**	20.1
L-NA	0.0±5.6	15.3±2.8	21.4±2.5	35.6±2.1**	73.2±3.5**	59.0
CAPE	0.0±5.6	35.2±3.0*	70.3±2.7**	97.6±2.4[a]**	99.5±2.7[a]**	5.0

[a] Cytotoxic effect was observed.
[b] Each value represents mean ± S.E.M. of four determinations.
Statistical significance, *p<0.05, **p<0.01

เอกสารอ้างอิง

Ahad, A. M.; Goto, Y.; Kiuchi, F.; Tsuda, Y.; Kondo, K. and Sato, T. (1991). *Chem. Pharm. Bull*. 39, 1043-1046.

Boonnak, N.; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Fun, H-K. Kanjana-Opas, A.; Laphookhieo, S. (2006). *Tetrahedron*, *62*, 8850–8859.

Boonnak, N.; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Fun, H.K.; Kanjana-Opas, A.; Chantrapromma, K.; Kato, S. (2009). *Tetrahedron*, 65, 3003-3013.

Boonnak, N.; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Kanjana-Opas, A.; Chantrapromma, K.; Kato, Shigeru. (2010). *Chem. Pharm. Bull*. 58, 386-389.

Boonnak, N.; Khamthip, A.; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Kanjana-Opas, A.; Tewtrakul, S.; Chantrapromma, K.; Fun, H. K.; Kato, S. (2010). *Aust. J. Chem.* 63, 1550-1556.

Boonsri, S.; Karalai, C.; Ponglimanont, C.; Kanjana-Opas, A.; Chantrapromma, K. (2006). *Phytochemstry*, 67, 723-727.

Chang, C.-H.; Lin, C.-C.; Kawata, Y.; Hattori, M.; Namba, T. (1989). *Phytochemistry* 28, 2823-2826.

Chantrapromma, S.; Boonnak, N.; Fun, H.-K.; Karalai, C.; Chantrapromma, K. (2010). *Acta. Cryst.* E66, o2066-o2067.

Deachathai, S.; Mahabusarakam, W.; Phongpaichit, S.; Taylor, W. C. *Phytochemistry* (2005). 66, 2368-2375.

Deachathai, S.; Mahabusarakam, W.; Phongpaichit, S.; Taylor, W. C.; Zhang, Y. J.; Yang, C. R. (2006). *Phytochemistry*, 67, 464–469.

Delle Monache, F.; Ferrari, F.; Marini-Bettolo, G. B.; Maxfield, P.; Cerrini, S.; Fedeli, W.; Gavuzzo, E.; Vaciago, A. (1979). *Gazz. Chim. Ital*. 109, 301-310.

Dutta, P. K.; Sen, A. K.; Sarkar, K. K.; Banerji, N. (1987).
Indian J. Chem., Sect. B: *Org. Chem. Incl. Med. Chem*. *26B*, 281–282.

Goncalves, M. D. L.; Mors, W. B. (1981). *Phytochemistry* 20, 1947-1950.

Gottlieb, O. R.; Magalhaes, M. T.; Stefani, G. M. (1966). *Tetrahedron* 22, 1785-1788.

Gunasekera, S. P. ; Selliah, S. ; Sultanbawa, M. (1975). *U. S. J. Chem. Soc., Perkin Trans. 1*. 1539–1544.

Hay, A.-E.; Merza, J.; Landreau, A.; Litaudon, M.; Pagniez, F.; Pape, P. L.; Richomme, P. (2008). *Fitoterapia* 79, 42-46.

Ilnuma, M.; Tosa, H.; Ito, T.; Tanaka, T.; Madulid, D. A. (1996). *Phytochemistry* 42, 1195-1198.

Kijjoa, A.; Jose, M.; Gonzalez, T. G.; Pinto, M. M. M.; Damas, A. M.; Mondranondra, I.-O.; Silva, A. M. S.; Herz, W. (1998). *Phytochemistry* 49, 2159-2162.

Kobayashi, M. ; Mahmud, T. ; Yoshioka, N.; Shibuya, H. ; Kitagawa, I. (1997)
Chem. Pharm. Bull., 45, 1615–1619.

Laphookhieo, S.; Maneerat, W.; Narmdorkmai, W.; Koysomboon, S. (2009).
Heterocycles 78 1299-1307.

Mahabusarakam, W.; Wiriyachitra, P.; Taylor, W. C. (1987) *J. Nat. Prod*. 50, 474-478.

Mahabusarakam, W.; Nuangnaowarat, W.; Taylor, W. C. (2006). *Phytochemistry* 67, 470-474.

Marques, V. L. L.; Oliveira, F. M. D.; Conserva, L. M.; Brito, R. G. L.; Guilhon, G. M. S. P. (2000). *Phytochemistry* 55, 815-818.

Munekazu, L. ; Hideki, T. ; Toshiyuki, T. ; Tetsuro, I. ; Fujio, A., (1996). TYKYDS Journal, 38, 409-414.

Nguyen. L. H. D.; Harrison, L. J. (1998). *Phytochemistry* 50, 471-476.
Pattanaprateeb, P., Nijsiri, R. & Cordell G. A. (2005). *Planta medica*., 71(2), 181-183.

Sen, A. K.; Sarkar, K. K.; Mazumder, P. C.; Banerji, N.; Uusvuori, R.; Hase, T. A. (1982). *Phytochemistry* 21, 1747-1750.

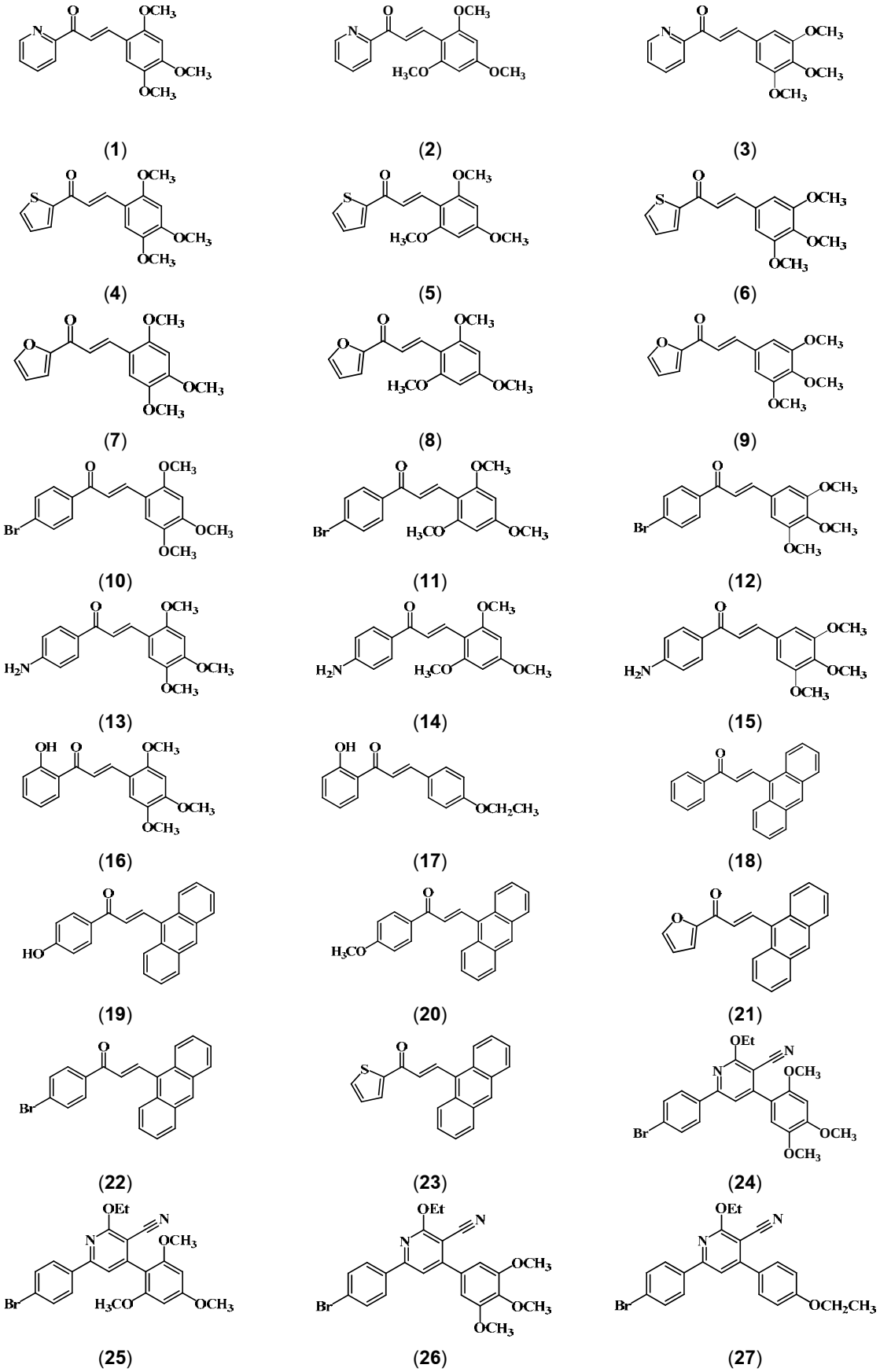
Seo, E. K.; Kim, N. C.; Wani, M. C.; Wall, M. E.; Navarro; H. A.; Burgess, J. P.; Kawanishi, K.; Kardono, L. B. S.; Riswan, S.; Rose, W. C.; Fairchild, C. R.; Farnsworth, N. R; Kinghorn A. D. (2002). *J. Nat. Prod*., 65, 299-305.

Sia, G.-L.; Bennett, G. J.; Harrison, L. J.; Sim, K.Y. (1995). *Phytochemistry* 38, 1521-1528.

Thongdeeying, P. (2005). “Chemical constituents from the leaves of *Ceriops decandra* (Griff.) Ding Hou” Master of Science Thesis in Organic Chemistry, Prince of Songkla University.

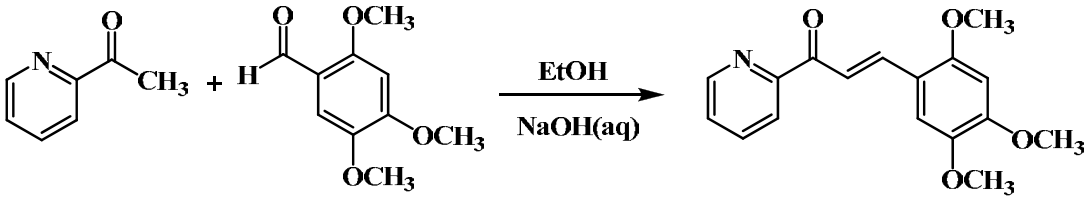
4. ผลการสังเคราะห์และฤทธิ์ทางชีวภาพของสารอนุพันธ์ chalcones

4.1 ทำการสังเคราะห์สารอนุพันธ์ chalcones 23 สาร (**1-23**) เพื่อนำสารที่ได้ไปทดสอบฤทธิ์ทางชีวภาพและสมบัติฟลูออเรสเซนต์ และได้ทำปฏิกิริยาการปิดวง (cyclization) ได้สารอนุพันธ์ nicotinonitrile 4 สาร (**24-27**) แสดงดัง Scheme 4.1



Scheme 4.1 Structures of 23 synthesized chalcones and 4 nicotinonitrile derivatives

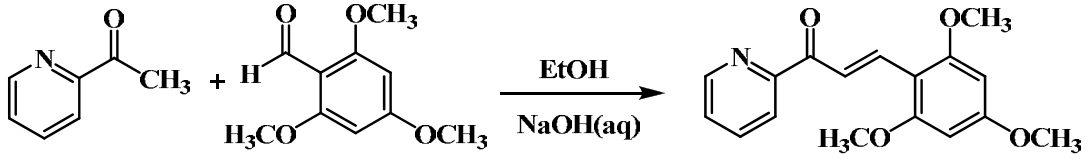
1. (*E*)-1-(pyridinyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one



(**1**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.18 g, 61%) จุดหลอมเหลว 155-156 °C, UV-Vis ($CHCl_3$) λ_{max} (nm): 275.9, 403.1, IR (KBr) ν (cm^{-1}): 3081 (*m*, C-H aromatic stretching), 2937-2960 (*s*, C-H stretching), 1638 (*s*, C=O stretching), 1576-1591 (*s*, C-C stretching), 1272-1317 (*s*, C-N stretching),1194-1208 (*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.76 (1H, *dd*, *J* = 0.6 Hz, *J* = 4.8 Hz), 8.26 (1H, *d*, *J* = 15.9 Hz), 8.14 (1H, *dd*, *J* = 0.9 Hz, *J* = 6.6 Hz), 8.11 (1H, *d*, *J* = 15.9 Hz), 7.92 (1H, *dt*, *J* = 1.8 Hz, *J* = 7.8 Hz), 7.54(2H, *dt*, *J* = 0.9 Hz, *J* = 4.8 Hz), 7.28-6.59 (2H, *s*), 3.95-3.91 (9H, *s*)

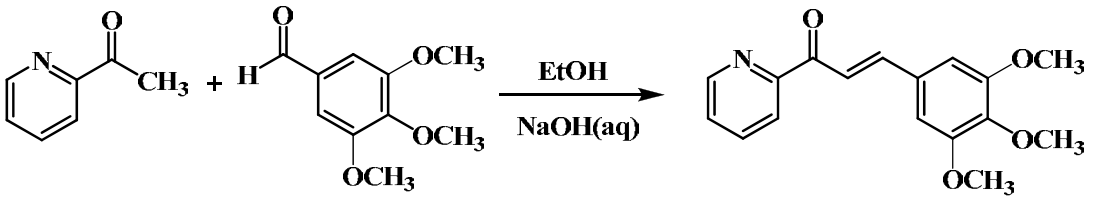
2. (*E*)-1-(pyridinyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one



(**2**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอ่อน (0.21 g, 70%) จุดหลอมเหลว 119-120 °C, UV-Vis ($CHCl_3$) λ_{max} (nm): 248.1, 262.6, 371.8, IR (KBr) ν (cm^{-1}): 2972 (*m*, C-H aromatic stretching), 1669 (*s*, C=O stretching), 1576 (*s*, C=C stretching), 1200 (*s*, C-N stretching), 1123 (*s*, C-O stretching), 810 (*w*, C-H bending), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.73 (1H, *d*, *J* = 4.5 Hz), 8.42-8.31 (2H, *d*, *J* = 16.2 Hz), 8.10 (1H, *d*, *J* = 7.8H), 7.89-7.49 (2H, *dt*, *J* = 1.5 Hz, *J* = 7.8 Hz), 6.18 (2H, *s*), 3.95-3.88 (9H, *s*)

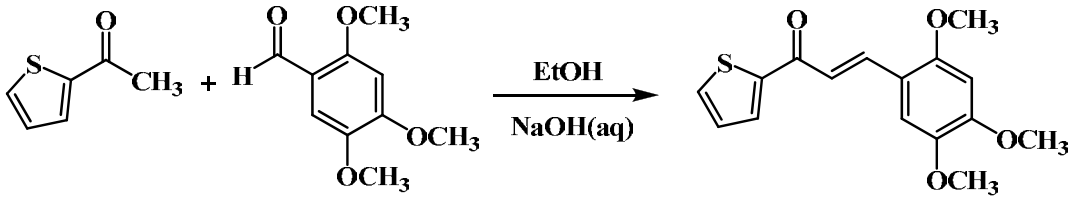
3. (*E*)-1-(pyridinyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one



(**3**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอ่อน (0.20 g, 66%) จุดหลอมเหลว 161-162 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 247.8, 355.4, IR (KBr) ν (cm^{-1}): 2998-2976 (*m*, C-H aromatic stretching), 2943 (*s*, C-H stretching), 1668 (*s*, C=O stretching), 1610-1579 (s, C=C stretching), 1322-1290 (*s*, C-N stretching), 1125(*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.76 (1H, *dd*, *J* = 0.9 Hz, *J* = 4.8 Hz), 8.20 (1H, *dd*, *J* = 0.9 Hz, *J* = 6.9 Hz),8.18 (1H, *d*, *J* = 15.9 Hz), 7.90 (1H, *dt*, *J* = 1.8 Hz, *J* = 7.5 Hz), 7.88 (1H, *d*, *J* = 15.9 Hz),7.50 (1H, *dt*, *J* = 0.9 Hz, *J* = 4.8 Hz), 6.96 (2H, *s*), 3.94-3.91 (9H, *s*)

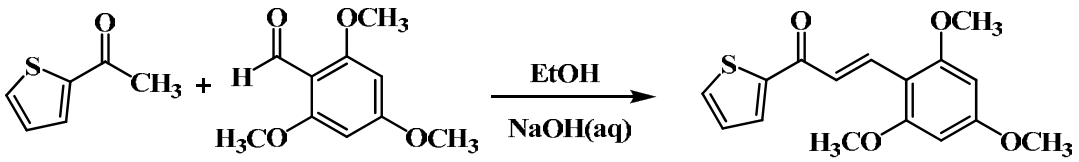
4. (*E*)-1-(thienyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one



(**4**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.23 g, 74%) จุดหลอมเหลว 128-129 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 273.4, 394.9, IR (KBr) ν (cm^{-1}): 3107 (*m*, C-H aromatic stretching), 2932 (*s*, C-H stretching) 1638–1611 (*s*, C=O stretching), 1583–1509 (*s*, C=C stretching), 1206 (*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.12 (1H, *d*, *J* = 15.6 Hz), 7.86 (1H, *dd*, *J* = 0.9 Hz, *J* = 3.9 Hz), 7.65 (1H, *dd*, *J* = 0.9 Hz, *J* = 4.8 Hz), 7.39 (1H, *d*, *J* = 15.6 Hz), 7.17 (1H, *t*, *J* = 3.9 Hz), 7.12-6.53 (2H, *s*), 3.95-3.91 (9H, *s*)

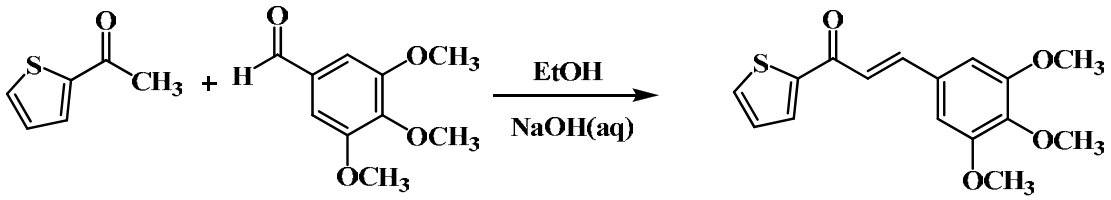
5. (*E*)-1-(thienyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one



(**5**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอ่อน (0.27 g, 89%) จุดหลอมเหลว 108-109 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 266.7, 367.7, IR (KBr) ν (cm^{-1}): 2937 (*m*, C-H aromatic stretching), 1638 (*s*, C=O stretching), 1610–1570 (*s*, C=C stretching), 1204 (*s*, C-O stretching), 1124 (*s*, C-S stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.28 (1H, *d*, *J* = 15.6 Hz), 7.81 (1H, *d*, *J* = 5.1 Hz), 7.79 (1H, *d*, *J* = 15.6 Hz),7.60 (1H, *d*, *J* = 5.1 Hz), 7.15 (1H, *t*, *J* = 5.1 Hz), 6.14 (2H, *s*), 3.92-3.86 (9H, *s*).

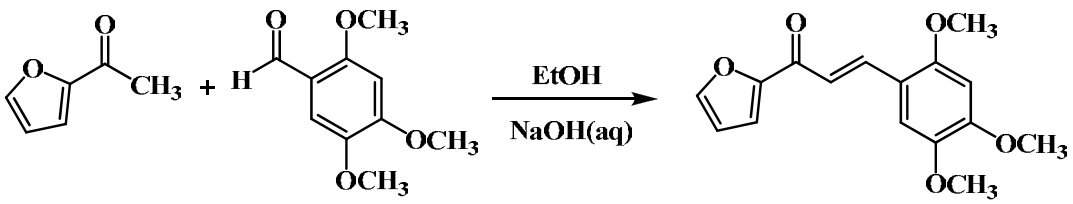
6. (*E*)-1-(thienyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one



(**6**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอ่อน (0.22 g, 72 %) จุดหลอมเหลว 147-148 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 263.6, 349.8, IR (KBr) ν (cm^{-1}): 3086-2975 (*v*, C-H aromatic stretching), 2943 (*s*, C-H stretching), 1646 (*s*, C=O stretching), 1598-1579 (*s*, C=C stretching), 1125 (*s*, C-O stretching), 1004-979 (*s*, C-H stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 7.90 (1H, *dd*, *J* = 0.9 Hz, *J* = 3.9 Hz), 7.79 (1H, *d*, *J* = 15.6 Hz), 7.70 (1H, *dd*, *J* = 0.9 Hz, *J* = 3.9 Hz), 7.32 (1H, *d*, *J* = 15.6 Hz), 7.21 (1H, *t*, *J* = 3.9 Hz), 6.88 (2H, *s*),3.95-3.92 (9H, *s*)

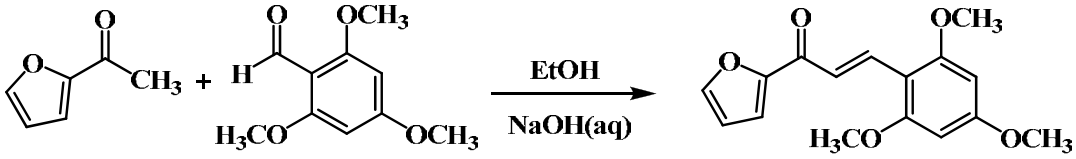
7. (*E*)-1-(furyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one



(**7**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.18 g, 64%) จุดหลอมเหลว 83-84 °C, UV-Vis ($CHCl_3$) λ_{max} (nm): 244.8, 312.3, 396.3, IR (KBr) ν (cm^{-1}): 3404 (*v*, C-H aromatic stretching), 2985 (*s*, C-H stretching), 1642 (*s*, C=O stretching), 1585-1515 (*s*, C=C stretching), 1217 (*s*, C-O stretching), 1040-1026 (*s*, C-H stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.19 (1H, *d*, *J* = 15.6 Hz), 7.64 (1H, *d*, *J* = 3.9 Hz), 7.39 (1H, *d*, *J* = 15.6 Hz), 7.30 (1H, *d*, *J* = 3.9 Hz), 7.14–6.53 (2H, *s*), 6.58 (1H, *t*, *J* = 3.9 Hz), 3.95-3.92 (9H, *s*)

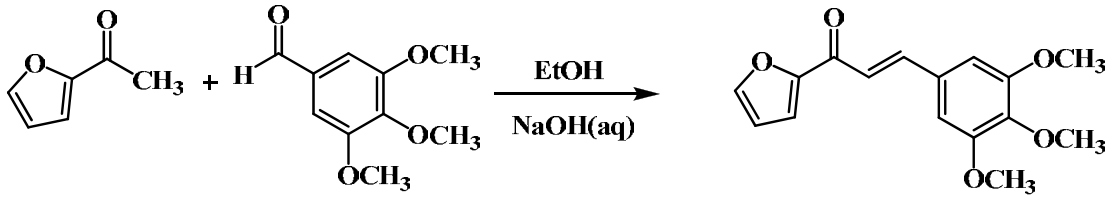
8. (*E*)-1-(furyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one



(**8**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอ่อน (0.22 g, 76%) จุดหลอมเหลว 117-118 °C, UV-Vis ($CHCl_3$) λ_{max} (nm): 260.5, 365.0, IR (KBr) ν (cm^{-1}): 3001–2977 (*v*, C-H aromatic stretching), 2946 (*s*, C-H stretching), 1654 (*s*, C=O stretching), 1582 (*s*, C=C stretching), 1123 (*s*, C-O stretching), 1064-1011 (*s*, C-H stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.24 (1H, *d*, *J* = 15.6 Hz), 7.74 (1H, *d*, *J* = 15.9 Hz), 7.67 (1H, *d*, *J* = 3.9 Hz), 7.25 (1H, *d*, *J* = 3.9 Hz), 6.60 (1H, *t*, *J* = 1.8Hz), 6.17 (2H, *s*), 3.94-3.88 (9H, *s*)

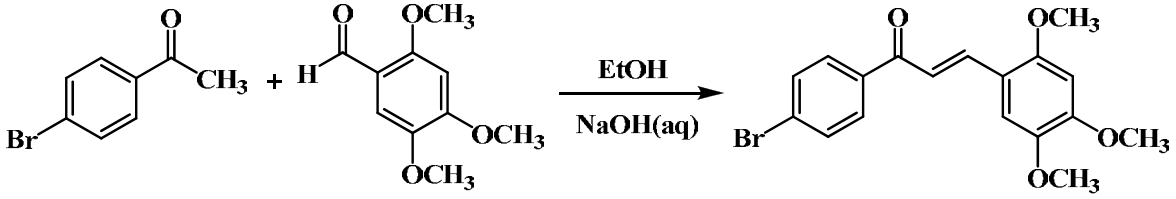
9. (*E*)-1-(furyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one



(**9**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอ่อน (0.22 g, 76%) จุดหลอมเหลว 117-118 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 244.0, 351.9, IR (KBr) ν (cm^{-1}): 3001-2977 (*v*, C-H aromatic stretching), 2946 (*s*, C-H stretching), 1654 (*s*, C=O stretching), 1582 (*s*, C=C stretching), 1123 (*s*, C-O stretching), 1064-1011 (*s*, C-H stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.24 (1H, *d*, *J* = 15.6 Hz), 7.74 (1H, *d*, *J* = 15.9 Hz), 7.67 (1H, *d*, *J* = 3.9 Hz), 7.25 (1H, *d*, *J* = 3.9 Hz), 6.60 (1H, *t*, *J* = 1.8Hz), 6.17 (2H, *s*), 3.94-3.88 (9H, *s*)

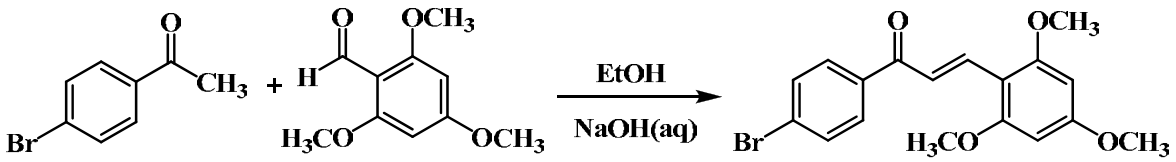
10. (*E*)-1-(4-Bromophenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one



(**10**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.33 g, 90%) จุดหลอมเหลว 153-154 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 273.4, 393.3, IR (KBr) ν (cm^{-1}): 2952 (*v*, C-H aromatic stretching), 2910 (*s*, C-H stretching), 1654 (*s*, C=O stretching), 1577 (*s*, C=C stretching), 1204 (*s*, C-O stretching), 1007-1028 (*s*, C-O stretching), 656 (*s*, C-Br stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.15 (1H, *d*, *J* = 15.9 Hz), 7.69-7.94 (4H, *d*, *J* = 8.4 Hz), 7.46 (1H, *d*, *J* = 15.9 Hz), 6.59-7.18 (2H, *s*), 3.97-4.02 (9H, *s*).

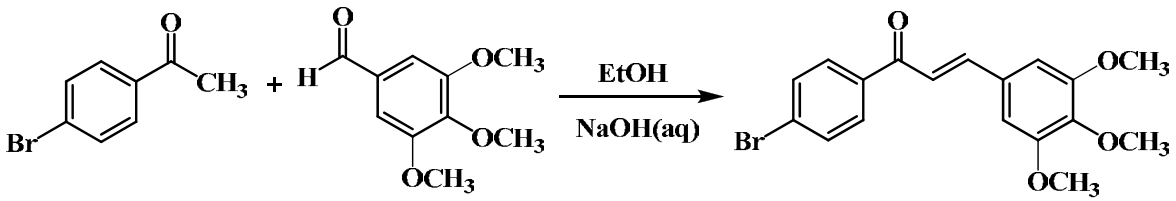
11. (*E*)-1-(4-Bromophenyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one



(**11**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอ่อน (0.29 g, 76%) จุดหลอมเหลว 152-153 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 264.3, 364.4, IR (KBr) ν (cm^{-1}): 2979 (*v*, C-H aromatic stretching), 2941 (*s*, C-H stretching), 1675-1690 (*s*, C=O stretching), 1572 (*s*, C=C stretching), 1418-1458 (*m*, C-C aromatic stretching), 1209 (*s*, C-O stretching), 1028, 1117-1154 (*s*, C-(C=O)-C stretching), 672 (*s*, C-Br stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.26 (1H, *d*, *J* = 15.9 Hz), 7.87 (2H, *d*, *J* = 9.0 Hz), 7.81 (1H, *d*, *J* = 15.9 Hz), 7.61 (2H, *d*, *J* = 9.0 Hz,), 6.14 (2H, s), 3.91 (3H, *s*), 3.87 (6H, *s*)

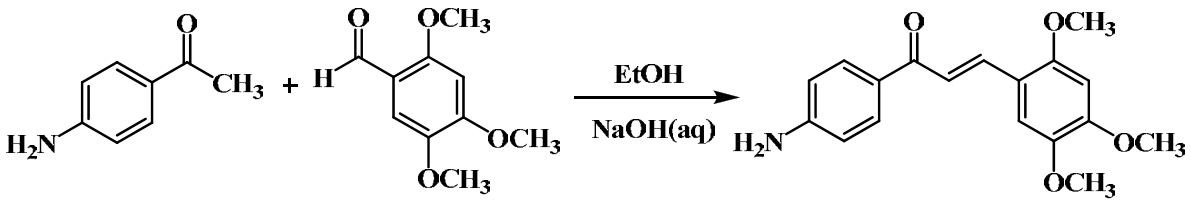
12. (*E*)-1-(4-Bromophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one



(**12**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอ่อน (0.32 g, 84%) จุดหลอมเหลว 128-129 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 266.1, 347.3, IR (KBr) ν (cm^{-1}): 3002-3058 (*s*, C-H aromatic stretching), 2944 (*s*, C-H stretching), 1667 (*s*, C=O stretching), 1584 (*s*, C=C stretching), 1124 (*s*, C-O stretching), 816 (*s*, C-H bending), 604 (*s*, C-Br stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 7.88 (2H, *d*, *J* = 8.4 Hz), 7.72 (1H, *d*, *J* = 15.6 Hz), 7.65 (2H, *d*, *J* = 8.4 Hz), 7.34 (1H, *d*, *J* = 15.6 Hz), 7.26 (2H, *s*), 3.93 (6H, *s*), 3.91 (3H, *s*)

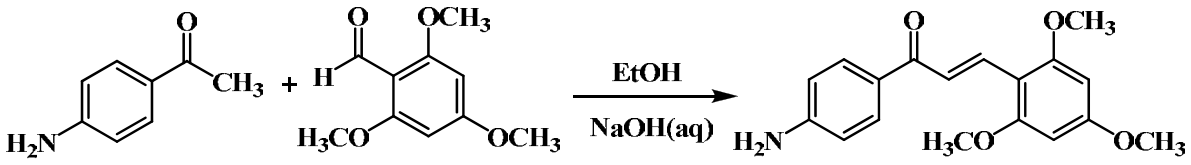
13. (*E*)-1-(4-Aminophenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one



(**13**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.60 g, 96%) จุดหลอมเหลว 207-208 °C, UV-Vis ($CHCl_3$) λ_{max} (nm): 244.6, 322.7, 379.8, IR (KBr) ν(cm^{-1}): 3376 (N-H stretching), 2927 (sp^2 C-H aromatic stretching), 1603 (C=O stretching), 1556 (C=C aromatic stretching), 1206 (*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.24 (2H, *d*, *J* = 15.3 Hz), 7.53 (2H, *d*, *J* = 8.1 Hz), 7.41 (1H, *d*, *J* = 15.3 Hz), 6.71 (1H, *s*), 6.42 (2H, *s*, *J* = 8.1 Hz), 6.28 (2H, *s*), 3.82 (6H, *s*), 3.79 (3H, *s*).

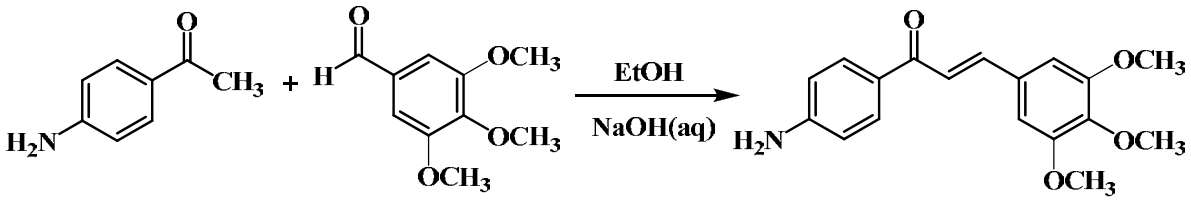
14. (*E*)-1-(4-Aminophenyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one



(**14**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.48 g, 76%) จุดหลอมเหลว 228-229 °C, UV-Vis ($CHCl_3$) λ_{max} (nm): 253.7, 356.7, IR (KBr) ν(cm^{-1}): 3301 (N-H stretching), 2957 (sp^2 C-H aromatic stretching), 1621 (C=O stretching), 1589 (C=C aromatic stretching), 1187 (*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.24 (2H, *d*, *J* = 15.3 Hz), 7.76 (2H, *d*, *J* = 8.1 Hz), 7.42 (1H, *d*, *J* = 15.3 Hz), 6.66 (2H, *d*, *J* = 8.1 Hz), 6.13 (2H, *s*), 3.92 (9H, *s*).

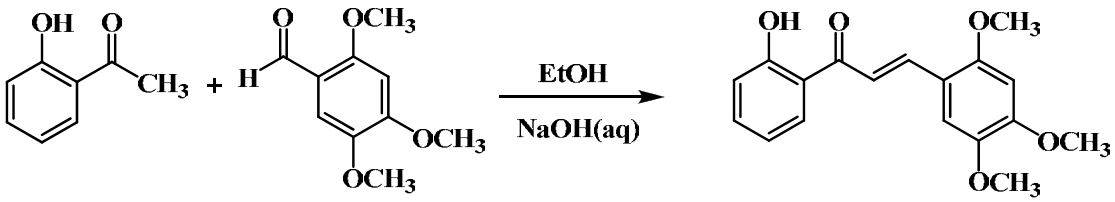
15. (*E*)-1-(4-Aminophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one



(**15**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.58 g, 92%) จุดหลอมเหลว 240-241 $^{\circ}$C, UV-Vis ($CHCl_3$) λ_{max} (nm): 253.8, 354.7, IR (KBr) ν(cm^{-1}): 3216 (N-H stretching), 2929 (sp^2 C-H aromatic stretching), 1617 (C=O stretching), 1567 (C=C aromatic stretching), 1189 (*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.12 (2H, *d*, *J* = 15.3 Hz), 7.64 (2H, *d*, *J* = 8.1 Hz), 7.56 (1H, *d*, *J* = 15.3 Hz), 6.85 (2H, *d*, *J* = 8.1 Hz), 6.27 (2H, *s*), 3.66 (3H, *s*), 3.64 (6H, *s*).

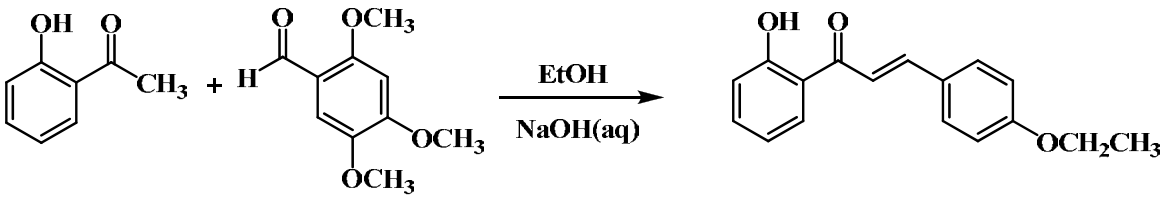
16. (*E*)-1-(2-Hydroxyphenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one



(**16**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอมส้ม (0.24 g, 78%) จุดหลอมเหลว 187-188 $^{\circ}$C, ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 13.10 (1H, *s*), 8.12 (1H, *d*, *J* = 15.6 Hz), 7.94 (1H, *d*, *J* = 8.4 Hz), 7.48 (1H, *d*, *J* = 15.6 Hz), 7.47 (1H, *t*, *J* = 7.8 Hz), 7.12 (1H, *s*), 7.01 (1H, *d*, *J* = 8.4 Hz), 6.98 (1H, *t*, *J* = 8.4 Hz), 6.62 (1H, *s*), 3.91-3.95 (9H, *s*)

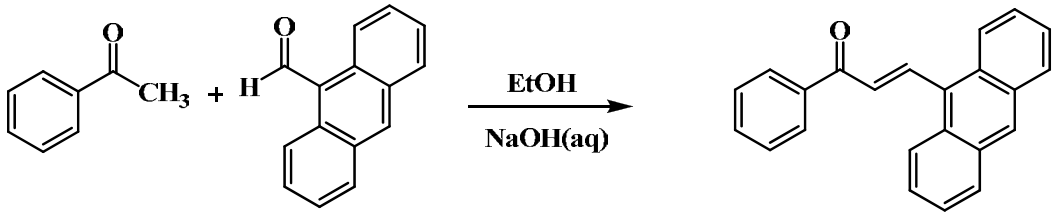
17. (*E*)-3-(4-Ethoxyphenyl)-1-(2-hydroxyphenyl)prop-2-en-1-one



(**17**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.12 g, 22%) จุดหลอมเหลว 114-115 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 245.1, 297.4, 371.3, IR (KBr) ν(cm^{-1}): 3350 (OH stretching), 2980 (sp^2 C-H aromatic stretching), 1650 (C=O stretching), 1605 (C=C aromatic stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 12.92 (1H, *s*), 7.93 (1H, *d*, *J* = 8.0 Hz), 7.92 (1H, *d*, *J* = 15.6 Hz), 7.62 (2H, *d*, *J* = 8.7 Hz), 7.54 (1H, *d*, *J* = 15.6 Hz), 7.47 (2H, *td*, *J* = 8.0, 2.4 Hz), 7.02 (1H, *d*, *J* = 8.0 Hz), 6.95 (2H, *d*, *J* = 8.7 Hz), 6.95 (1H, *m*), 4.09 (2H, *q*, *J* = 6.9 Hz), 1.45 (3H, *t*, *J* = 6.9 Hz).

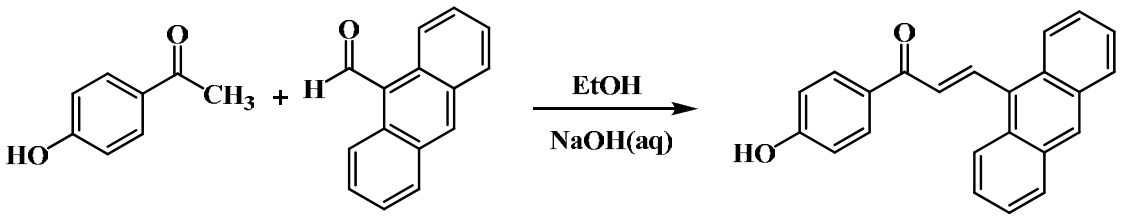
18. (*E*)-3-(9-Anthryl)-1-(phenyl)-prop-2-en-1-one



(**18**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.55 g, 83%) จุดหลอมเหลว 143-144 ^{o}C, UV-Vis ($CHCl_3$) λ_{max} (nm): 256.1, 370.7, IR (KBr) ν(cm^{-1}): 3010 (Ar CH stretching), 1670 (C=O stretching), 1600 (C=C aromatic stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.80 (1H, *d*, *J* = 15.9 Hz), 8.48 (1H, *s*), 8.31 (2H, *dd*, *J* = 9.3 Hz), 8.08 (1H, *t*, *J* = 9.3 Hz), 8.05 (1H, *d*, *J* = 15.9 Hz), 8.04 (2H, *dd*, *J* = 9.3 Hz), 7.50-7.59 (9H, *m*).

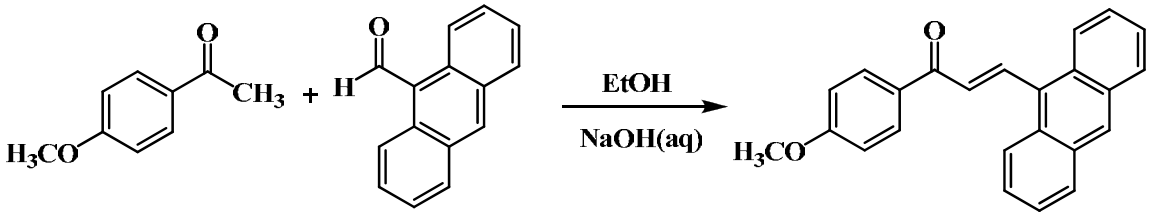
19. (*E*)-3-(9-Anthryl)-1-(4-hydroxyphenyl)-prop-2-en-1-one



(**19**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.34 g, 53%) จุดหลอมเหลว 143-144 °C, UV ($CHCl_3$) λ_{max} (nm): 257.85, 391.63, IR (KBr) ν(cm^{-1}): 3050 (Ar CH stretching), 1670 (C=O stretching), 1600 (C=C aromatic stretching), 3368 (-OH), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 9.04 (1H, *s*), 8.67 (1H, *s*), 8.52 (1H, *d*, *J* = 16.8 Hz), 8.24 (2H, *d*, *J* = 9.3 Hz), 8.14 (2H, *d*, *J* = 9.3 Hz), 7.50-7.65 (9H, *m*), 6.56 (1H, *d*, *J* = 16.8 Hz).

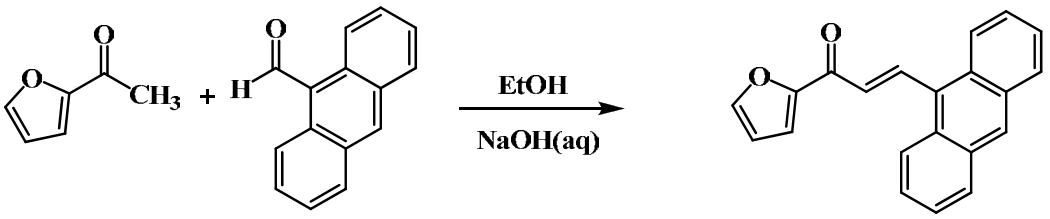
20. (*E*)-3-(9-Anthryl)-1-(4-methoxyphenyl)-prop-2-en-1-one



(**20**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.64 g, 83%) จุดหลอมเหลว 167-168 °C, UV ($CHCl_3$) λ_{max} (nm): 256.62, 297.41, 371.27, IR (KBr) ν(cm^{-1}): 2952 (Ar CH stretching), 1655 (C=O stretching), 1600 (C=C aromatic stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.71 (1H, *d*, *J* = 15.6 Hz), 8.48 (1H, *s*), 8.31 (2H, *dd*, *J* = 9.9 Hz), 8.07 (1H, *d*, *J* = 15.6 Hz), 8.04 (2H, *dd*, *J* = 9.9 Hz), 7.50-7.59 (9H, *m*), 3.89 (3H, *s*).

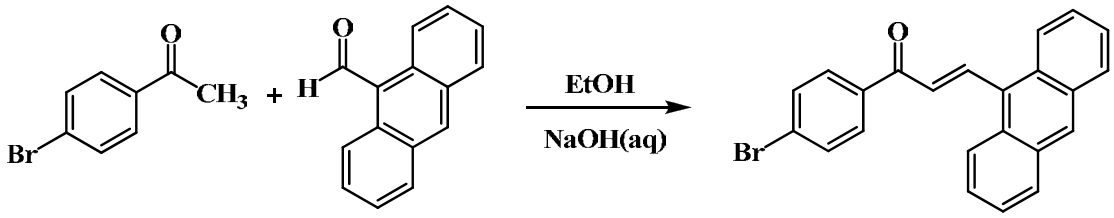
21. (*E*)-3-(Anthracen-9-yl)-1-(furan-2-yl)prop-2-en-1-one



(**21**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.55 g, 83%) จุดหลอมเหลว 150-151 °C, UV-Vis ($CHCl_3$) λ_{max} (nm): 256.6, 285.3, 370.7, IR (KBr) ν(cm^{-1}): 3030 (Ar CH stretching), 1670 (C=O stretching), 1580 (C=C aromatic stretching), 1250 (C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.48 (1H, *d*, *J* = 16.8 Hz), 8.04 (1H, *d*, *J* = 1.5 Hz), 7.70 (1H, *d*, *J* = 9.9 Hz), 7.48 (1H, *d*, *J* = 9.9 Hz), 7.33-7.60 (9H, *m*), 6.60 (1H, *d*, *J* = 16.8 Hz),.

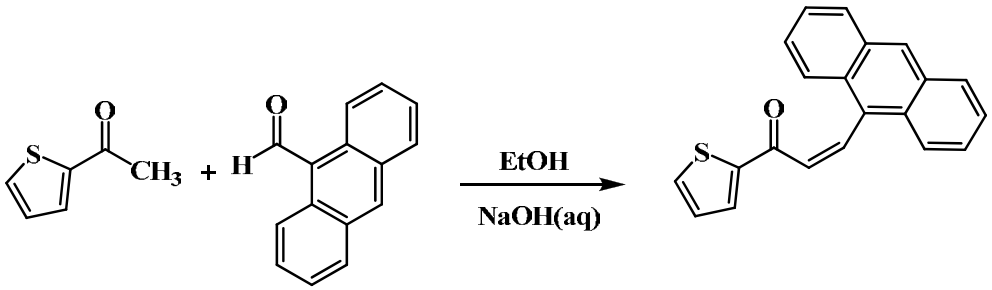
22. (*E*)-3-(9-Anthryl)-1-(4-bromophenyl)prop-2-en-1-one



(**22**)

ผลิตภัณฑ์เป็นของแข็งสีเหลือง (0.35 g, 92%) ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.74 (1H, *d*, *J* = 15.6 Hz), 8.52 (1H, *s*), 8.26-8.29 (2H, *m*), 8. 08-8.05 (2H, *m*), 8.03 (1H, *d*, *J* = 15.6 Hz), 7.99 (2H, *d*, *J* = 8.7 Hz), 7.69 (2H, *d*, *J* = 8.7 Hz), 7.52-7.59 (4H, *m*)

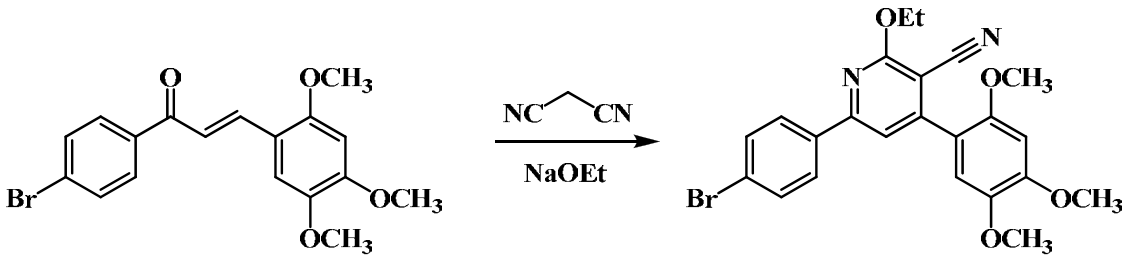
23. (*Z*)-3-(9-Anthryl)-1-(2-thienyl)prop-2-en-1-one



(**23**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอมส้ม (0.23 g, 72%) จุดหลอมเหลว 127-128 °C, ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 8.52 (1H, *s*), 8.13 (2H, *d*, *J* = 8.1 Hz), 8.07 (2H, *d*, *J* = 8.1 Hz), 8.01 (1H, *d*, *J* = 5.1 Hz), 7.91 (1H, *d*, *J* = 12.0 Hz), 7.82 (1H, *d*, *J* = 5.1 Hz), 7.73 (1H, *d*, *J* = 12.0 Hz), 7.42-7.51 (4H, *m*), 7.18 (1H, *t*, *J* = 5.1 Hz)

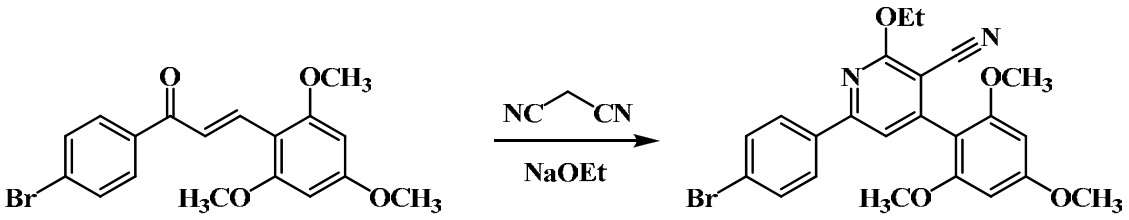
24. 6-(4-Bromophenyl)-2-ethoxy-4-(2,4,5-trimethoxyphenyl)nicotinonitrile



(**24**)

ผลิตภัณฑ์เป็นของแข็งสีเหลืองอ่อน (0.28 g, 61%) จุดหลอมเหลว 176-177 °C, IR (KBr) ν (cm^{-1}): 2831-2932 (*v*, C-H stretching), 2217 (*m*, C≡N stretching), 1517-1585 (*s*, C=C stretching), 1337 (*s*, C=N stretching), 1033-1214 (*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 7.93 (2H, *d*, *J* = 8.7 Hz), 7.61 (2H, *d*, *J* = 8.7 Hz), 7.40 (1H, *s*), 6.86 (1H, *s*), 6.65 (1H, *s* 4.64 (2H, *q*, *J* = 6.9 Hz), 3.97 (3H, *s*), 3.88 (3H, *s*), 3.86 (3H, *s*), 1.51(3H, *t*, *J* = 6.9 Hz).

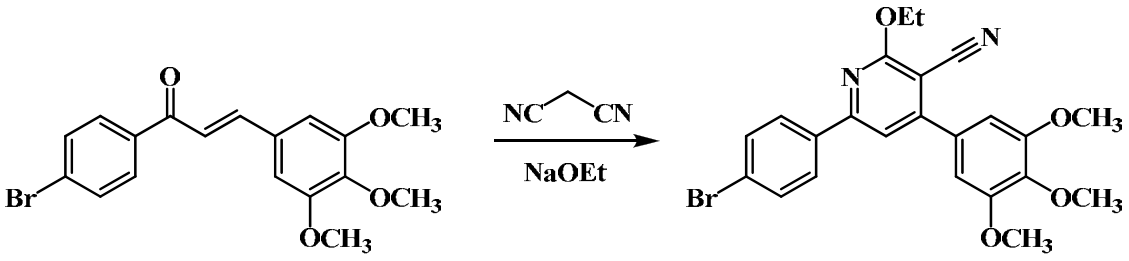
25. 6-(4-Bromophenyl)-2-ethoxy-4-(2,4,6-trimethoxyphenyl)nicotinonitrile



(**25**)

ผลิตภัณฑ์เป็นของแข็งสีขาว (0.27 g, 58%) จุดหลอมเหลว 137-138 ^{o}C, IR (KBr) ν (cm^{-1}): 2840-2977 (*v*, C-H stretching), 2222 (*m*, C≡N stretching), 1552-1611 (*s*, C=C stretching), 1337 (*s*, C=N stretching), 1026-1126 (*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 7.91 (2H, *d*, *J* = 8.7 Hz), 7.58 (2H, *d*, *J* = 8.7 Hz), 7.33 (1H, *s*), 6.23 (2H, *s*), 4.62 (2H, *q*, *J* = 6.9 Hz), 3.87 (3H, *s*), 3.77 (6H, *s*), 1.51 (3H, *t*, *J* = 6.9 Hz)

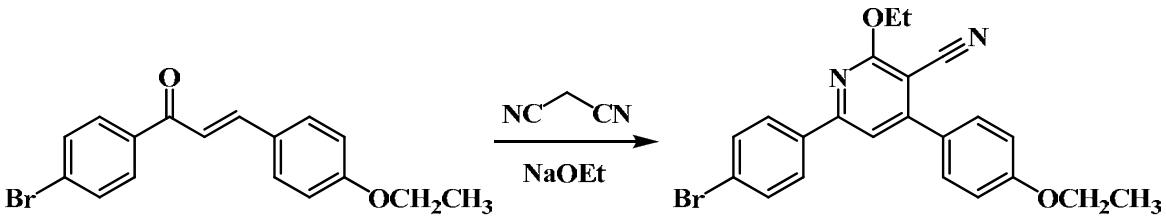
26. 6-(4-bromophenyl)-2-ethoxy-4-(3,4,5-trimethoxyphenyl)nicotinonitrile



(**26**)

ผลิตภัณฑ์เป็นของแข็งสีขาว (0.42, 62%) จุดหลอมเหลว 157-158 ^{o}C, IR (KBr) ν (cm^{-1}): 2842-2997 (*v*, C-H stretching), 2221 (*m*, C≡N stretching), 1509-1586 (*s*, C=C stretching), 1378 (*s*, C=N stretching), 1001-1132 (*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 7.96 (2H, *d*, *J* = 8.4 Hz), 7.63 (2H, *d*, *J* = 8.4 Hz), 7.42 (1H, *s*), 6.85 (2H, *s*), 4.66 (2H, *q*, *J* = 6.9 Hz), 3.94 (6H, *s*), 3.93 (3H, *s*), 1.53 (3H, *t*, *J* = 6.9 Hz).

27. 6-(4-Bromophenyl)-2-ethoxy-4-(4-ethoxyphenyl)nicotinonitrile

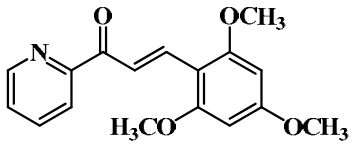


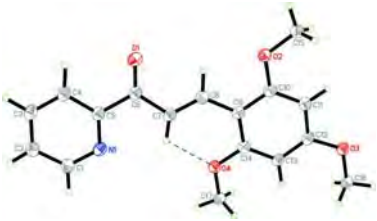
(**27**)

ผลิตภัณฑ์เป็นของแข็งสีขาว (0.28 g, 68%) จุดหลอมเหลว 134-135 ^{o}C, IR (KBr) ν (cm^{-1}): 2875-2973 (*v*, C-H stretching), 2221 (*m*, C≡N stretching), 1516-1588 (*s*, C=C stretching), 1344 (*s*, C=N stretching), 1145-1248 (*s*, C-O stretching), ^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz): 7.95 (2H, *d*, *J* = 8.7 Hz), 7.62 (2H, *d*, *J* = 8.7 Hz), 7.61 (2H, *d*, *J* = 8.7 Hz), 7.41 (1H, *s*), 7.03 (2H, *d*, *J* = 8.7 Hz), 4.65 (2H, *q*, *J* = 6.9 Hz), 4.11 (2H, *q*, *J* = 6.9 Hz), 1.52 (3H, *t*), 1.48 (3H, *t*, *J* = 6.9 Hz)

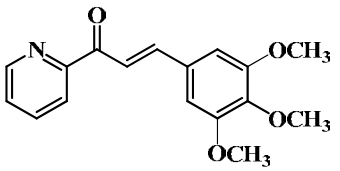
4.2 โครงสร้างทางรังสีเอกซ์

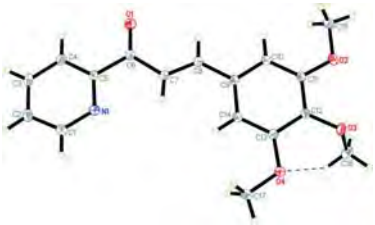
นอกจากทำการหาโครงสร้างสารด้วยเทคนิคทางสเปกโตสโกปีแล้ว สามารถตกผลึกและหาโคงสร้างสารด้วยเทคนิคการเลี้ยวเบนของรังสีเอกซ์บนผลึกเดี่ยวของสารได้ 15 สาร แสดงดัง Scheme 4.2 – 4.3


O
OCH3
N
H3CO
OCH3

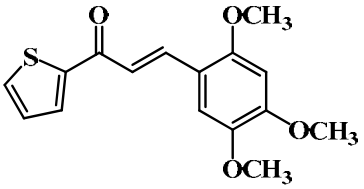


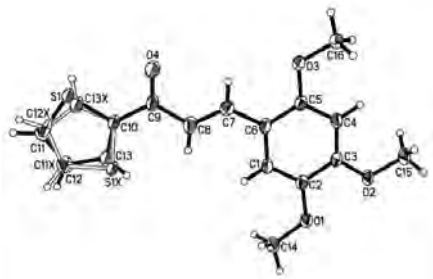
(**2**)


O
N
OCH3
OCH3
OCH3

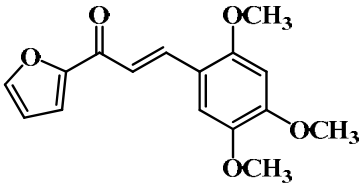


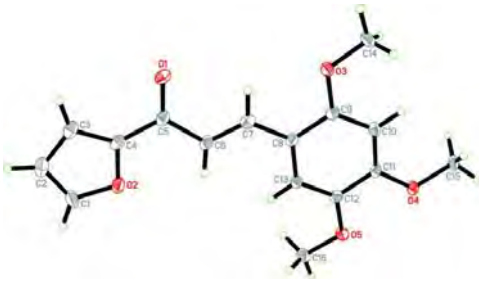
(**3**)


O
S
OCH3
OCH3
OCH3

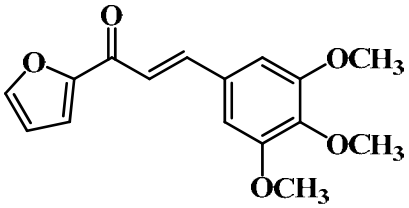


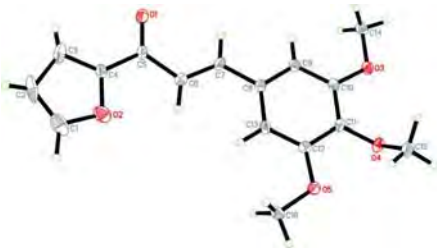
(**4**)


O
O
OCH3
OCH3
OCH3

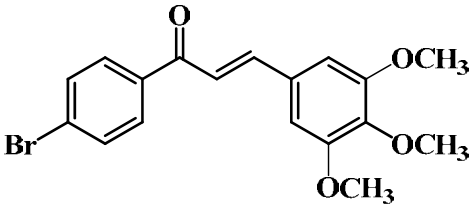


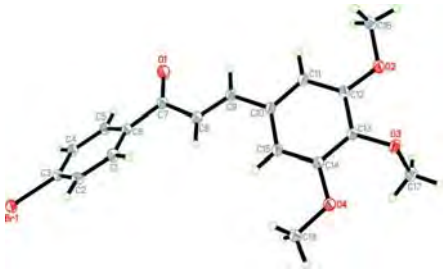
(**7**)


O
O
OCH3
OCH3
OCH3



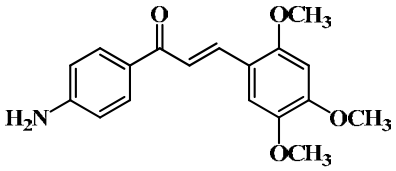
(**9**)

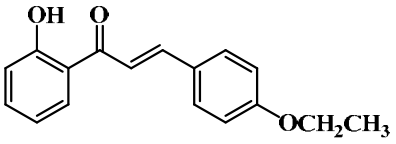

O
Br
OCH3
OCH3
OCH3

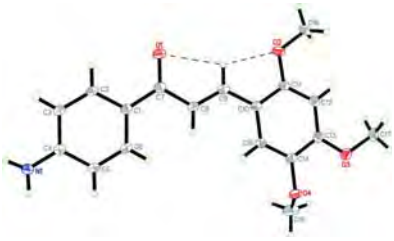


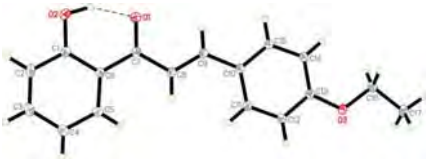
(**12**)

Scheme 4.2 X-ray structures of chalcones derivatives



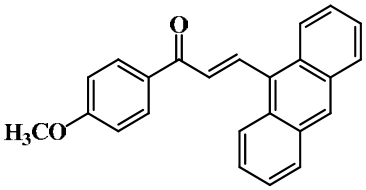


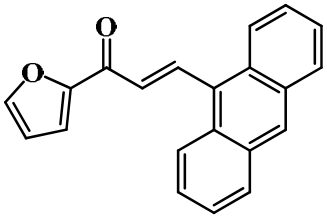


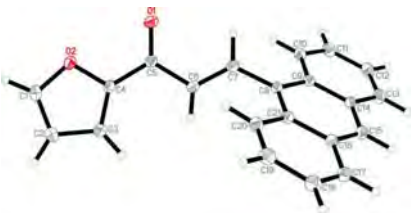


(**13**)

(**17**)

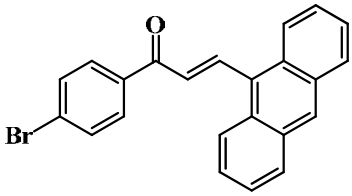


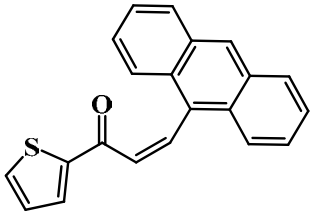


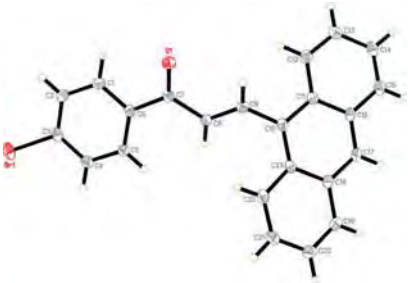


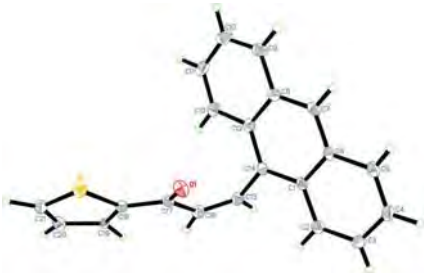
(**20**)

(**21**)





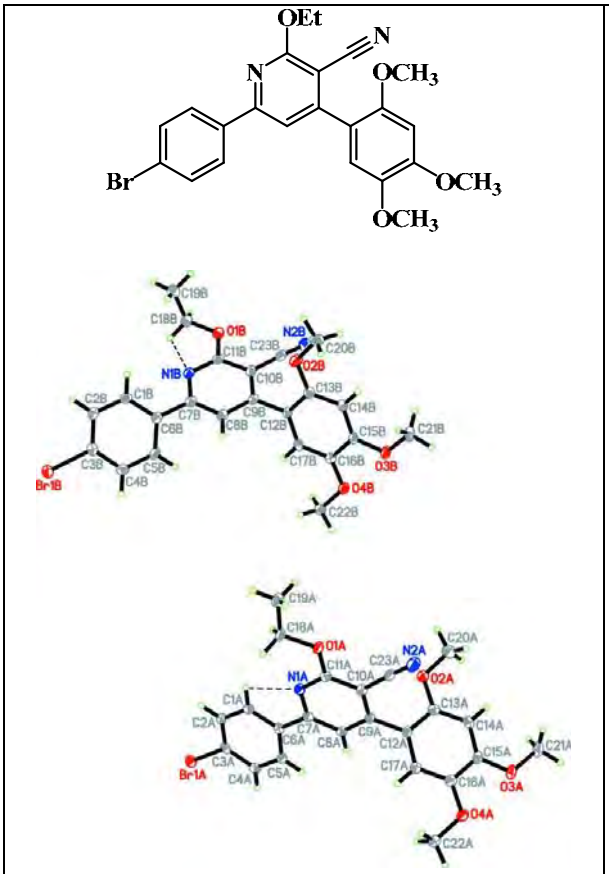




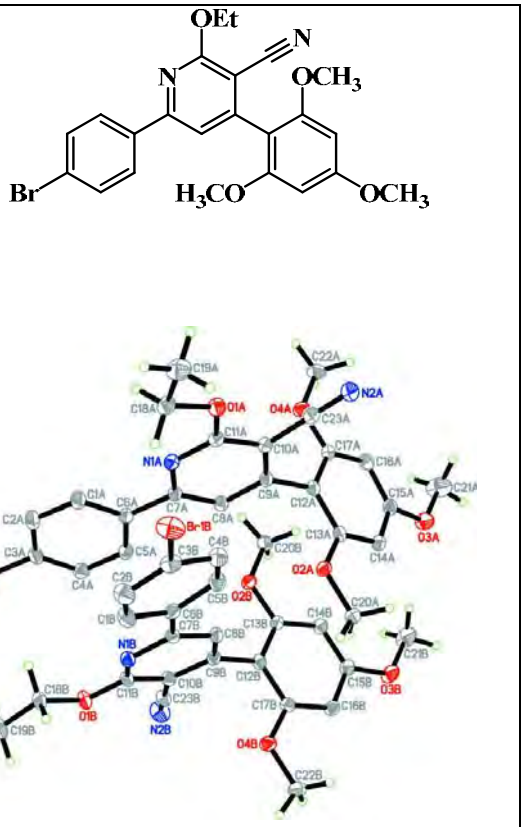
(**22**)

(**23**)

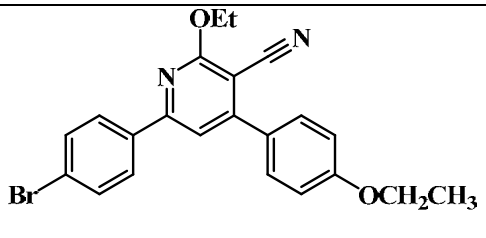
Scheme 4.2 (continue) X-ray structures of chalcone derivatives

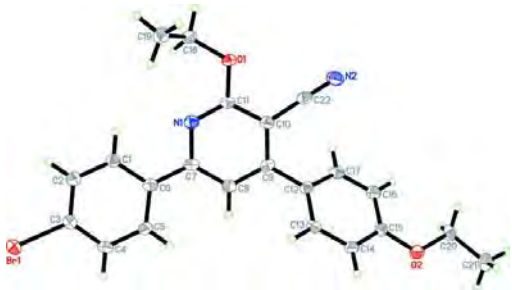


(**24**)



(**25**)





(**27**)

Scheme 4.3 X-ray structures of nicotinonitrile derivatives

รายละเอียดของโครงสร้างทางรังสีเอกซ์แสดงดังปรากฏในเอกสารผลงานตีพิมพ์

4.3 การทดสอบการออกฤทธิ์ยับยั้งเชื้อแบคทีเรียของสารอนุพันธ์ chalcones 1-23

ทำการทดสอบฤทธิ์ต้านเชื้อแบคทีเรียของสารอนุพันธ์ chalcones **1-23** ได้ผลดัง Table 4.1 จากผลการทดสอบพบว่าสารอนุพันธ์ chalcones ทั้ง 23 สาร ไม่แสดงฤทธิ์ยังยั้งเชื้อแบคทีเรียที่ทดสอบ คือ *Bacillus subtilis*, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR 459, methicillin-resistant *Staphylococcus aureus* (MRSA) ATCC 43300, vancomycin-resistant *Enterococcus faecalis* (VRE) ATCC 51299, *Salmonella typhi*, *Shigella sonei* and *Pseudomonas aeruginosa*.

Table 4.1. Antimicrobial Activity (MIC, μg/ml) of compounds 1-23

Compounds	Antibacterial activity							
	Gram-positive bacteria[(a)]					Gram-negative bacteria[(b)]		
	B. subtilis	*S. aureus*	*E. faecalis*	MRSA	VRE	*S. typhi*	*S. sonei*	*P. aeruginosa*
1	>300	>300	>300	>300	>300	>300	>300	>300
2	>300	>300	>300	>300	>300	>300	>300	>300
3	>300	>300	>300	>300	>300	>300	>300	>300
4	300	>300	>300	300	>300	>300	>300	>300
5	>300	>300	>300	>300	>300	>300	>300	>300
6	>300	>300	>300	>300	>300	>300	>300	>300
7	>300	>300	>300	>300	>300	>300	>300	>300
8	>300	>300	>300	>300	>300	>300	>300	>300
9	>300	>300	>300	>300	>300	>300	>300	>300
10	>300	>300	>300	>300	>300	>300	>300	>300
11	>300	>300	>300	>300	>300	>300	>300	>300
12	>300	>300	>300	>300	>300	>300	>300	>300
13	>300	>300	>300	>300	>300	>300	>300	>300
14	>300	>300	>300	300	>300	300	300	>300
15	>300	>300	>300	>300	>300	>300	>300	>300
16	>300	150	>300	>300	>300	150	150	>300
17	>300	>300	>300	>300	>300	>300	>300	>300
18	>300	>300	>300	>300	>300	>300	>300	>300
19	>300	>300	>300	>300	>300	>300	>300	>300
20	>300	>300	>300	>300	>300	>300	>300	>300
21	>300	>300	>300	>300	>300	>300	>300	>300
22	>300	>300	>300	>300	>300	>300	>300	150
23	>300	>300	>300	>300	>300	>300	>300	>300
Vancomycin (Standard)	<2.34	<2.34	<2.34	<2.34	<2.34	<2.34	<2.34	<2.34

(a) Bacillus subtilis, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR 459, methicillin-resistant *Staphylococcus aureus* (MRSA) ATCC 43300, vancomycin-resistant *Enterococcus faecalis* (VRE) ATCC 51299. *(b) Salmonella typhi*, *Shigella sonei* and *Pseudomonas aeruginosa*.

4.4 สมบัติฟลูออเรสเซนต์ของสาร

ทำการศึกษาสมบัติฟลูออเรสเซนต์ของสารอนุพันธ์ chalcones ที่มีหมู่แทนที่เป็น trimethoxy (สาร **1**-**9**) เพื่อศึกษาผลของตำแหน่งหมู่แทนที่ trimethoxy ที่มีต่อสมบัติฟลูออเรสเซนต์ของอนุพันธ์ chalcones ได้ผลดังแสดง Fig 4.1 และ Table 4.2

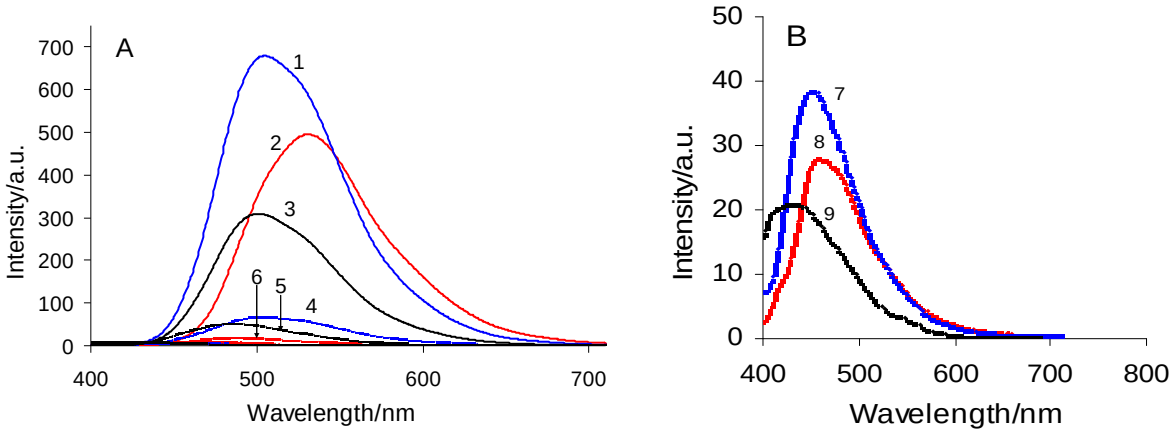


Fig. 4.1 Fluorescence emission spectra of heteroaryl chalcones (5 µM in chloroform) excited at 380 nm at room temperature: compounds **1**-**6** using slit width 4.5 (A) and compounds **7**-**9** using slit width 10 (B).

Table 4.2 Fluorescence spectral data (excitation (λ_{ex}), absorption (λ_{abs}), and emission (λ_{em}) maxima, fluorescent quantum yield (Φ_F))^{a,b} and Stokes shift of heteroaryl chalcones (**1**-**9**) (5 μM in chloroform)

Compound	λ_{ex}/nm	λ_{abs}/nm	λ_{em}/nm	Φ_F	Stokes shift/nm
1	401	403	530	0.18	127
2	350	372	453	< 0.01	81
3	377	355	502	0.05	147
4	394	395	504	0.28	109
5	354	368	433	< 0.01	65
6	374	350	485	0.06	135
7	394	396	501	0.20	105
8	352	365	431	< 0.01	66
9	372	352	481	0.08	129

a) The emission wavelength was set at 485 nm for excitation spectra study and the excitation wavelength was set at 380 nm for emission spectra study; *b*) coumarin 1 (in ethanol, Φ_F = 0.73) was used as the standard for Φ_F measurements.

ผลการศึกษาสมบัติฟลูออเรสเซนต์และผลของตำแหน่งหมู่แทนที่ trimethoxy ที่มีต่อสมบัติฟลูออเรสเซนต์ของอนุพันธ์ chalcones แสดงดังรายละเอียดในผลงานตีพิมพ์ Reprint 3/4

Thitipone Suwunwong, Suchada Chantrapromma* and Hoong-Kun Fun (2011)

“Influence of trimethoxy-substituted positions on fluorescence of heteroaryl chalcone derivatives” *Chemical Papers*, **65**, 890-897.

เอกสารอ้างอิง

L. E. Alcaradz, S. E. Blanco, O. N. Puig, F. Tomads and F. H. Ferretti. 2000, “Antibacterial Activity of Flavonoids Against Methicillin-resistant *Staphylococcus aureus* strains” *J. theor. Biol*. **205**: 231-240.

Y. R. Prasad, P. R. Kumar, D. J. Smiles and P. A. Babu. 2008, “QSAR studies on chalcone derivatives as antibacterial agents against *Bacillus pumilis*” *Arkivoc*. **11**: 266-276.

V. Tomar, G. Bhattacharjee, Kamaluddina and A. Kumar. 2007, “Synthesis and antimicrobial evaluation of new chalcones containing piperazine or 2,5-dichlorothiophene moiety” *Bioorg. Med. Chem. Lett.* **17**: 5321–5324.

P. M. Sivakumar, S. P. Seenivasan, V. Kumarb and M. Doble. 2007, “Synthesis, antimycobacterial activity evaluation, and QSAR studies of chalcone derivatives” *Bioorg. Med. Chem. Lett.* **17**: 1695–1700.

Z. Nowakowska, B. Kedzia and G. Schroeder. 2007, “Synthesis, physicochemical properties and antimicrobial evaluation of new (*E*)-chalcones” *European Journal of Medicinal Chemistry*. **43**: 707-713.

M. D. Bowman, J. C. O’Neill, J. R. Stringer and H. E. Blackwell. “Rapid Identification of Antibacterial Agents Effective against *Staphylococcus aureus* Using Small-Molecule Macroarrays” *Chemistry & Biology*. **14**: 351–357.

N. S. H. N. Moorthy, R. J. Singh, H. P. Singh and S. D. Gupta. “Synthesis, Biological Evaluation and *In Silico* Metabolic and Toxicity Prediction of Some Flavanone Derivatives” *Chem. Pharm. Bull.* **54**: 1384-1390.

S. F. Nielsen, T. Boesen, M. Larsen, K. Schønning and H. Kromann. “Antibacterial chalcones-bioisosteric replacement of the 4’-hydroxy group” *Bioorg. Med. Chem.* **12**: 3047–3054.

Y. M. Lin, Y. Zhou, M. T. Flavin, L. M. Zhou, W. Niea and F. C. Chen. “Chalcones and Flavonoids as Anti-Tuberculosis Agents” *Bioorg. Med. Chem.* **10**: 2795–2802.

ผลงานตีพิมพ์ในวารสารวิชาการระดับนานาชาติ

ได้ตีพิมพ์ผลงานวิจัยในวารสารวิชาการระดับนานาชาติ จำนวน 31 เรื่อง ดังนี้
(Reprints 1/1 –1/9, 2/1-2/10 และ 3/1-3/12 ดังภาคผนวก)

1. Nawong Boonnak, Chatchanok Karalai*, Suchada Chantrapromma, Chanita Ponglimanont, Hoong-Kun Fun, Akkharawit Kanjana-Opas, Kan Chantrapromma and Shigeru Kato (2009)
   "Anti-Pseudomonas aeruginosa xanthones from the resin and green fruits of *Cratoxylum cochinchinense*" *Tetrahedron*, **65**, 3003–3013. (IF = 3.219 Reprints 1/1)

2. Suchada Chantrapromma*, Nawong Boonnak and Hoong-Kun Fun (2009)
   "7-Acetoxycochinchinone I"
   Acta Crystallographica, E**65**, o2223–o2224. (IF = 0.411 Reprints 1/2)

3. Thitipone Suwunwong, Suchada Chantrapromma* and Hoong-Kun Fun (2009)
   "(*E*)-1-(4-Bromophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one"
   Acta Crystallographica, E**65**, o120. (IF = 0.411 Reprints 1/3)

4. Thitipone Suwunwong, Suchada Chantrapromma*, Chatchanok Karalai, Paradorn Pakdeevanich and Hoong-Kun Fun (2009)
   "(*E*)-3-(Anthracen-9-yl)-1-(4-bromophenyl)prop-2-en-1-one"
   Acta Crystallographica, E**65**, o420–o421. (IF = 0.411 Reprints 1/4)

5. Hoong-Kun Fun, Thitipone Suwunwong, Nawong Boonnak and Suchada Chantrapromma* (2009)
   "(*Z*)-3-(9-Anthryl)-1-(2-thienyl)prop-2-en-1-one"
   Acta Crystallographica, E**65**, o2168–o2169. (IF = 0.411 Reprints 1/5)

6. Suchada Chantrapromma*, Jirapa Horkaew, Thitipone Suwunwong and Hoong-Kun Fun (2009)
   "(*Z*)-3-(9-Anthryl)-1-(4-methoxyphenyl)prop-2-en-1-one"
   Acta Crystallographica, E**65**, o2673–o2674. (IF = 0.411 Reprints 1/6)

7. Suchada Chantrapromma*, Hoong-Kun Fun, Thitipone Suwunwong, Mahesh Padaki and Arun M. Isloor (2009)
"6-(4-Bromophenyl)-2-ethoxy-4-(2,4,6-trimethoxyphenyl)nicotinonitrile"
Acta Crystallographica, E**65**, o2914–o2915. (IF = 0.411 Reprints 1/7)

8. Suchada Chantrapromma*, Hoong-Kun Fun, Thitipone Suwunwong, Mahesh Padaki and Arun M. Isloor (2010)
"6-(4-Bromophenyl)-2-ethoxy-4-(4-ethoxyphenyl)nicotinonitrile"
Acta Crystallographica, E**66**, o79–o80. (IF = 0.411 Reprints 1/8)

9. Suchada Chantrapromma*, Thitipone Suwunwong, Nawong Boonnak and Hoong-Kun Fun (2010)
"A second orthorhombic polymorph of (*Z*)-3-(9-anthryl)-1-(2-thienyl)prop-2-en-1-one" *Acta Crystallographica*, E**66**, o312–o313. (IF = 0.411 Reprints 1/9)

10. Nawong Boonnak, Chatchanok Karalai*, Suchada Chantrapromma, Chanita Ponglimanont, Akkharawit Kanjana-Opas, Kan Chantrapromma and Shigeru Kato (2010)
"Chromene and Prenylated Xanthones from the Roots of *Cratoxylum formosum* ssp. *Pruniflorum*"
Chemical & Pharmaceutical Bulletin, **58**, 386-389. (IF = 1.698 Reprint 2/1)

11. Nawong Boonnak, Achjana Khamthip, Chatchanok Karalai*, Suchada Chantrapromma, Chanita Ponglimanont, Akkharawit Kanjana-Opas, Supinya Tewtrakul, Kan Chantrapromma, Hoong-Kun Fun and Shigeru Kato (2010)
"Nitric Oxide Inhibitory Activity of Xanthones from the Green Fruits of *Cratoxylum formosum* ssp. *Pruniflorum*"
Australian Journal of Chemistry, **63**, 1550-1556. (IF = 1.959 Reprint 2/2)

12. Nawong Boonnak, Suchada Chantrapromma*, Hoong-Kun Fun and Chatchanok Karalai (2010)
"Vieillardiixanthone B"
Acta Crystallographica, E**66**, o817–o818. (IF = 0.411 Reprint 2/3)

13. Suchada Chantrapromma*, Nawong Boonnak, Hoong-Kun Fun, Chatchanok Karalai and Kan Chantrapromma (2010) “Brasilixanthone”
Acta Crystallographica, E**66**, o2066–o2067. (IF = 0.411 Reprint 2/4)

14. Suchada Chantrapromma*, Hoong-Kun Fun, Mahesh Padaki, Thitipone Suwunwong and Arun M. Isloor (2010)
“6-(4-Bromophenyl)-2-ethoxy-4-(2,4,5-trimethoxyphenyl)nicotinonitrile”
Acta Crystallographica, E**66**, o641-o642. IF = 0.411 (Reprint 2/5)

15. Jirapa Horkaew, Thitipone Suwunwong, Suchada Chantrapromma*, Chatchanok Karalai and Hoong-Kun Fun (2010)
“(*E*)-3-(Anthracen-9-yl)-1-(furan-2-yl)prop-2-en-1-one”
Acta Crystallographica, E**66**, o800–o801. (IF = 0.411 Reprint 2/6)

16. Hoong-Kun Fun, Thawanrat Kobkeatthawin, Pumsak Ruanwas and Suchada Chantrapromma* (2010)
“(*E*)-1-(4-Aminophenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one”
Acta Crystallographica, E**66**, o1973–o1974. (IF = 0.411 Reprint 2/7)

17. Jirapa Horkaew, Suchada Chantrapromma*, Nisakorn Saewan and Hoong-Kun Fun (2010) “(*E*)-3-(4-Ethoxyphenyl)-1-(2-hydroxyphenyl)prop-2-en-1-one”
Acta Crystallographica, E**66**, o2346–o2347. (IF = 0.411 Reprint 2/8)

18. Hoong-Kun Fun, Thitipone Suwunwong**,** Suchada Chantrapromma* and Chatchanok Karalai (2010)
“(*E*)-1-(2-Furyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one”
Acta Crystallographica, E**66**, o2559-o2560. (IF = 0.411 Reprint 2/9)

19. Hoong-Kun Fun, Thitipone Suwunwong**,** Suchada Chantrapromma* and Chatchanok Karalai (2010)
“(*E*)-1-(2-Furyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one”
Acta Crystallographica, E**66**, o3070–o3071. (IF = 0.411 Reprints 2/10)

20. Suchada Chantrapromma*, Thawanrat Kobkeatthawin, Kullapa Chanawanno, Pitikan Wisitsak and Hoong-Kun Fun (2011)
"(*E*)-1-(4-Aminophenyl)-3-(pyridin-3-yl)prop-2-en-1-one"
Acta Crystallographica, E**67**, o1770–o1771. (IF = 0.411 Reprints 3/1)

21. Thawanrat Kobkeatthawin, Suchada Chantrapromma*, Nisakorn Saewan and Hoong-Kun Fun (2011)
"(*E*)-1-(4-Aminophenyl)-3-(naphthalen-2-yl)prop-2-en-1-one"
Acta Crystallographica, E**67**, o1204–o1205. (IF = 0.411 Reprints 3/2)

22. Hoong-Kun Fun, Suchada Chantrapromma*, Nawong Boonnak, Chatchanok Karalai and Kan Chantrapromma (2011)
"Redetermination and absolute configuration of pruniflorone M monohydrate"
Acta Crystallographica, E**67**, o1916–o1917. (IF = 0.411 Reprints 3/3)

23. Thitipone Suwunwong, Suchada Chantrapromma* and Hoong-Kun Fun (2011)
"Influence of trimethoxy-substituted positions on fluorescence of heteroaryl chalcone derivatives"
Chemical Papers, **65**, 890-897. (IF = 0.791 Reprints 3/4)

24. Suchada Chantrapromma*, Thawanrat Kobkeatthawin, Kullapa Chanawanno, Jaruwan Joothamongkhon and Hoong-Kun Fun (2011)
"Redetermination of (*E*)-3-(anthracen-9-yl)-1-(2-hydroxyphenyl)prop-2-en-1-one"
Acta Crystallographica, E**67**, o2554–o2555. (IF = 0.411 Reprints 3/5)

25. Hoong-Kun Fun, Thitipone Suwunwong, Kullapa Chanawanno, Pitikan Wisitsak and Suchada Chantrapromma* (2011)
"(*E*)-1-(2-Hydroxyphenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one"
Acta Crystallographica, E**67**, o2287–o2288. (IF = 0.411 Reprints 3/6)

26. Hoong-Kun Fun, Thitipone Suwunwong and Suchada Chantrapromma* (2011)
"(*E*)-1-(Pyridin-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one"
Acta Crystallographica, E**67**, o2406–o2407. (IF = 0.411 Reprints 3/7)

27. Hoong-Kun Fun, Suchada Chantrapromma* and Thitipone Suwunwong (2011)
"(2*E*)-1-(Pyridin-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one"
Acta Crystallographica, E**67**, o2789–o2790. (IF = 0.411 Reprints 3/8)

28. Hoong-Kun Fun, Thitipone Suwunwong, Teerasak Anantapong, Chatchanok Karalai and Suchada Chantrapromma* (2011)
"(*E*)-1-(Thiophen-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one"
Acta Crystallographica, E**67**, o3074–o3075. (IF = 0.411 Reprints 3/9)

29. Hoong-Kun Fun, Thitipone Suwunwong and Suchada Chantrapromma* (2012)
"3-(4-Chlorophenyl)-5-(thiophen-2-yl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide"
Acta Crystallographica, E**68**, o259–o260. (IF = 0.411 Reprints 3/10)

30. Thitipone Suwunwong, Suchada Chantrapromma*, Chatchanok Karalai, Pitikan Wisitsak and Hoong-Kun Fun (2012)
"(*E*)-1-(Furan-2-yl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one"
Acta Crystallographica, E**68**, o317–o318. (IF = 0.411 Reprints 3/11)

31. Thitipone Suwunwong, Suchada Chantrapromma*, and Hoong-Kun Fun (2012)
"6-(4-Aminophenyl)-4-(4-ethoxyphenyl)-2-methoxynicotinonitrile"
Acta Crystallographica, E**68**, o2812–o2813. (IF = 0.411 Reprints 3/12)

ภาคผนวก

Reprints 1/1-1/9, 2/1-2/10 และ 3/1-3/12

Reprints จากวารสาร *Australian Journal of Chemistry* และ *Acta Crystallographica*

แนบเฉพาะหน้าที่เกี่ยวข้องไม่แสดง Supplementary materials

Tetrahedron 65 (2009) 3003–3013

Contents lists available at ScienceDirect

Tetrahedron

journal homepage: www.elsevier.com/locate/tet

Anti-*Pseudomonas aeruginosa* xanthones from the resin and green fruits of *Cratoxylum cochinchinense*

Nawong Boonnak [a,b], Chatchanok Karalai [a,b,*], Suchada Chantrapromma [a], Chanita Ponglimanont [b], Hoong-Kun Fun [c], Akkharawit Kanjana-Opas [d], Kan Chantrapromma [e], Shigeru Kato [f]

[a] *Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand*
[b] *Department of Chemistry and Center of Excellence for Innovation in Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand*
[c] *X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia*
[d] *Department of Industrial Biotechnology, Faculty of Agro-Industry, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand*
[e] *Institute of Research and Development, Walailak University, Thasala, Nakhon Si Thammarat, 80160, Thailand*
[f] *Department of Materials and Life Science, Seikei University, Tokyo 180 8633, Japan*

ARTICLE INFO

Article history:
Received 23 October 2008
Received in revised form 8 January 2009
Accepted 22 January 2009
Available online 29 January 2009

Keywords:
Cratoxylum cochinchinense
1,3,7-Trihydroxyxanthone
1,3,7-Trioxygenated xanthone
Antifungal activity
Antibacterial activity
Pseudomonas aeruginosa

ABSTRACT

Cochinchinones I–L (**1**–**3** and **13**) along with 11 known xanthones (**4**–**12**, **14**, and **15**) were isolated from the resin and green fruits of *Cratoxylum cochinchinense*. In addition, four new acetylated compounds (**16**–**19**) were derivatized from 7-geranyloxy-1,3-dihydroxyxanthone (**14**) and 3-geranyloxy-1,7-dihydroxyxanthone (**15**). All compounds were characterized on the basis of spectroscopic analyses. The structures of cochinchinone I (**1**), a monoacetate (**18**) and a dibrosylate (**20**), were also confirmed by X-ray diffraction analysis. The antibacterial and antifungal activities of selected compounds were evaluated as well.

1. Introduction

Cratoxylum cochinchinense belongs to the family Guttiferae, which is distributed in several parts of Thailand.[1] This plant is a well-known tropical tree, and is commonly known in Thailand as Tui Kliang. The bark, roots, and leaves of this plant have been used in folk medicine to treat fever, coughs, diarrhea, itches, ulcers, and abdominal complaints.[2] Previous investigations revealed the major components from the *Cratoxylum* genus as xanthones and anthraquinones.[3–6] Xanthones have been reported to exhibit biological activities such as antibacterial,[3–5] antioxidant,[6,7]cytotoxic,[3–5,8,9] and antiprotozoal activities.[10] A preliminary screening of the bioactivity of the crude extracts from the resin and green fruits of *C. cochinchinense* has shown strong antibacterial activity specifically against *Pseudomonas aeruginosa*. Although *Pseudomonas sepsis* is a major cause of death in transplant recipients, while *P. aeruginosa* is one of the most common Gram-negative pathogenic bacteria causing life-threatening infections.[11–13] This result led us to investigate the bioactive compounds from this plant. The antibacterial and antifungal activities of isolated compounds were also evaluated.

2. Results and discussion

The crude CH_2Cl_2 extract of the resin of *C. cochinchinense* was subjected to chemical investigation leading to the isolation of three new xanthones, cochinchinones I–K (**1**–**3**), together with nine known xanthones identified as cochinchinone A (**4**),[6] 1,3,7-trihydroxy-2,4-diisoprenylxanthone (**5**),[14] celebixanthone methyl ether (**6**),[15] dulxis-xanthone F (**7**),[15] β-mangostin (**8**),[16] α-mangostin (**9**),[16] macluraxanthone (**10**),[17] pruniflorone G (**11**),[3] and a caged-prenylated xanthone (**12**).[6] Their structures were elucidated by NMR analysis and comparison of their spectroscopic data with those reported in the literature.

Cochinchinone I (**1**) was obtained as a yellow powder, which was further recrystallized from acetone to yield yellow single crystals. The X-ray structure (Fig. 1) suggested a xanthone skeleton with a chromene ring. A molecular formula $C_{28}H_{30}O_5$ was implied by HRMS. Its structure was supported by the ^1H and ^{13}C NMR spectral data (Table 1). The ^1H and ^{13}C NMR spectral data of **1** were

* Corresponding author. Tel.: +66 7428 8444; fax: +66 7421 2918.
E-mail addresses: chatchanok.k@psu.ac.th, chatchanok_k@yahoo.com (C. Karalai).

0040-4020/$ – see front matter © 2009 Elsevier Ltd. All rights reserved.
doi:10.1016/j.tet.2009.01.083

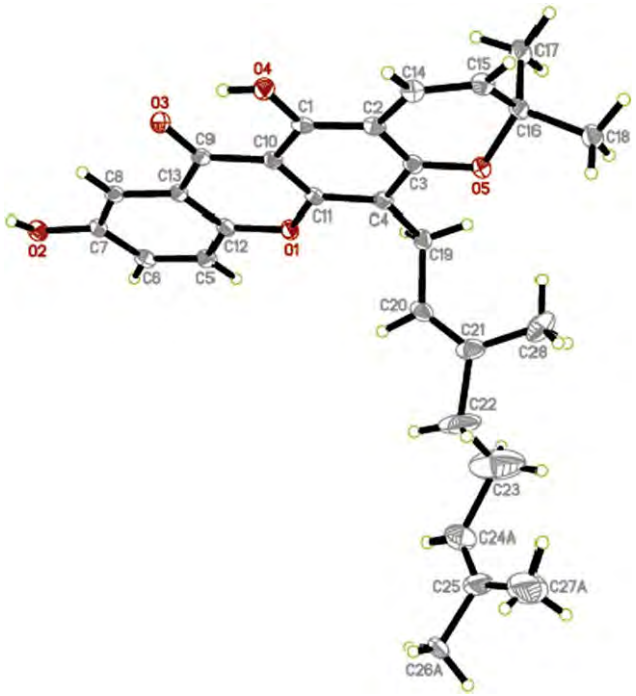


Figure 1. The X-ray structure of **1**.

comparable to cochinchinone A (**4**), which was previously isolated from the roots of *C. cochinchinense*.[6] The main difference was observed at C-2 and C-3, where the ^{1}H NMR spectral data of **1** showed the signals of a chromene ring at δ 6.74 (d, *J* 9.9 Hz, H-1′), 5.60 (d, *J* 9.9 Hz, H-2′), and 1.48 (s, Me-4′ and Me-5′) instead of a prenyl group at C-2 and a free hydroxyl group at C-3 as in **4** (Table 1). The chromene ring was connected to a xanthone skeleton in a linear fashion whose structure was confirmed by HMBC correlations as summarized in Table 2, which agreed well with the X-ray structure (Fig. 1). From these data, the structure of cochinchinone I was assigned as **1**. In addition, the asymmetric unit of a cocrystal (Fig. 1a) consisted of a disordered mixture of cochinchinone I (**1**) (Fig. 1) and a new chromenoxanthone **1a** (Fig. 1b). The latter was different from **1** in which a geranyl side chain in **1** was replaced by a (*E*)-3″,7″,7″-trimethyloct-2″-ene side chain in **1a**. Therefore, a trace amount of chromenoxanthone **1a** in a single crystal should be an unexpected contaminated compound from cochinchinone I (**1**), which might be transformed during the resin formation.

Cochinchinone J (**2**) was obtained as a yellow viscous oil with a molecular ion peak at *m*/*z* 446.2092 $[M]^+$ in the HRMS, corresponding to a molecular formula of $C_{28}H_{30}O_5$. The UV spectrum showed absorption bands of a xanthone (243, 289, 298, 320, 351, and 391 nm),[14] while the IR spectrum exhibited the hydroxyl and conjugated carbonyl functionalities at ν_{max} 3397 and 1649 cm^{-1}, respectively. The ^{1}H and ^{13}C NMR data of **2** (Table 1) were similar to those of **4**,[6] except for the appearance of the signals of a chromene ring bearing a methyl group and six-carbon side chain of 4-methylpent-3-enyl group, which appeared at δ_H 5.10 (br t, *J* 7.2 Hz, H-6″), 1.89 (m, 1H-4″), 1.68 (m, 1H-4″), 2.12 (m, H_2-5″), 1.66 (s, Me-8″), and 1.57 (s, Me-10″) (Table 1) instead of a geranyl moiety at C-4 as in **4**. The loss of 4-methylpent-3-enyl moiety in EIMS, *m*/*z* 363 ($[M]^+ -83$), also supported the proposed structure. Finally, the location of the angular chromene ring was confirmed by HMBC correlations (Table 2) in which the methine proton H-1″ (δ 6.88) was correlated with C-3 (δ 158.9), C-4 (δ 100.2), C-4a (δ 150.2), and C-3″ (δ 80.6). Therefore, the structure of cochinchinone J was deduced to be **2**.

Compound **3** was isolated as an inseparable mixture with compound **2**, and the mixture was thus acetylated with Ac_2O in pyridine. The resulting product was further separated by CC eluting with 70% $CHCl_3$–hexane to give monoacetates **3a** (18.9 mg) and **2a**

Table 1
^{1}H and ^{13}C (300 MHz) spectral data of **1**, **2**, **3a**, and **4** in $CDCl_3$

Position	**1**		**2**		**3a**		**4**	
	^{1}H (*J* in Hz)	^{13}C (δ)	^{1}H (*J* in Hz)	^{13}C (δ)	^{1}H (*J* in Hz)	^{13}C (δ)	^{1}H (*J* in Hz)	^{13}C (δ)
1-OH	12.97, s	155.5	13.15, s	160.2	13.02, s	158.4	12.79, s	158.1
2		104.2		111.2		103.9		109.1
3		158.4		158.9		159.6		161.1
4		107.4		100.2		107.3		104.9
5	7.30, d, 9.0	119.0	7.35, d, 8.7	118.9	7.46, d, 8.7	118.7	7.04, d, 9.0	118.7
6	7.23, dd, 2.4, 9.0	124.1	7.25, m	123.8	7.39, dd, 8.7, 2.7	128.6	7.07, br d, 9.0	124.7
7		150.5		150.3		146.3		150.1
8	7.60, d, 2.4	108.9	7.60, br d, 1.8	109.3	7.92, d, 2.7	117.8	7.44, br s	108.7
9		180.9		180.5		180.3		180.9
4a		154.5		150.2		152.4		152.9
4b		152.4		152.2		153.7		152.4
8a		120.6		121.1		121.2		120.2
9a		103.3		102.9		102.3		103.0
1′	6.74, d, 9.9	115.8	3.36, d, 7.2	21.2	2.75, br t, 6.9	16.2	3.32, d, 6.9	21.8
2′	5.60, d, 9.9	127.3	5.25, br t, 7.2	122.1	1.81, br t, 6.9	31.7	5.19, br t, 6.9	121.6
3′		78.1		131.5		76.3		134.8
4′	1.48, s	28.4	1.68, s	25.8	1.39, s	26.9	1.55, s	25.6
5′	1.48, s	28.4	1.81, s	17.9	1.39, s	26.9	1.75, s	17.9
1″	3.46, d, 7.2	21.3	6.88, d, 10.2	115.9	3.48, d, 7.5	21.5	3.39, d, 6.9	21.6
2″	5.22, br t, 7.2	122.1	5.54, d, 10.2	125.4	5.22, br t, 6.3	122.3	5.16, br t, 6.9	121.7
3″		135.0		80.6		134.9		137.9
4″	1.99, m[a]	39.7	1.89, m,[a] 1.68, m[a]	41.8	1.96, m	39.8	1.99, m[a]	39.7
5″	2.04, m[a]	26.6	2.12, m[a]	22.8	2.03, m	26.7	1.97, m[a]	26.4
6″	5.04, br t, 6.6	124.2	5.10, br t, 7.2	123.8	5.05, br t, 6.6	124.4	4.95, br t, 7.2	123.9
7″		131.3		131.9		131.3		131.8
8″	1.59, s	25.6	1.66, s	25.6	1.60, s	25.6	1.67, s	25.8
9″	1.86, s	16.3	1.44, s	27.1	1.86, s	16.3	1.78, s	16.2
10″	1.53, s	17.6	1.57, s	17.6	1.55, s	17.7	1.48, s	17.6
7-OAc					2.34, s	20.9/169.4		

[a] Deduced from HMQC experiment.

Table 2
HMBC (300 MHz) spectral data of **1**, **2**, **3a**, and **4** in $CDCl_3$

Position	1	2	3a	4
1-OH	C-1, C-2, C-9a	C-1, C-2, C-9a	C-1, C-2, C-9a	C-1, C-2, C-9, C-9a
5	C-7, C-9, C-4b, C8a	C-7, C8a	C-7, C-8a, C-9	C-7, C-9, C-4b, C8a
6	C-5, C-7, C-8, C-4b	C-7, C-4b	C-5, C-7, C-4b	C-7, C-8
8	C-6, C-7, C-9, C-4b	C-8	C-6, C-7, C-4b, C-9	C-6, C-7, C-9, C-4b
1′	C-1, C-2, C-3, C-4, C-3′	C-1, C-2, C-2′, C-3′	C-1, C-2, C-3, C-2′, C-3′	C-1, C-2, C-3, C-2′, C-3′ C-4′, C-5′
2′	C-2, C-3′, C-4′, C-5′	C-2′	C-2, C-1′, C-3′, C-4′, C-5′	C-2, C-1′, C-5′
4′	C-2′, C-3′, C-5′	C-2′, C-3′, C-5′	C-2′, C-3′	C-2′, C-3′, C-5′
5′	C-2′, C-3′, C-4′	C-2′, C-3′, C-4′	C-2′, C-3′	C-2′, C-3′, C-4′
1″	C-3, C-4, C-4a, C-2″, C-3″	C-3, C-4, C-4a, C-3″	C-3, C-4, C-4a, C-2″, C-3″	C-3, C-4, C-4a, C-2″, C-3″, C-9″
2″	C-4, C-1″, C-4″, C-9″	C-4, C-3″, C-4″, C-5″, C-9″	C-1″, C-3″, C-4″, C-9″	C-4, C-1″, C-4″, C-9″
4″	C-2″, C-3″, C-5″	C-2″, C-3″	C-2″, C-3″, C-6″	C-2″, C-6″
5″	C-3″, C-4″, C-6″, C-7″	C-6″, C-7″	C-3″, C-6″, C-7″	C-3″, C-4″, C-6″, C-7″
6″	C-4″, C-5″, C-8″, C-10″	C-6″	C-5″, C-8″, C-10″	C-4″, C-5″, C-8″
8″	C-6″, C-7″, C-10″	C-6″, C-7″, C-10″	C-6″, C-7″, C-10″	C-6″, C-7″, C-10″
9″	C-2″, C-3″	C-2″, C-3″, C-4″	C-2″,C-3″	C-2″, C-3″, C-4″
10″	C-6″, C-7″, C-8″	C-6″, C-7″, C-8″	C-6″, C-7″, C-10″	C-6″, C-7″, C-8″
7-OAc			C-7	

(5.1 mg). The latter was confirmed as an acetylated derivative of **2** by comparison of its spectral data with those of compound **2**.

Compound **3a** is a yellow powder. The HRMS spectrum showed a molecular ion peak at m/z 490.2355 $[M]^+$, corresponding to $C_{30}H_{34}O_6$. The UV spectrum showed absorption bands of a xanthone (243, 271, 303, 326, and 383 nm),[14] while the IR spectrum exhibited the hydroxyl and conjugated carbonyl functionalities at ν_{max} 3392 and 1648 cm^{-1}, respectively. The ^1H and ^{13}C NMR spectral data of **3a** (Table 1) were closely related to those of **1**, except for the appearance of the signals of a dimethylchromane ring and acetoxy group revealed at δ_H 2.75 (br t, J 6.9 Hz, H-1′), 1.81 (br t, J 6.9 Hz, H-2′), 1.39 (s, Me-4′ and Me-5′), and 2.34 (s, 7-OAc) (Table 1) instead of a chromene ring and a hydroxyl group in **1**. The chromane ring was fused to the xanthone nucleus in a linear fashion, which was confirmed by HMBC correlations (Table 2), in which the methylene protons H_2-1′ at δ_H 2.75 were correlated with C-1 (δ 158.4), C-2 (δ 103.9), C-3 (δ 159.6), C-2′ (δ 31.7), and C-3′ (δ 76.3), while the H-bonded hydroxyl proton 1-OH (δ 13.02) was correlated with C-1 (δ 158.4), C-2 (δ 103.9), and C-9a (δ 102.3). Therefore, the structure of **3a**, an acetylated form of **3**, was deduced. Compound **3** was named cochinchinone K.

It is interesting to note that the three new chromenoxanthones (**1** and **2**) and chromanoxanthone (**3**) could be derived from cochinchinone A (**4**). The isoprenyl or geranyl side chains of **4** were epoxidized and further cyclized via a free hydroxyl at C-3 to produce the chromanol ring[18] as shown in Scheme 1. Further dehydration led to the linear or angular chromene rings of **1** and **2**, respectively. On the other hand, the new chromanoxanthone (**3**) could be produced by cyclization via a free hydroxyl group to an isoprenyl side chain of **4** to afford the chromane ring (Scheme 1). Therefore, cochinchinone A (**4**) should be a precursor of cochinchinones I–K (**1**–**3**).

The investigation of the crude CH_2Cl_2 extract of the green fruits (approximate 4 weeks maturity stage) led to the isolation and identification of a new xanthone, cochinchinone L (**13**) along with two major known oxygeranyl xanthones **14**[14] and **15**.[19] Compounds **14** and **15** were structural isomers of xanthones with an oxygeranyl

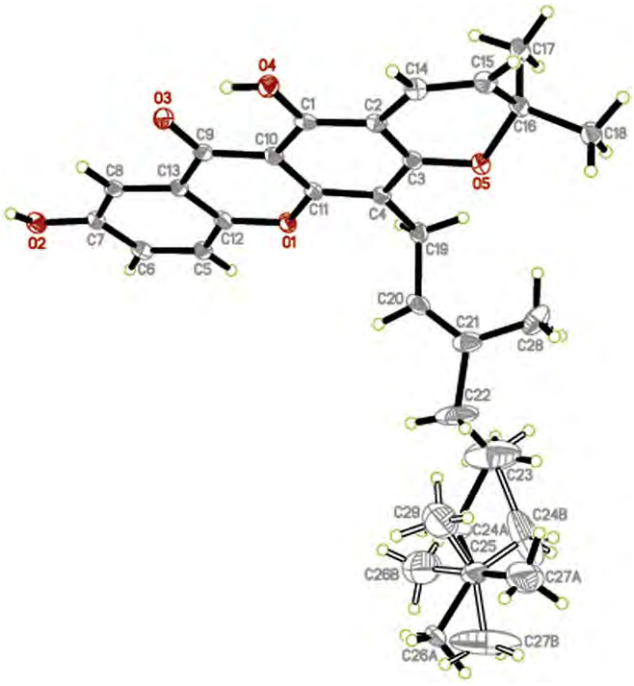


Figure 1a. The asymmetric unit of a cocrystal of **1** and **1a**.

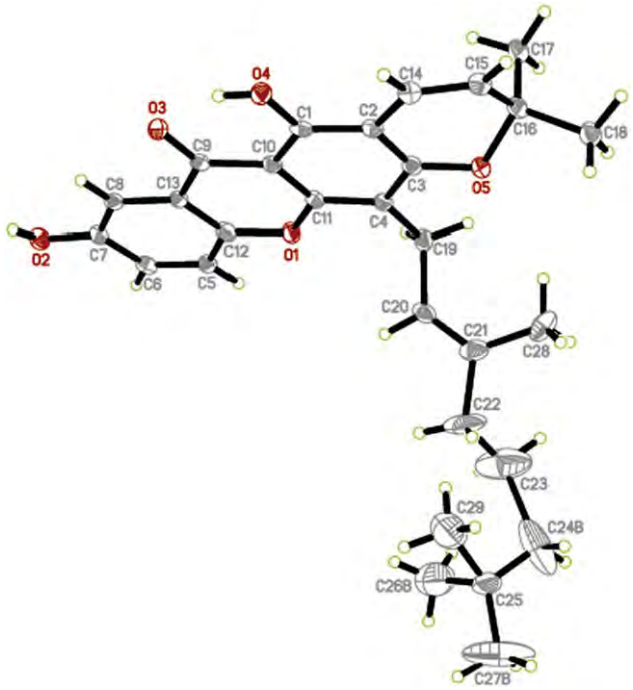
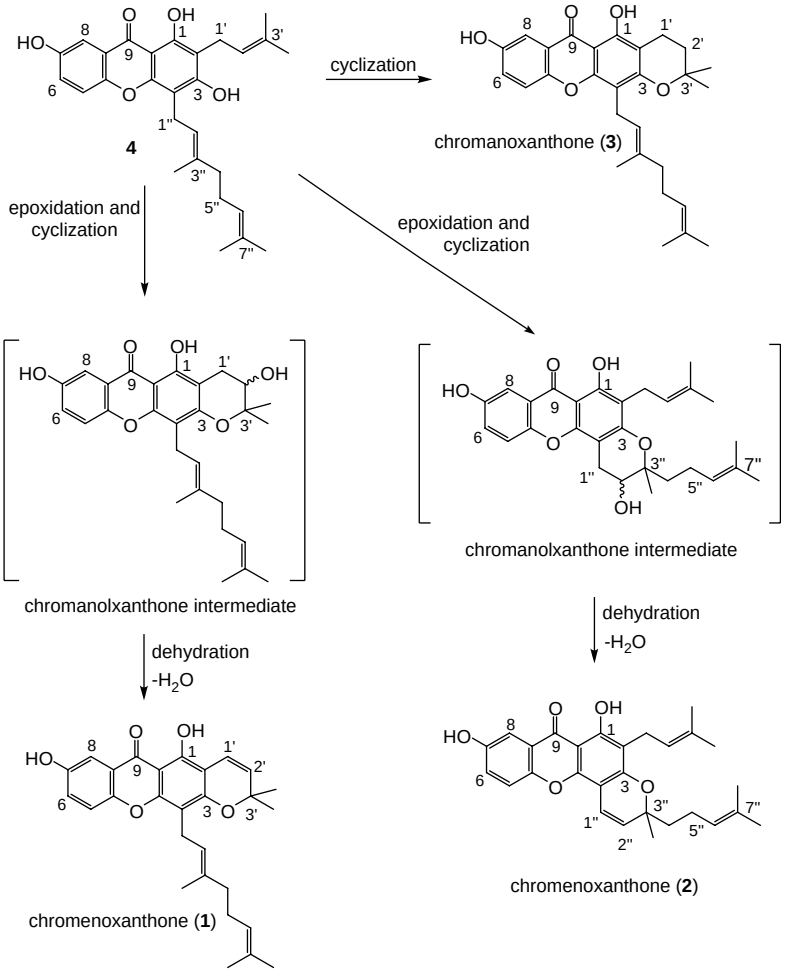


Figure 1b. The X-ray structure of **1a**.



Scheme 1. Plausible biosynthesis pathway of **1**, **2**, and **3**.

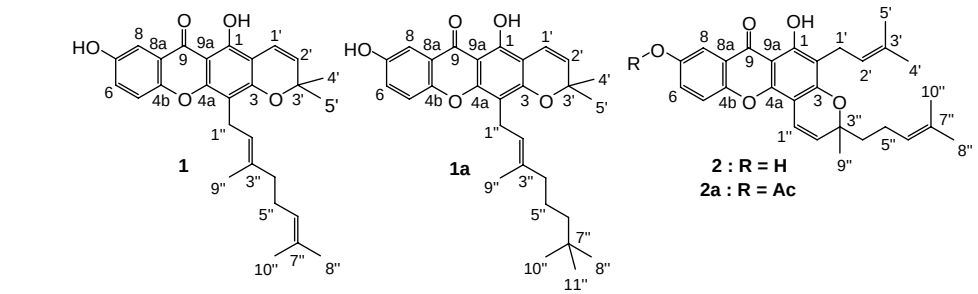
side chain. The ^{1}H and ^{13}C NMR spectral data of both **14** and **15** (Tables 3 and 4) were almost identical. However, the main difference was observed in the NOESY experiment in which H_2-1′ (δ 4.45) of **14** showed a cross-peak with H-8 (δ 7.40) and H-6 (δ 7.18), whereas the H_2-1′ (δ 4.63) of **15** showed a cross-peak with H-4 (δ 6.40) and H-2 (δ 6.34). These results implied that the oxygeranyl side chain of **14** was connected to C-7 and that of **15** to C-3. To confirm these structural assignments of **14** and **15**, compound **14** was further reacted with *p*-bromobenzenesulfonyl chloride in CH_2Cl_2 at room temperature to afford a crystalline dibrosylate **20**

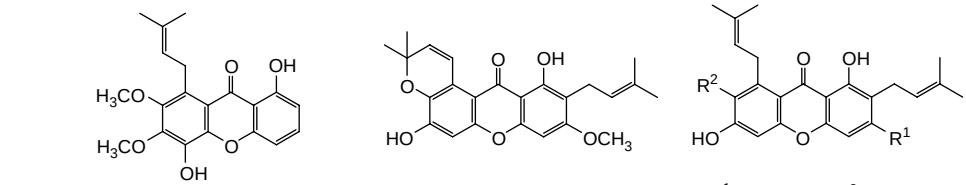
Table 3
^{1}H NMR (300 MHz) spectral data of **13**–**19** in $CDCl_3$

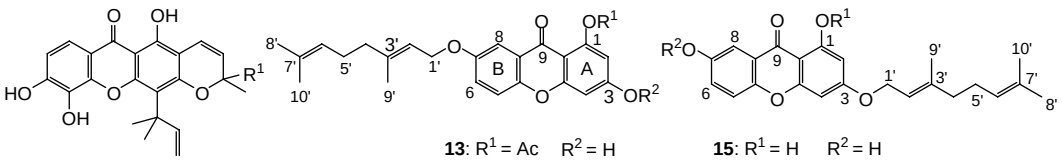
Position	13	14	15	16	17	18	19
2	6.43, d, 2.1	6.22, d, 1.8	6.34, d, 2.4	6.42, br d, 2.1	7.15, d, 2.1	6.19, br d, 2.1	6.41, d, 2.1
4	6.57, d, 2.1	6.23, d, 1.8	6.40, d, 2.4	6.59, br d, 2.1	6.79, d, 2.1	6.24, br d, 2.1	6.46, d, 2.1
5	7.08, br d, 9.0	7.15, br d, 9.0	7.30, d, 9.3	7.21, br d, 8.1	7.23, d, 9.3	7.25, d, 9.3	7.25, d, 8.1
6	7.10, br d, 9.0	7.18, br d, 9.0	7.26, d, 9.3	7.20, br d, 8.1	7.28, dd, 9.3, 2.1	7.29, br d, 9.3	7.26, br d, 8.1
8	7.45, br s	7.40, br s	7.59, br s	7.44, br s	7.55, d, 2.1	7.76, br s	7.78, br d, 1.5
1′	4.42, d, 6.3	4.45, d, 6.3	4.63, d, 6.6	4.50, d, 6.6	4.56, d, 6.3	4.49, d, 6.3	4.51, d, 6.3
2′	5.73, t, 6.3	5.39, br t, 6.3	5.50, br t, 6.6	5.40, br t, 6.6	5.48, br t, 6.3	5.37, br t, 6.3	5.37, br t, 6.3
4′	1.99, m	2.01, m	2.13, m	2.03, m	2.11, m	2.03, m	2.03, m
5′	1.97, m	1.95, m	2.10, m	2.02, m	2.09, m	1.97, m	2.01, m
6′	4.98, t, 6.6	4.98, br t, 5.7	5.11, br t, 5.7	5.00, br t, 6.3	5.09, br t, 6.3	4.99, br t, 6.9	4.99, br t, 6.6
8′	1.57, s	1.57, s	1.69, s	1.57, s	1.66, s	1.57, s	1.57, s
9′	1.60, s	1.64, s	1.78, s	1.68, s	1.73, s	1.66, s	1.65, s
10′	1.50, s	1.50, s	1.62, s	1.51, s	1.59, s	1.51, s	1.51, s
1-OAc	2.38, s	—	—	—	—	—	—
1-OH	—	12.85, s	12.72, s	12.71, s	—	12.55, s	—
3-OH	—	7.86, br s		—	—	—	—
7-OH	—	—	7.03, br s	—	—	—	—
1-OAc	—	—	—	—	2.47, s	—	2.40, s
3-OAc	—	—	—	2.23, s	2.27, s	—	—
7-OAc	—	—	—	—	—	2.22, s	2.19, s

(Fig. 3), while compound **15** was acetylated in the usual manner with Ac_2O in pyridine to give a crystalline monoacetate **18** (Fig. 2). Both **20** and **18** were subjected to X-ray diffraction analysis whose results supported the structures of **14** and **15**.

Cochinchinone L (**13**) was isolated as a yellow powder, with a molecular ion peak at m/z 422.1718 $[M]^+$ in the HRMS, corresponding to a molecular formula of $C_{25}H_{26}O_6$. The ^1H and ^{13}C NMR spectral data of **13** (Tables 3 and 4) showed characteristic signals similar to those of 7-geranyloxy-1,3-dihydroxyxanthone (**14**),[14] except that the hydroxyl group at C-1 of **14** was replaced by an acetoxy group in **13**, which appeared as a proton singlet signal at δ 2.38. The higher field chemical shift of C-1 (δ 151.2) and lower field resonances of protons and carbons of C-2 (δ_H 6.43 (d, J 2.1 Hz, H-2); δ_C 108.3) and C-4 (δ_H 6.57 (d, J 2.1 Hz, H-4); δ_C 101.3) as compared to those of **14** (δ_H 6.22 (d, J 1.8 Hz, H-2); δ_C 98.5 and δ_H 6.23 (d, J 1.8 Hz, H-4); δ_C 94.4) supported the title structure (Tables 3 and 4). The ^4J HMBC correlations of methyl protons of the acetoxy group (δ 2.38) with C-1 (δ 151.2) supported its location at C-1, which was also confirmed by NOESY cross-peak between acetoxy moiety with H-2. Cochinchinone L (**13**), an acetylated analogue of







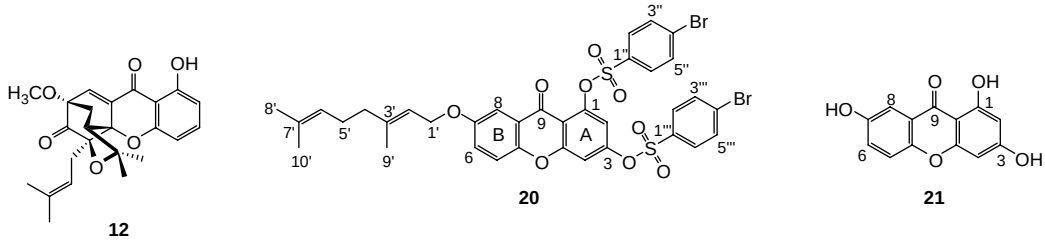


Table 4
^{13}C NMR (75 MHz) spectroscopic data of **13**–**19** in $CDCl_3$

Position	13	14	15	16	17	18	19
1	151.2	163.8	163.2	162.8	150.9	163.3	158.7
2	108.3	98.5	97.6	104.0	108.7	97.8	99.4
3	162.6	164.0	166.1	156.8[a]	154.5	166.2	163.6
4	101.3	94.4	93.2	100.7	112.4	93.4	108.1
5	118.8	118.9	188.9	119.0	118.9	118.7	118.6
6	125.1	125.5	124.2	126.0	125.2	128.9	128.3
7	155.4	155.2	152.5	155.4	155.5	146.5	146.7
8	106.4	105.9	109.0	106.0	106.6	117.8	118.4
9	175.7	180.5	180.5	181.1	174.7	179.8	174.1
4a	158.8	157.8	157.8	156.7[a]	157.7	157.5	151.4
4b	150.0	150.7	150.5	150.8	149.9	153.3	152.7
8a	122.2	120.3	120.9	120.7	122.3	121.1	123.6
9a	107.8	103.3	103.5	106.6	112.2	103.4	108.6
1′	65.51	65.6	65.6	65.6	65.5	65.6	65.8
2′	118.8	118.6	118.3	118.8	118.8	118.4	118.0
3′	141.9	142.1	142.3	141.9	141.5	142.2	142.6
4′	39.5	39.5	39.5	39.5	39.5	39.5	39.5
5′	26.3	26.3	26.2	26.3	26.3	26.2	26.2
6′	123.8	123.8	123.6	123.7	123.8	123.9	123.6
7′	131.8	131.8	131.9	131.8	131.7	131.9	131.9
8′	25.7	26.3	25.6	25.6	25.6	25.6	25.6
9′	16.7	16.7	16.7	16.7	16.7	16.7	16.7
10′	17.7	17.7	17.7	17.7	17.6	17.7	17.7
1-OAc	171.0	—	—	—	21.1	—	21.1
	21.3	—	—	—	169.2	—	169.5
3-OAc	—	—	—	21.1	21.0	—	—
	—	—	—	168.2	167.8	—	—
7-OAc	—	—	—	—	—	20.9	20.9
	—	—	—	—	—	169.2	169.2

[a] May be interchangeable.

14, was therefore assigned as 1-acetoxy-3-hydroxy-7-geranyloxyxanthone.

The isolated compounds were evaluated for their antibacterial activities against both Gram-positive bacteria: *Bacillus subtilis*, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR 459, Methicillin-Resistant *S. aureus* (MRSA) ATCC 43300, Vancomycin-Resistant *E. faecalis* (VRE) ATCC 51299 and Gram-negative bacteria: *Salmonella typhi*, *Shigella sonei*, and *P. aeruginosa*. All compounds were also subjected to antifungal assay against *Candida albicans*. The results showed that most of the isolated compounds (**4**–**6** and **13**–**15**) exhibited strong antibacterial activity specifically against *P. aeruginosa* (Table 5). Interestingly, only compounds **9** and **10** exhibited strong activity against *C. albicans*. It is important to note that compound **9** also exhibited broad spectrum antimicrobial activity against all Gram-positive bacteria. Nearly all compounds, which are active against *P. aeruginosa* are the 1,3,7-trihydroxy xanthones (**4** and **5**) or 1,3,7-trioxygenated xanthones with an oxygeranyl side chain either at C-3 or C-7 and dihydroxyl groups (**14**–**15**) whose indicated structures might contribute to the strong antibacterial activity specifically against *P. aeruginosa*. However, when the free hydroxyl group of the 1,3,7-trihydroxy xanthone was cyclized onto the isoprenyl or geranyl side chain to form a chromane or chromene ring, the antibacterial activity against *P. aeruginosa* decreased drastically as shown in compounds **1**, **2**, and **3a**. In other previous reports, it was suggested that hydroxy xanthones play important roles in biological activity such as antibacterial,[3,5] α-glucosidase inhibitory,[20] anti-tumor,[21,22] and anti-inflammatory effects.[23] Because compounds **14** and **15** are the major components obtained from this plant, this prompts us to modify their structures for the structure–activity relationships (SARs). To investigate whether the free hydroxyl group was responsible for antibacterial activity, the acetylation with acetic anhydride in pyridine was therefore applied to **14** and **15**. Four acetylated geranyloxyxanthone derivatives: 3-acetoxy-7-geranyloxy-1-hydroxyxanthone (**16**) and 1,3-diacetoxy-7-geranyloxyxanthone (**17**) were obtained from **14**, whereas 7-acetoxy-3-geranyloxy-1-hydroxy-xanthone (**18**) and 1,7-diacetoxy-3-geranyloxy-xanthone (**19**) were obtained from **15**. The assignments of the ^{1}H and ^{13}C NMR spectral data of **16**–**19** are summarized in Tables 3 and 4. The antibacterial activity of acetylated geranyloxyxanthone derivatives **16**–**19** and dibrosylate **20** was evaluated. All of them showed strong anti-*P. aeruginosa* (Table 5). This implied that the free hydroxyl groups should not be responsible for the inhibition of *P. aeruginosa*. For further investigation of the role of an oxygeranyl side chain, a 1,3,7-trihydroxyxanthone (**21**) obtained from the dried fruits of *C. cochinchinense*[24] was tested against *P. aeruginosa*. It was inactive against Gram-negative bacteria as shown in Table 5. From these results, it can be suggested that the geranyl side chain is necessary for anti-*P. aeruginosa* activity. Moreover, mixtures of compound **4** with compound **5**, and compound **14** with compound **15** were subjected to antimicrobial assay. Interestingly, the mixture of compounds **14** and **15** significantly increased the antibacterial activity against MRSA compared with the pure forms as indicated by the lower MIC values shown in Table 5. The mixture of compounds **4** and **5**, on the other hand, did not show any significant differences for antibacterial activity compared to the pure forms as also shown in Table 5. Therefore, it may be proposed that the 1,3,7-trihydroxyxanthone with the isoprenyl or geranyl side chain at C-2 and C-4 in

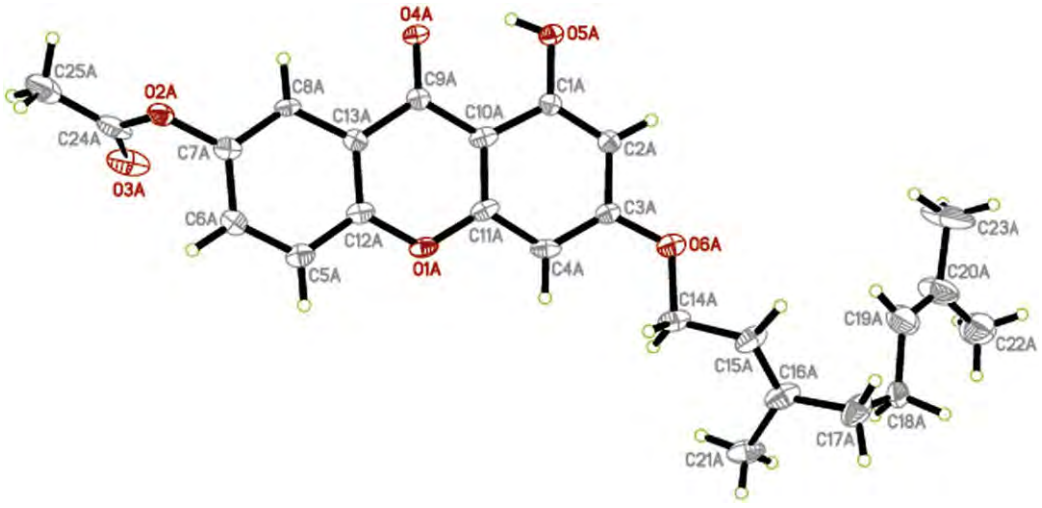


Figure 2. ORTEP plot of a monoacetate **18**.

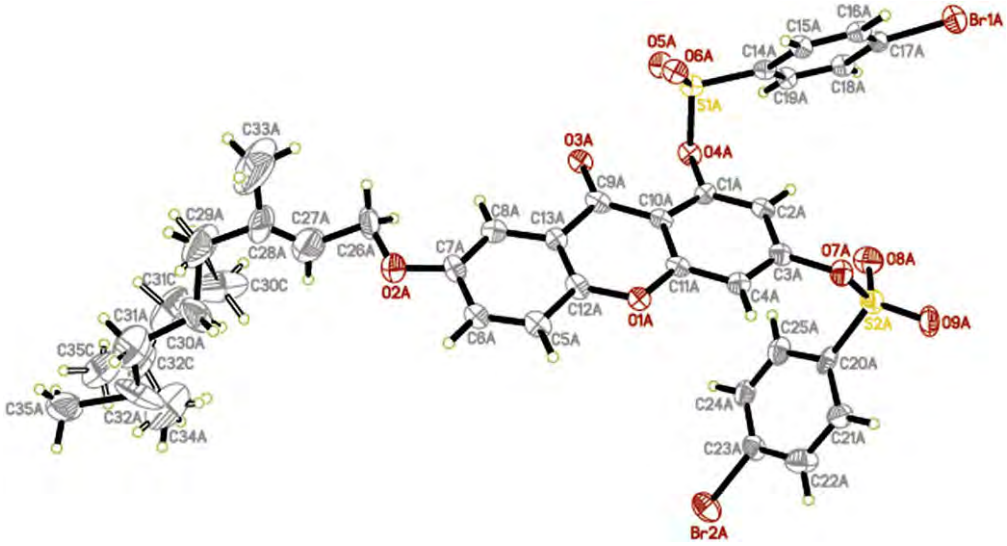


Figure 3. ORTEP plot of a dibrosylate **20**.

4 and **5**, respectively, and 1,3,7-trioxygenated xanthone with the geranyl side chain at C-3 or C-7 in **13**–**19** are essential for their antibacterial activity against *P. aeruginosa*. Therefore, 1,3,7-trihydroxyxanthones (**4** and **5**) and 1,3,7-trioxygenated xanthone with geranyl side chain (**13**–**19**) should be considered as potent candidates as anti-*P. aeruginosa*.

We further studied the possible mode of action of compounds **4** and **13** against *P. aeruginosa* by observing the bacteria cell morphology through scanning electron microscopy (SEM) at 3, 6, 9, and 15 h after applying compounds **4** and **13**. From the SEM results (see Figs 4 and 5), it was clearly indicated that the cell morphology of *P. aeruginosa*, when treated with compounds **4** and **13**, started to deform at 3 h onward, and at 15 h, most cells were completely deformed whose results was correlated to their strong antibacterial activity (Table 5). Therefore, it can be suggested that compounds **4** and **13** may interact with or damage the cell wall of *P. aeruginosa* as seen by the formation of pores on the cell wall of *P. aeruginosa* (Figs 4 and 5).

3. Experimental

3.1. General experimental procedures

Melting points were determined on a Fisher–John melting point apparatus. Optical rotations were measured on a JASCO P-1020 digital polarimeter. UV and IR spectra were recorded on SPECORD S 100 (Analytikjena) and Perkin–Elmer FTS FT-IR spectrophotometer,

Table 5
Antimicrobial activity (MIC, μg/ml) of **1**–**21**

No.	Antibacterial activity								Antifungal activity
	Gram-positive bacteria[a]					Gram-negative bacteria [b]			
	B. subtilis	*S. aureus*	*E. faecalis*	MRSA	VRE	*S. typhi*	*S. sonei*	*P. aeruginosa*	*C. albicans*[c]
1	>300	>300	>300	>300	>300	>300	>300	>300	>300
2	300	300	>300	300	300	>300	>300	300	75
3a	75	>300	300	>300	>300	>300	>300	>300	300
4	150	150	150	9.37	150	>150	>150	4.7	75
5	150	75	150	37.5	75	>150	>150	4.7	37.5
4+5	75	150	75	9.37	150	>150	>150	4.7	75
6	>150	>150	>150	150	150	>150	>150	4.7	150
7	150	300	300	150	150	>300	>300	150	300
8	300	75	150	75	150	>300	>300	300	150
9	9.37	9.37	9.37	9.37	9.37	>300	>300	18.75	2.4
10	18.75	37.5	37.5	37.5	37.5	>300	>300	37.5	4.7
11	>300	300	>300	150	150	>300	>300	>300	300
12	75	>300	300	150	150	>300	>300	>300	>300
13	150	>150	150	37.5	150	>150	>150	4.7	18.75
14	150	>150	150	18.7	150	>150	>150	4.7	75
15	150	150	150	9.3	150	>150	>150	4.7	37.5
14+15	75	37.5	37.5	4.7	37.5	>150	>150	4.7	37.5
16	150	>150	150	18.75	150	>150	>150	4.7	150
17	75	>150	75	37.5	150	>150	>150	4.7	18.75
18	>150	>150	150	18.75	150	>150	>150	4.7	150
19	>150	>150	>150	37.5	150	>150	>150	4.7	150
20	>150	>150	150	300	>300	>150	>150	4.7	>300
21	37.5	75	>300	150	300	>300	>300	300	300

[a] *B. subtilis*, *S. aureus*, *E. faecalis* TISTR 459, Methicillin-Resistant *S. aureus* (MRSA) ATCC 43300, Vancomycin-Resistant *E. faecalis* (VRE) ATCC 51299.
[b] *S. typhi*, *S. sonei*, and *P. aeruginosa*.
[c] *C. albicans*.

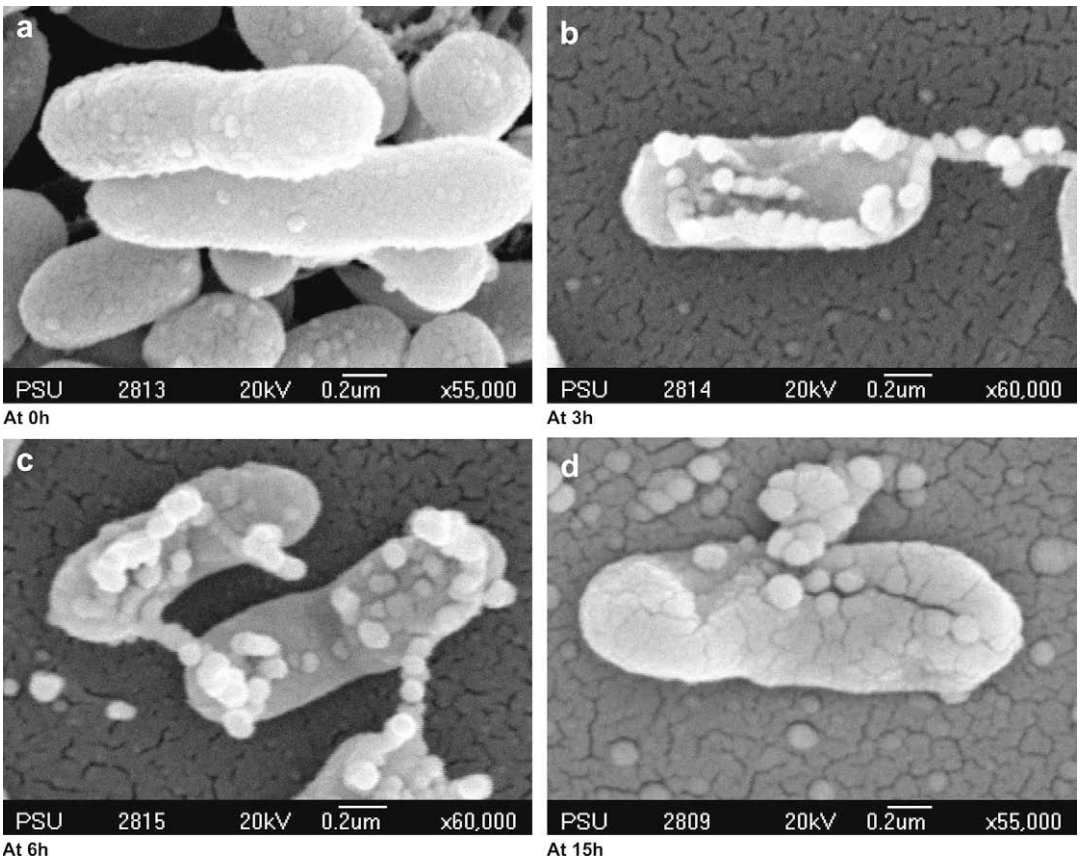


Figure 4. Scanning electron microscopy of cell morphology of *P. aeruginosa* treated with compound **4** at different time.

respectively. The ^{1}H and ^{13}C NMR spectra were recorded on 300 MHz Bruker FTNMR Ultra Shield™ spectrometer in $CDCl_3$ with TMS as the internal standard. Chemical shifts are reported in δ (ppm) and coupling constants (J) are expressed in hertz. EI and HREI mass spectra were measured on a Kratos MS 25 RFA spectrometer. Quick column chromatography (QCC) and column chromatography (CC) were carried out on silica gel 60 F_{254} (Merck) and silica gel 100 (Merck), respectively. All the bacteria images were viewed with a JSM-5800LV, JEOL SEM (scanning electron microscope).

3.2. Plant material

The resin of *C. cochinchinense* was collected in October 2003 at Prince of Songkla University, Hat-Yai campus, whereas the green fruits of *C. cochinchinense* were collected in October 2007 at Kaun Kha Long district, Satun Province, Southern part of Thailand. Botanical identification was achieved through comparison with a voucher specimen No. SL-1 (PSU) in the herbarium of Department of Biology, Prince of Songkla University, Songkhla, Thailand.

3.3. Isolation and extraction

The resin of *C. cochinchinense* (87.75 g) was extracted with CH_2Cl_2 (2×2.0 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (47.04 g), which was subjected to QCC (Quick column chromatography) on silica gel (Merck 60 F_{254}) using hexane as a first eluent and then increasing the polarity with acetone to give 16 fractions (FR1–FR16). Fractions FR4 and FR5 were separated by CC eluting with a gradient of acetone–hexane to give eight subfractions (FR4A–FR4H) and **8** (150.2 mg) (R_f (15% acetone–hexane (three runs)) 0.39). Subfraction FR4B was further purified by CC and eluted with a gradient of acetone–hexane to give **7** (31.4 mg) (R_f (15% acetone–hexane) 0.31) and **12** (56.5 mg) (R_f (15% acetone–hexane) 0.28). Fractions FR6 and FR7 were separated by QCC and eluted with a gradient of CH_2Cl_2–hexane to give six subfractions (FR6A–FR6F). Subfraction FR6B was further separated by QCC eluting with a gradient of acetone–hexane to give **6** (35.7 mg) (R_f (15% acetone–hexane (three runs)) 0.41), **8** (83.2 mg) (R_f (15% acetone–hexane (three runs)) 0.39), and **11** (1.8 mg) (R_f (15% acetone–hexane (three runs)) 0.24). Subfraction FR6E was purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give **4** (849.4 mg) (R_f (MeOH) 0.54) and **5** (1.25 g) (R_f (MeOH) 0.62). Fractions FR8 and FR9 were separated by QCC and eluted with a gradient of CH_2Cl_2–hexane to afford **5** (551.3 mg) (R_f (60% CH_2Cl_2–hexane (three runs)) 0.05), **7** (18.2 mg) (R_f (60% CH_2Cl_2–hexane (three runs)) 0.29), **8** (116.6 mg) (R_f (60% CH_2Cl_2–hexane (three runs)) 0.61), and **9** (148.7 mg) (R_f (60% CH_2Cl_2–hexane (three runs)) 0.10). Fractions FR10 and FR11 were separated by QCC and eluted with a gradient of acetone–hexane to give seven subfractions (FR10A–FR10G) and **7** (25.9 mg) (R_f (15% acetone–hexane) 0.31). Subfraction FR10B was further purified by CC on silica gel C-18 and eluted with MeOH to furnish **1** (8.5 mg) (R_f (MeOH) 0.36). The mixture of **1** and **1a** could not be separated due to the very low amount of **1a**. The purity of **1** is much more than 90% base on a single spot on TLC and the integral area signal in the ^{1}H NMR spectrum of **1**. Subfraction FR10D was separated by QCC eluting with a gradient of acetone–hexane to give six subfractions (FR10D1–FR10D6). Subfraction FR10D2 was further separated by CC and eluted with a gradient of acetone–hexane to give five subfractions (FR10D2A–FR10D2E), **2** (1.8 mg) (R_f (20% acetone–hexane)

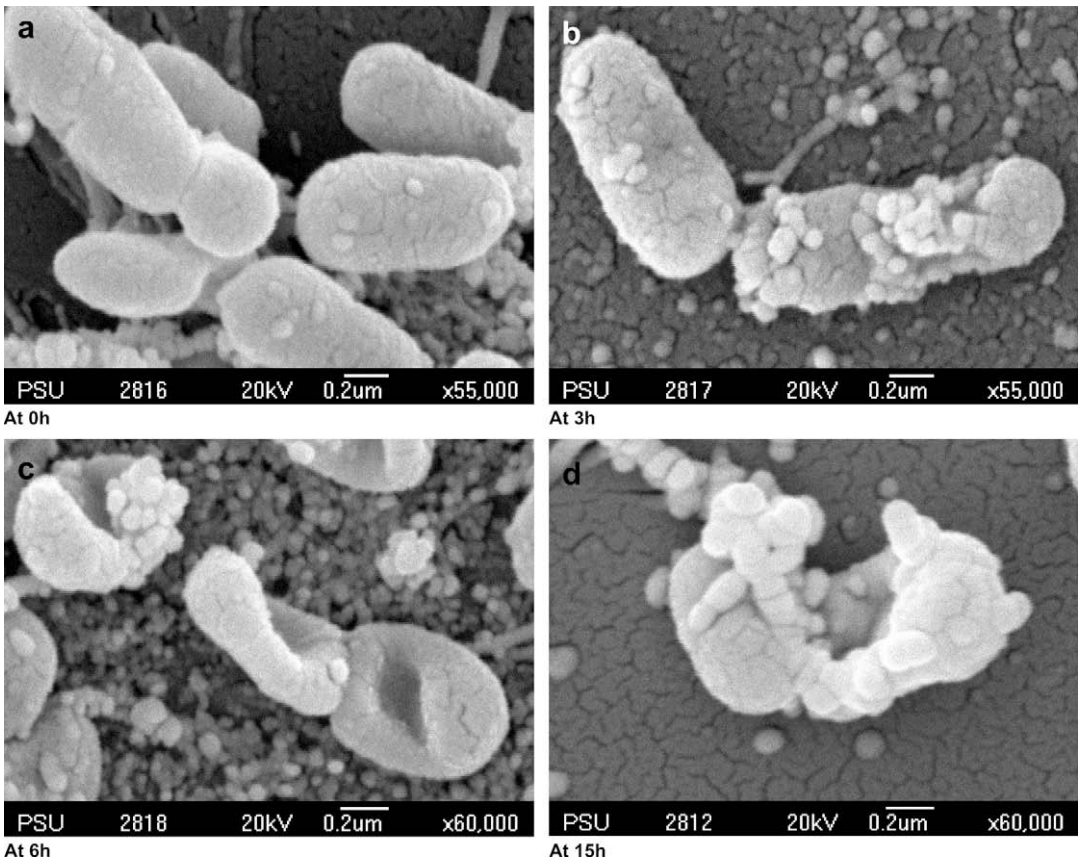


Figure 5. Scanning electron microscopy of cell morphology of *P. aeruginosa* treated with compound **13** at different time.

0.36), **4** (23.1 mg) (R_f (20% acetone–hexane) 0.22), **5** (34.2 mg) (R_f (20% acetone–hexane) 0.17), and an inseparable mixture of **2** and **3** (20.2 mg) (R_f (20% acetone–hexane) 0.35). The mixture was separated by acetylation with Ac_2O (0.1 ml) in pyridine (2.0 ml) and stirred overnight at room temperature to give a yellow gum, which was further purified by CC eluting with 70% $CHCl_3$–hexane to give acetylated derivatives **2a** (5.1 mg) (R_f (70% $CHCl_3$–hexane) 0.68) and **3a** (18.9 mg) (R_f (70% $CHCl_3$-hexane) 0.40), respectively. Subfraction FR10D2E was purified by CC and eluted with 80% CH_2Cl_2–hexane to give **8** (16.7 mg) (R_f (80% CH_2Cl_2–hexane) 0.91), **10** (5.0 mg) (R_f (80% CH_2Cl_2–hexane) 0.85), and **11** (1.5 mg) (R_f (80% CH_2Cl_2–hexane) 0.38).

Air-dried green fruits of *C. cochinchinense* (5.5 kg) were extracted with CH_2Cl_2 (2×20 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (40.04 g), which was subjected to QCC on silica gel using hexane as a first eluent and increasing polarity with EtOAc to give nine fractions (FS1–FS9). Fraction FS5 was purified by CC eluting with pure $CHCl_3$ to give **14** (1.88 g) (R_f (70% $CHCl_3$–hexane) (three runs) 0.46). Fraction FS6 was further separated by CC eluting with pure $CHCl_3$ to furnish six subfractions (FS6A–FS6F), **14** (2.10 g) (R_f (70% $CHCl_3$–hexane) (three runs) 0.46) and **15** (490.2 mg) (R_f (70% $CHCl_3$–hexane) (three runs) 0.39). Subfraction FS6B was further purified by CC eluting with a gradient of acetone–hexane to give **13** (53.3 mg) (R_f (15% acetone–hexane (three runs)) 0.17).

3.3.1. *Cochinchinone I* (***1***)

Yellow needle single crystals, mp 160–162 °C; UV ($CHCl_3$) λ_{max} (log ε) 261 (4.01), 297 (4.31), 342 (3.69), 393 (3.46) nm; IR (neat) ν_{max} 3406, 1650, 1612 cm^{-1}; HRMS *m*/*z* 446.2279 for $C_{28}H_{30}O_5$ (calcd 446.2093). EIMS *m*/*z* (rel int.): 446 $[M]^+$ (76), 431 (100), 377 (73), 363 (76), 323 (26), 307 (15), 295 (13), 137 (5), 69 (6). For ^1H and ^{13}C NMR spectroscopic data, see Table 1.

3.3.2. *Cochinchinone J* (***2***)

Yellow viscous oil; $[\alpha]_D^{25}$ −69.8 (*c* 0.08, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ε) 243 (3.90), 289 (4.10), 298 (4.13), 320 (3.68), 351 (3.47), 391 (3.33) nm; IR (neat) ν_{max} 3397, 1649, 1613 cm^{-1}; HRMS *m*/*z* 446.2092 for $C_{28}H_{30}O_5$ (calcd 446.2093). EIMS *m*/*z* (rel int.): 446 $[M]^+$ (11), 363 (100), 307 (18), 69 (5). For ^1H and ^{13}C NMR spectroscopic data, see Table 1.

3.3.3. *Acetylated derivative of cochinchinone K* (***3a***)

Yellow powder, mp 85–87 °C; UV ($CHCl_3$) λ_{max} (log ε) 243, 271, 303, 326, 383 nm; IR (neat) ν_{max} 3392, 1709, 1648, 1612 cm^{-1}; HRMS *m*/*z* 490.2355 for $C_{30}H_{34}O_6$ (calcd 490.2355). EIMS *m*/*z* (rel int.): 490 $[M]^+$ (53), 473 (53), 419 (44), 405 (31), 365 (100), 323 (52), 311 (41), 267 (51), 69 (24). For ^1H and ^{13}C NMR spectroscopic data, see Table 1.

3.3.4. *Cochinchinone L* (***13***)

Yellow powder, mp 114–116 °C; UV ($CHCl_3$) λ_{max} (log ε) 248 (4.59), 273 (4.03), 305 (4.13), 354 (3.80) nm; IR (neat) ν_{max} 3237, 1774, 1728, 1628 cm^{-1}; HRMS *m*/*z* 422.1718 for $C_{25}H_{26}O_6$ (calcd 422.1729). EIMS *m*/*z* (rel int.): 422 $[M]^+$ (1), 286 (40), 244 (100), 187 (4), 81 (9), 69 (28). For ^1H and ^{13}C NMR spectroscopic data, see Tables 3 and 4.

3.4. Acetylation of 14 and 15

Compound **14** (82.5 mg) was treated with Ac_2O (2.5 ml) in pyridine (2.0 ml) and stirred for 6 h at room temperature. The reaction mixture was diluted with water and extracted with CH_2Cl_2.

The combined organic extract was washed with 10% HCl and then washed with water again. After the organic solvent was removed, the resulting residue was dried over anhydrous Na_2SO_4. Chromatography over silica gel yielded a pale yellow powder of **16** (80.6 mg) (R_f (5% acetone–hexane (three runs)) 0.44).

Compound **14** (200.5 mg) was treated with Ac_2O (6.0 ml) in pyridine (3.0 ml) and stirred overnight at room temperature. Chromatography over silica gel yielded a pale yellow powder of **16** (10.6 mg) (R_f (5% acetone–hexane (three runs)) 0.44) and **17** (177.8 mg) (R_f (15% acetone–hexane (three runs)) 0.46).

Compound **15** (85.5 mg) was treated with Ac_2O (2.5 ml) in pyridine (2.0 ml) and stirred for 6 h at room temperature. Chromatography over silica gel yielded a pale yellow powder of **18** (83.7 mg) (R_f (5% acetone–hexane (three runs)) 0.32), which was further recrystallized from MeOH–acetone (1:99 v/v) to give yellow single crystals.

Compound **15** (190.0 mg) was treated with Ac_2O (6.0 ml) in pyridine (3.0 ml) and stirred overnight at room temperature. Chromatography over silica gel yielded a pale yellow powder of **18** (8.7 mg) (R_f (5% acetone–hexane (three runs)) 0.32) and **19** (170.0 mg) (R_f (15% acetone–hexane (three runs)) 0.37).

*3.4.1. 3-Acetoxy-7-geranyloxy-1-hydroxyxanthone (**16**)*

Yellow powder, mp 94–95 °C; UV ($CHCl_3$) λ_{max} (log ε) 239 (4.40), 262 (4.66), 289 (3.97), 379 (3.93) nm; IR (neat) ν_{max} 3429, 1768, 1649, 1612 cm^{-1}; HRMS *m/z* 422.1725 for $C_{25}H_{26}O_6$ (calcd 422.1729). EIMS *m/z* (rel int.): 422 $[M]^+$ (1), 286 (43), 244 (100), 187 (4), 81 (9), 69 (24). For ^1H and ^{13}C NMR spectroscopic data, see Tables 3 and 4.

*3.4.2. 1,3-Diacetoxy-7-geranyloxyxanthone (**17**)*

Yellow powder, mp 96–97 °C; UV ($CHCl_3$) λ_{max} (log ε) 253 (4.59), 300 (3.45), 361 (3.86) nm; IR (neat) ν_{max} 3429, 1776, 1656, 1624 cm^{-1}; HRMS *m/z* 464.1838 for $C_{27}H_{28}O_7$ (calcd 464.1835). EIMS *m/z* (rel int.): 464 $[M]^+$ (2), 328 (3), 286 (58), 244 (100), 187 (5), 81 (17), 69 (36). For ^1H and ^{13}C NMR spectroscopic data, see Tables 3 and 4.

*3.4.3. 7-Acetoxy-3-geranyloxy-1-hydroxy-xanthone (**18**)*

Yellow powder, mp 104–106 °C; UV ($CHCl_3$) λ_{max} (log ε) 243 (4.44), 257 (4.52), 310 (4.29), 358 (3.82) nm; IR (neat) ν_{max} 3429, 1758, 1665, 1607 cm^{-1}; HRMS *m/z* 422.1726 for $C_{25}H_{26}O_6$ (calcd 422.1729). EIMS *m/z* (rel int.): 422 $[M]^+$ (5), 286 (16), 244 (100), 187 (2), 81 (16), 69 (56). For ^1H and ^{13}C NMR spectroscopic data, see Tables 3 and 4.

*3.4.4. 1,7-Diacetoxy-3-geranyloxyxanthone (**19**)*

Yellow powder, mp 85–87 °C; UV ($CHCl_3$) λ_{max} (log ε) 246 (4.61), 275 (4.01), 302 (4.28), 334 (3.86) nm; IR (neat) ν_{max} 3453, 1770, 1655, 1629 cm^{-1}; HRMS *m/z* 464.1834 for $C_{27}H_{28}O_7$ (calcd 464.1835). EIMS *m/z* (rel int.): 464 $[M]^+$ (4), 328 (4), 286 (32), 244 (100), 187 (2), 81 (24), 69(73). For ^1H and ^{13}C NMR spectroscopic data, see Tables 3 and 4.

3.5. Brosylation of 14

Compound **14** (40.0 mg, 105.14 mmol) was stirred overnight at room temperature with *p*-bromobenzenesulfonyl chloride (40.30 mg, 190.2 mmol) and K_2CO_3 (44.1 mg, 315.4 mmol) in CH_2Cl_2 (3 ml). After the reaction was complete, water (10 ml) was added to the reaction mixture. The resulting solution was then extracted with CH_2Cl_2 (10 ml, three times). The combined organic extract was dried over anhydrous sodium sulfate and evaporated under reduced pressure to give a crude extract, which was further purified by column chromatography over silica gel eluting with 5% acetone–hexane to yield the dibrosylate **20** (75.2 mg) (R_f (5% acetone–hexane (three runs)) 0.20). Yellow needle-shaped single crystals of **20** were obtained after recrystallization from CH_3OH–$CHCl_3$ (1:4 v/v), mp 106–108 °C. δ_H (300 MHz, $CDCl_3$+CD_3OD) 7.82 (dd, *J* 9.0, 2.1 Hz, H-2″, H-6″), 7.68 (d, *J* 2.4 Hz, H-4), 7.65 (dd, *J* 8.7, 2.1 Hz, H-3″, H-5″, H-2‴, H-6‴), 7.63 (dd, *J* 8.7, 2.1 Hz, H-3‴, H-5‴), 7.50 (d, *J* 2.4 Hz, H-8), 7.32 (d, *J* 2.4 Hz, H-2), 7.26 (d, *J* 9.0 Hz, H-5), 7.19 (dd, *J* 9.3, 2.4 Hz, H-6), 5.44 (br t, *J* 6.6, H-2′), 5.04 (br t, *J* 6.3 Hz, H-6′), 4.56 (d, *J* 6.6 Hz, H-1′), 2.07 (m, 2H-5′), 2.05 (m, 2H-4′), 1.71 (s, 3H-9′),1.60 (s, 3H-8′), 1.54 (s, 3H-10′); δ_C (75 MHz, $CDCl_3$+CD_3OD) 181.6 (C-9), 157.4 (C-1), 155.9 (C-7), 151.9 (C-3), 149.7 (C-4b), 148.6 (C-4a), 142.1 (C-3′), 134.3 (C-1″), 133.4 (C-1‴), 133.1 (C-3″, C-5″), 132.5 (C-3‴, C-5‴), 131.8 (C-7′), 130.6 (C-4″), 130.2 (C-2″, C-6″), 130.1 (C-4‴), 129.8 (C-2‴, C-6‴), 124.9 (C-6), 123.7 (C-6′), 122.4 (C-8a), 118.8 (C-5), 118.6 (C-2′), 114.2 (C-9a), 112.8 (C-4), 111.1 (C-2), 106.8 (C-8), 65.6 (C-1′), 39.5 (C-4′), 26.2 (C-5′), 25.5 (C-8′), 17.5 (C-10′), 16.6 (C-9′). EIMS *m/z* (rel int.): 816 $[M-2]^+$ (1), 683 (7), 681 (14), 679 (7), 619 (5), 617 (10), 615 (5), 461 (55), 463 (56), 399 (41), 397 (41), 371 (19), 369 (19), 357 (34), 355 (34), 244 (27), 229 (84), 215 (100), 186 (16), 157 (53), 155 (53), 131 (11), 108 (13), 76 (16).

3.6. X-ray crystallographic studies of cocrystal of 1 and 1a, monoacetate 18, and dibrosylate 20

Crystallographic data were collected at 100.0 (1) K with the Oxford Cryosystem Cobra low-temperature attachment. The data were collected using a Bruker Apex2 CCD diffractometer with a graphite monochromated Mo Kα radiation at a detector distance of 5 cm using *APEX2*.[25] The collected data were reduced using *SAINT* program,[25] and the empirical absorption corrections were performed using *SADABS* program.[25] The structures were solved by direct methods and refined by least-squares using the *SHELXTL* software package[26] All non-hydrogen atoms were refined anisotropically, whereas all H atoms were placed in calculated positions with an O–H distance of 0.82 Å and C–H distances in the range 0.93–0.98 Å after checking their positions in the difference map. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atoms for methyl H atoms and $1.2U_{eq}$ for hydroxyl and the other H atoms. The final refinement converged well. Materials for publication were prepared using *SHELXTL*[26] and *PLATON*.[27]

Crystal data for cocrystal of **1** and **1a**: $C_{28.60}H_{32.40}O_5$, M=465.15, 0.60×0.19×0.10 mm^3, monoclinic, $P2_1/c$, a=21.6161(6) Å, b=5.3826(2) Å, c=22.8296(8) Å, α=90.00°, β=121.267°(2), γ=90.00°, V=2270.44(13) $Å^3$, Z=4, D_x=1.334 Mg m^{-3}, μ(Mo Kα)=2.512 mm^{-1}, 29,338 reflection measured, 6634 unique reflections, R=0.0995, R_w=0.2794.

Crystal data for **18**: $C_{25}H_{26}O_6$, M=422.46, 0.58×0.27×0.06 mm^3, orthorhombic, $P2_12_12_1$, a=7.3280(2) Å, b=13.0634(5) Å, c=45.6190 (18) Å, α=β=γ=90.00°, V=4367.0(3) $Å^3$, Z=4, D_x=1.285 Mg m^{-3}, μ(Mo Kα)=0.091 mm^{-1}, 81,117 reflection measured, 4877 unique reflections, R=0.1258, R_w=0.3299.

Crystal data for **20**: $C_{35}H_{30}Br_2O_9S_2$, M=818.53, 0.60×0.18×0.03 mm^3, triclinic, P−1, a=9.0779(4) Å, b=19.3468(7) Å, c=20.7939(7) Å, α=108.160(2)°, β=91.755(2)°, γ=90.046(2)°, V=3468.3(2) $Å^3$, Z=4, D_x=1.568 Mg m^{-3}, μ(Mo Kα)=2.512 mm^{-1}, 41,231 reflection measured, 12,219 unique reflections, R=0.0817, R_w=0.2121.

The crystallographic-information files for cocrystal of **1** and **1a**, **18**, and **20** have been deposited in the Cambridge Crystallographic Data Centre as CCDC694467, CCDC689924, and CCDC689378, respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/data_request/cif, or by e-mailing data_request@ccdc.cam.ac.uk, or by contacting the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

3.7. Antimicrobial activity

3.7.1. Antibacterial assay

The isolated compounds from the resin and green fruits of *C. cochinchinense* were tested against both Gram-positive and Gram-negative bacteria: *B. subtilis*, *S. aureus*, TISTR517, *E. faecalis* TISTR459, Methicillin-Resistant *S. aureus* (MRSA) ATCC43300, Vancomycin-Resistant *E. faecalis* (VRE) ATCC 51299, *Streptococcus faecalis*, *S. typhi*, *S. sonei* and *P. aeruginosa*. The microrganisms were obtained from the culture collections, Department of Industrial Biotechnology and Department of Pharmacognosy and Botany, PSU, except for the TISTR and ATCC strains, which were obtained from Microbial Research Center (MIRCEN), Bangkok, Thailand. The antibacterial assay employed was the same as described in Boonsri et al.[5] Vancomycin, which was used as a standard, showed antibacterial activity against Vancomycin-Resistant *E. faecalis* (VRE) ATCC 51299 at 75.0 μg/ml.

3.7.2. Antifungal assay

C. albicans was obtained from Department of Pharmacognosy and Botany, PSU. The antifungal assay employed was the same as described in Boonsri et al.[5] by using amphotencin B as a positive control.

3.7.3. Antibacterial assay against P. aeruginosa and SEM analysis for treated cells

Antibacterial activity against *P. aeruginosa* and the potential mode of action of compounds **4** and **13** were investigated by the same antibacterial assay. The cells of *P. aeruginosa* were sampled at 0, 3, 6, and 15 h for analysis by SEM.

Acknowledgements

We are greatly indebted to the Thailand Research Fund (TRF) for research grant (RSA5280033). Financial support from the Center of Excellence for Innovation in Chemistry (PERCH-CIC), Commission on Higher Education, Ministry of Education is gratefully acknowledged. N.B. thanks the Development and Promotion of Science and Technology Talents Project for fellowship and Graduate School, Prince of Songkla University for financial supports. The authors also thank the Koshinocorporation Group, Japan and Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012. N.B. thanks Asst. Prof. Dr. Surat Laphookhieo for supplying the authentic sample of 1,3,7-trihydroxyxanthone. The authors also would like to thank the Scientific Equipment Center, Prince of Songkla University for the SEM analysis.

References and notes

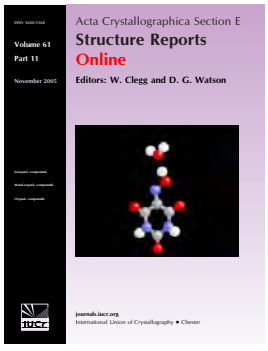
1. Smitinand, T. *Thai Plant Names*; Prachachon: Bangkok, 2001; p 152.
2. Vo, V. V. *A Dictionary of Medicinal Plants in Vietnam*; Y Hoc: HoChiMinh City, 1997; Vol. 435.
3. Boonnak, N.; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Hoong-Kun, F.; Kanjana-Opas, A.; Laphookhieo, S. *Tetrahedron* **2006**, *62*, 8850–8859.
4. Boonnak, N.; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Kanjana-Opas, A.; Chantrapromma, K.; Hoong-Kun, F. *Can. J. Chem.* **2007**, *85*, 341–345.
5. Boonsri, S.; Karalai, C.; Ponglimanont, C.; Kanjana-Opas, A.; Chantrapromma, K. *Phytochemistry* **2006**, *67*, 723–727.
6. Mahabusarakam, W.; Nuangnaowarat, W.; Taylor, W. C. *Phytochemistry* **2006**, *67*, 470–474.
7. Deachathai, S.; Mahabusarakam, W.; Phongpaichit, S.; Taylor, W. C. *Phytochemistry* **2005**, *66*, 2368–2375.
8. Suksamrarn, S.; Komutiban, O.; Ratananukul, P.; Chimnoi, N.; Lartpornmatulee, N.; Suksamrarn, A. *Chem. Pharm. Bull.* **2006**, *54*, 301–305.
9. Suksamrarn, S.; Suwannapoch, N.; Phakhodee, W.; Thanuhiranlert, J.; Ratananukul, P.; Chimnoi, N.; Suksamrarn, A. *Chem. Pharm. Bull.* **2003**, *51*, 857–859.
10. Molinar-Toribio, E.; González, J.; Ortega-Barría, E.; Capson, T. L.; Coley, P. D.; Kursar, T. A.; McPhail, K.; Cubilla-Rios, L. *Pharm. Biol.* **2006**, *44*, 550–553.
11. Engelhard, D. *Bone Marrow Transplant* **1998**, *21*, S78–80.
12. Collin, B. A.; Leather, H. L.; Wingard, J. R.; Ramphal, R. *Clin. Infect. Dis.* **2001**, *33*, 947–953.
13. Mencacci a, A.; Cenci, E.; Repetto, A.; Mazzolla, R.; Bistoni, F.; Aversa, F.; Aloisi, T.; Vecchiarelli. *J. Infect.* **2006**, *53*, 259–264.
14. Nguyen, L. H. D.; Harrison, L. J. *Phytochemistry* **1998**, *50*, 471–476.
15. Dechathai, S.; Mahabusarakam, W.; Phongpaichit, S.; Taylor, W. C.; Zhang, Y. J. *Phytochemistry* **2006**, *67*, 464–469.
16. Mahabusarakam, W.; Wiriyachitra, P.; Taylor, W. C. *J. Nat. Prod.* **1987**, *50*, 474–478.
17. Delle Monache, F.; Botta, B.; Nicoletti, M.; De Barros Coelho, J. S.; Lyra, F. D.; De Andrade Lyra, F. D. *J. Chem. Soc., Perkin Trans. 1* **1981**, 484–488.
18. Sadaquat, A.; Goundar, R.; Sotheeswaran, S.; Beaulieu, C.; Spino, C. *Phytochemistry* **2000**, *53*, 281–284.
19. Rattanaburee, S; Mahabusarakam, W. Chemical Constituents from the Twigs and Flowers of Cratoxylum cochinchinense, In *32nd Congress on Science and Technology of Thailand (STT32)*, Queen Sirikit National Convention Center, Bangkok, Thailand, Oct. 10–12, 2006.
20. Liu, Y.; Zou, L.; Ma, L.; Chen, W. H.; Wang, B.; Xu, Z. L. *Bioorg. Med. Chem.* **2006**, *14*, 5683–5690.
21. Pedro, M.; Cerqueira, F.; Sousa, M. E.; Nascimento, M. S. J.; Pinto, M. *Bioorg. Med. Chem.* **2002**, *10*, 3725–3730.
22. Yoshimi, N.; Matsunaga, K.; Katayama, M.; Yamada, Y.; Kuno, T.; Qiao, Z.; Hara, A.; Yamahara, J.; Mori, H. *Cancer Lett.* **2001**, *163*, 163–170.
23. Lin, C.-N.; Chung, M.-I.; Liou, S.-J.; Lee, T.-H.; Wang, J.-P. *J. Pharm. Pharmacol.* **1996**, *48*, 532–538.
24. Laphookhieo, S.; Maneerat, W.; Buatip, T.; Syers, J. K. *Can. J. Chem.* **2008**, *86*, 757–760.
25. Bruker. *APEX2, SAINT and SADABS*; Bruker AXS Inc.: Madison, WI, 2005.
26. Sheldrick, G. M. *SHELXTL Version 5.1*; Bruker AXS; USA: Madison, WI, 1998.
27. Spek, A. L. *J. Appl. Crystallogr.* **2003**, *36*, 7–13.

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

7-Acetoxycochinchinone I

Suchada Chantrapromma, Nawong Boonnak and Hoong-Kun Fun

Acta Cryst. (2009). E**65**, o2223–o2224



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

7-Acetoxycochinchinone I[1]

Suchada Chantrapromma,[a]*§ Nawong Boonnak[a] and Hoong-Kun Fun[b]¶

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

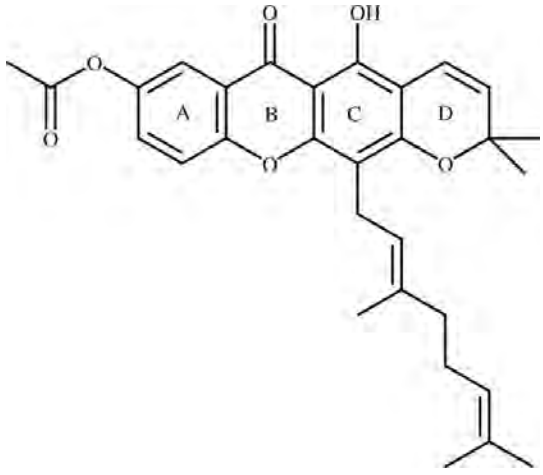
Received 22 July 2009; accepted 14 August 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.095; wR factor = 0.169; data-to-parameter ratio = 15.5.

The title compound {systematic name: 12-[(2*E*)-3,7-dimethyl-2,6-octadienyl]-5,8-dihydroxy-2,2-dimethyl-2*H*,6*H*-pyrano-[3,2-*b*]xanthen-6-one}, $C_{30}H_{32}O_6$, has four fused rings (*A*/*B*/*C*/*D*) and the xanthone ring system (*A*/*B*/*C*) is essentially planar, with dihedral angles of 1.85 (13) and 2.47 (13)°, respectively, between rings *A* and *B*, and between rings *B* and *C*. The chromene ring *D* is in a sofa form. The geranyl side chain is axially attached to ring C with an (−)-synclinal conformation. The 3-methyl-2-butenyl terminal of the geranyl side chain is disordered with the site-occupancy ratio of 0.513 (5):0.487 (5). The acetoxy group is attached axially to ring *A* with an (+)-synclinal conformation. An intramolecular O—H···O hydrogen bond involving the carbonyl and hydroxyl groups generates an *S*(6) ring motif. In the crystal, weak C—H···O and C—H···π interactions, and π–π interactions with centroid–centroid distances of 3.6562 (16) and 3.6565 (16) Å are observed.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987). For ring conformations, see: Cremer & Pople (1975). For the bioactivity of xanthones, see, for examples: Boonnak *et al.* (2006, 2007, 2009); Molinar-Toribio *et al.* (2006); Vo (1997). For related structures, see, for example: Boonnak *et al.* (2009); Kosela *et al.* (1999). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{30}H_{32}O_6$
M_r = 488.56
Triclinic, $P\overline{1}$
a = 6.1047 (1) Å
b = 8.6019 (1) Å
c = 25.4475 (4) Å
α = 97.705 (2)°
β = 91.888 (1)°
γ = 106.581 (1)°
V = 1265.60 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.37 × 0.14 × 0.07 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.968, T_{max} = 0.994
20232 measured reflections
5745 independent reflections
4481 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.095
$wR(F^2)$ = 0.169
S = 1.22
5745 reflections
370 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H1*O5*···O4	0.89	1.73	2.576 (3)	157
C5—H5*A*···O5^{i}	0.93	2.58	3.275 (3)	132
C20—H20*A*···O4^{ii}	0.96	2.47	3.285 (4)	143
C17—H17*A*···*Cg*3^{iii}	0.97	2.74	3.694 (3)	171

Symmetry codes: (i) $x-1, y-1, z$; (ii) $-x, -y+1, -z+1$; (iii) $x+1, y, z$. *Cg*3 is the centroid of the C1–C4/C11/C10 ring.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for research grant (RSA 5280033). NB thanks the Development and Promotion of Science and Technology Talents Project for

[1] This paper is dedicated to the late Her Royal Highness Princess Galyani Vadhana Krom Luang Naradhiwas Rajanagarindra for her patronage of Science in Thailand.

§ Thomson Reuters ResearcherID: A-5085-2009.
¶ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

a fellowship. The authors also thank the Koshino Corporation Group, Japan, Prince of Songkla University and Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2444).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Fun, H.-K., Kanjana-Opas, A. & Chantrapromma, K. (2009). *Tetrahedron* **65**, 3003–3013.
Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Fun, H.-K., Kanjana-Opas, A. & Laphookhieo, S. (2006). *Tetrahedron*, **62**, 8850–8859.
Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Kanjana-Opas, A., Chantrapromma, K. & Fun, H.-K. (2007). *Can. J. Chem.* **85**, 341–345.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Kosela, S., Hu, L. H., Yip, S. C., Rachmatia, T., Sukri, T., Daulay, T. S., Tan, G. K., Vittal, J. J. & Sim, K. Y. (1999). *Phytochemistry*. **52**, 1375–1377.
Molinar-Toribio, E., González, J., Ortega-Barría, E., Capson, T. L., Coley, P. D., Kursar, T. A., McPhail, K. & Cubilla-Rios, L. (2006). *Pharm. Biol.* **44**, 550–553.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Vo, V. V. (1997). *A Dictionary of Medicinal Plants in Vietnam,* Vol. 435. Ho Chi Minh City: Y Hoc Publisher.

supplementary materials

Acta Cryst. (2009). E**65**, o2223-o2224 [**doi:10.1107/S1600536809032292**]

7-Acetoxycochinchinone I

S. Chantrapromma, N. Boonnak and H.-K. Fun

Comment

Cratoxylum cochinchinense belongs to the family Guttiferae, which is distributed in several parts of Thailand. This plant is a well known tropical tree and is commonly known in Thailand as "Tui Kliang". The bark, roots, and leaves of this plant have been used in folk medicine to treat fever, coughs, diarrhea, itches, ulcers, and abdominal complaints (Vo, 1997). Previous investigations revealed the major components from the *Cratoxylum* genus as xanthones and anthraquinones (Boonnak *et al.*, 2006, 2007, 2009). Xanthones have been reported to exhibit biological activities such as antibacterial and cytotoxic (Boonnak *et al.*, 2006, 2007, 2009) as well as antiprotozoal activities (Molinar-Toribio *et al.*, 2006). From our previous work it was found that cochinchinone I, the isolated xanthone from the resin of *Cratoxylum cochinchinense*, was active against *Pseudomonas aeruginosa* (Boonnak *et al.*, 2009). The title compound (I) is an acetylated product of cochinchinone I, which was modified in order to compare their antibacterial and cytotoxic activities. Herein we report the crystal structure of (I).

The title molecule (Fig. 1) has a four-fused rings (*A*/*B*/*C*/*D*), the xanthone skeleton is essentially planar, the dihedral angles between ring *A*/*B* and *B*/*C* are 1.85 (13) and 2.47 (13)°, respectively. The chromene ring *D* is in a sofa form with puckering parameter Q = 0.400 (3) Å, θ = 64.0 (4)° and φ = 314.5 (5)° (Cremer & Pople, 1975), the puckered C16 atom having the maximum deviation of -0.269 (3) Å. The two methyl groups are axially and bisectionally attached to chromine ring at atom C16 with torsion angles C14/C15/C16/C18 of 80.2 (3)° and C14/C15/C16/C17 of -155.0 (3)°, respectively. The geranyl side chain is axially attached to ring *C* at C4 with C3—C4—C21—C22 = -84.1 (3)°, indicating an (-)-*syn*-clinal conformation (Fig. 1). Moreover the 3-methyl-2-butenyl terminal of this geranyl side chain is disordered with the refined site-occupancy ratio of 0.513 (5)/0.487 (5). The acetoxy moiety is axially attached to ring *A* at C7 with C8—C7—O2—C19 = 60.2 (4)°, indicating an (+)-*syn*-clinal conformation and the dihedral angle between the mean plane through the acetoxy group [C19/C20/O2/O3] and ring *A* is 59.05 (14)°. O—H···O intramolecular hydrogen bond involving the carbonyl and hydroxyl moieties generates an S(6) ring motif (Bernstein *et al.*, 1995). The bond lengths in (I) show normal values (Allen *et al.*, 1987) and are comparable to the related structures; cochinchinone I (Boonnak *et al.*, 2009) and dulxanthone E (Kosela *et al.*, 1999).

In the crystal packing (Fig. 2), the symmetric weak C20—H20A···O4 interactions (Table 1) linked the molecules into dimers and generate $R_2^2(18)$ motifs (Bernstein *et al.*, 1995). These dimers are arranged into molecular sheets parallel to the *bc* plane and these sheets are stacked along the *a* axis arising from π–π interactions with the $Cg_1\cdots Cg_2$ distances of 3.6562 (16) Å (symmetry code: -1 + *x*, *y*, *z*) and 3.6565 (16) Å (symmetry code: 1 + *x*, *y*, *z*); Cg_1 and Cg_2 are the centroids of C1–C4/C10/C11 and C5–C8/C12/C13 rings, respectively. The crystal is also stabilized by a C—H···π interaction (Table 1).

Experimental

The resin of *C. cochinchinense* (87.75 g) was extracted with CH_2Cl_2 (2 × 2.0 *L*, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (47.04 g), which was subjected to QCC (Quick Column Chromatography) on silica gel (Merck 60 F254) using hexane as a first eluent and then increasing the

polarity with acetone to give 16 fractions (FR1—FR16). Fractions FR10 and FR11 were separated by QCC eluting with a gradient of acetone-hexane to give seven fractions (FR10A—FR10G). Subfraction FR10B was further purified by CC on silica gel C-18 and eluted with CH_3OH to give cochinchinone I which was further converted to its derivative form by acetylation with Ac_2O in pyridine to give the title compound, which was recrytalized out from $CHCl_3/CH_3OH$ (9:1, *v*/*v*) to afford the yellow single crystals of the title compound after several days (m.p. 389–391 K).

Refinement

Hydroxy H atom was located from the difference map and isotropically refined. The remaining H atoms were placed in calculated positions with d(C—H) = 0.93 Å for aromatic, 0.97 for CH_2 and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxy and methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.72 Å from C10 and the deepest hole is located at 0.45 Å from H14A.

Figures

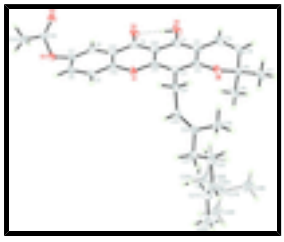


Fig. 1. The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Open bonds show the minor component. Hydrogen bonds were drawn as dash lines.

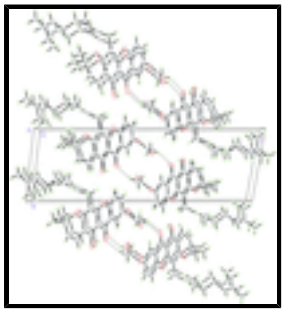


Fig. 2. The crystal packing of the major component of the title compound, viewed along the *a* axis, showing the arrangement of the dimers into molecular sheets. Hydrogen bonds are shown as dashed lines.

12-[(2*E*)-3,7-dimethyl-2,6-octadienyl]-5,8-dihydroxy-2,2-dimethyl- 2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one

Crystal data

$C_{30}H_{32}O_6$	$Z = 2$
$M_r = 488.56$	$F_{000} = 520$
Triclinic, $P\overline{1}$	$D_x = 1.282$ Mg m^{-3}
Hall symbol: -P 1	Melting point = 389–391 K
$a = 6.1047$ (1) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 8.6019$ (1) Å	Cell parameters from 5745 reflections
$c = 25.4475$ (4) Å	$\theta = 2.4$–27.5^o
$\alpha = 97.705$ (2)º	$\mu = 0.09$ mm^{-1}
$\beta = 91.888$ (1)º	$T = 100$ K

γ = 106.581 (1)º	Needle, yellow
V = 1265.60 (3) Å^3	0.37 × 0.14 × 0.07 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	5745 independent reflections
Radiation source: sealed tube	4481 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.056
T = 100 K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -7 \rightarrow 7$
T_{min} = 0.968, T_{max} = 0.994	$k = -11 \rightarrow 11$
20232 measured reflections	$l = -33 \rightarrow 33$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.095	H-atom parameters constrained
$wR(F^2)$ = 0.169	$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 1.0353P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.22	$(\Delta/\sigma)_{max} < 0.001$
5745 reflections	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
370 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.4903 (3)	0.0153 (2)	0.34364 (7)	0.0215 (4)	

O2	−0.2013 (3)	0.1650 (2)	0.45392 (8)	0.0274 (5)
O3	0.0140 (3)	0.3410 (2)	0.52310 (8)	0.0290 (5)
O4	0.5260 (3)	0.5024 (2)	0.37753 (8)	0.0303 (5)
O5	0.8916 (3)	0.5734 (2)	0.32694 (9)	0.0338 (5)
H1O5	0.7623	0.5744	0.3421	0.051*
O6	1.1288 (3)	0.1277 (2)	0.24297 (8)	0.0249 (4)
C1	0.8620 (5)	0.4101 (3)	0.31609 (12)	0.0275 (7)
C2	1.0173 (5)	0.3570 (3)	0.28511 (12)	0.0261 (6)
C3	0.9824 (4)	0.1868 (3)	0.27332 (11)	0.0223 (6)
C4	0.8103 (5)	0.0702 (3)	0.29359 (11)	0.0225 (6)
C5	0.1645 (5)	−0.0618 (3)	0.39063 (11)	0.0231 (6)
H5A	0.1686	−0.1697	0.3833	0.028*
C6	−0.0072 (5)	−0.0238 (3)	0.41912 (11)	0.0246 (6)
H6A	−0.1205	−0.1062	0.4311	0.030*
C7	−0.0089 (5)	0.1388 (3)	0.42974 (11)	0.0242 (6)
C8	0.1559 (5)	0.2631 (3)	0.41349 (11)	0.0240 (6)
H8A	0.1523	0.3710	0.4214	0.029*
C9	0.5139 (5)	0.3543 (3)	0.36676 (11)	0.0244 (6)
C10	0.6792 (5)	0.2985 (3)	0.33615 (11)	0.0243 (6)
C11	0.6618 (4)	0.1303 (3)	0.32475 (11)	0.0209 (6)
C12	0.3303 (5)	0.0636 (3)	0.37321 (10)	0.0220 (6)
C13	0.3306 (5)	0.2261 (3)	0.38476 (11)	0.0235 (6)
C14	1.2182 (5)	0.4675 (3)	0.26653 (13)	0.0318 (7)
H14A	1.2611	0.5795	0.2789	0.038*
C15	1.3398 (5)	0.4041 (4)	0.23125 (13)	0.0334 (7)
H15A	1.4824	0.4677	0.2235	0.040*
C16	1.2444 (5)	0.2300 (3)	0.20441 (11)	0.0243 (6)
C17	1.4310 (5)	0.1556 (4)	0.18603 (12)	0.0308 (7)
H17A	1.5389	0.1660	0.2155	0.046*
H17B	1.5084	0.2121	0.1588	0.046*
H17C	1.3640	0.0417	0.1721	0.046*
C18	1.0709 (5)	0.2163 (4)	0.15850 (13)	0.0346 (7)
H18A	0.9483	0.2564	0.1715	0.052*
H18B	1.0103	0.1035	0.1426	0.052*
H18C	1.1444	0.2802	0.1324	0.052*
C19	−0.1699 (5)	0.2723 (3)	0.50082 (11)	0.0242 (6)
C20	−0.3962 (5)	0.2855 (4)	0.51718 (13)	0.0369 (8)
H20A	−0.3840	0.3261	0.5545	0.055*
H20B	−0.5075	0.1794	0.5101	0.055*
H20C	−0.4434	0.3597	0.4975	0.055*
C21	0.7812 (5)	−0.1116 (3)	0.27952 (11)	0.0203 (6)
H21A	0.9307	−0.1293	0.2771	0.024*
H21B	0.7063	−0.1685	0.3074	0.024*
C22	0.6412 (5)	−0.1810 (3)	0.22756 (11)	0.0210 (6)
H22A	0.4926	−0.1726	0.2263	0.025*
C23	0.7016 (5)	−0.2524 (3)	0.18336 (11)	0.0225 (6)
C24	0.5354 (5)	−0.3151 (4)	0.13489 (11)	0.0292 (7)
H24A	0.3826	−0.3189	0.1452	0.035*
H24B	0.5344	−0.4264	0.1215	0.035*

C25	0.5913 (6)	−0.2108 (5)	0.09019 (14)	0.0499 (10)	
H25A	0.7531	−0.1799	0.0861	0.060*	0.513 (6)
H25B	0.5456	−0.1130	0.0986	0.060*	0.513 (6)
H25C	0.7262	−0.2265	0.0748	0.060*	0.487 (6)
H25D	0.6274	−0.0978	0.1060	0.060*	0.487 (6)
C26A	0.4508 (14)	−0.3190 (11)	0.0352 (3)	0.0395 (18)	0.513 (6)
H26A	0.4791	−0.4172	0.0221	0.047*	0.513 (6)
C27A	0.2990 (14)	−0.2709 (9)	0.0089 (3)	0.0411 (18)	0.513 (6)
C28A	0.1669 (19)	−0.3770 (13)	−0.0404 (4)	0.069 (3)	0.513 (6)
H28A	0.2035	−0.4791	−0.0455	0.103*	0.513 (6)
H28B	0.2073	−0.3217	−0.0706	0.103*	0.513 (6)
H28C	0.0056	−0.3978	−0.0366	0.103*	0.513 (6)
C29A	0.2355 (14)	−0.1167 (10)	0.0241 (3)	0.060 (2)	0.513 (6)
H29A	0.3303	−0.0533	0.0548	0.090*	0.513 (6)
H29B	0.0775	−0.1434	0.0320	0.090*	0.513 (6)
H29C	0.2577	−0.0541	−0.0049	0.090*	0.513 (6)
C26B	0.4121 (13)	−0.2419 (10)	0.0485 (3)	0.0317 (17)	0.487 (6)
H26B	0.2618	−0.2663	0.0582	0.038*	0.487 (6)
C27B	0.4451 (14)	−0.2384 (8)	−0.0025 (3)	0.0322 (16)	0.487 (6)
C28B	0.2448 (16)	−0.2883 (12)	−0.0447 (3)	0.045 (2)	0.487 (6)
H28D	0.1039	−0.3268	−0.0283	0.067*	0.487 (6)
H28E	0.2627	−0.3742	−0.0709	0.067*	0.487 (6)
H28F	0.2416	−0.1953	−0.0615	0.067*	0.487 (6)
C29B	0.6724 (13)	−0.1817 (10)	−0.0245 (3)	0.046 (2)	0.487 (6)
H29D	0.7908	−0.1484	0.0040	0.069*	0.487 (6)
H29E	0.6766	−0.0906	−0.0429	0.069*	0.487 (6)
H29F	0.6966	−0.2697	−0.0488	0.069*	0.487 (6)
C30	0.9318 (5)	−0.2815 (4)	0.17768 (12)	0.0326 (7)	
H30A	1.0354	−0.2197	0.2074	0.049*	
H30B	0.9913	−0.2472	0.1453	0.049*	
H30C	0.9158	−0.3962	0.1768	0.049*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0212 (10)	0.0155 (9)	0.0258 (10)	0.0021 (7)	0.0038 (8)	0.0026 (8)
O2	0.0187 (10)	0.0284 (11)	0.0305 (11)	0.0043 (8)	−0.0016 (8)	−0.0051 (9)
O3	0.0274 (11)	0.0314 (11)	0.0239 (11)	0.0014 (9)	−0.0005 (9)	0.0060 (9)
O4	0.0288 (11)	0.0160 (10)	0.0433 (13)	0.0043 (8)	0.0008 (9)	0.0007 (9)
O5	0.0256 (11)	0.0141 (10)	0.0594 (15)	0.0019 (8)	0.0052 (10)	0.0053 (9)
O6	0.0197 (10)	0.0205 (10)	0.0343 (12)	0.0025 (8)	0.0039 (8)	0.0101 (8)
C1	0.0234 (15)	0.0145 (13)	0.0417 (18)	0.0007 (11)	−0.0038 (13)	0.0068 (12)
C2	0.0206 (14)	0.0203 (14)	0.0350 (17)	0.0010 (11)	−0.0049 (12)	0.0081 (12)
C3	0.0162 (13)	0.0228 (14)	0.0279 (15)	0.0060 (11)	−0.0035 (11)	0.0041 (12)
C4	0.0219 (14)	0.0173 (13)	0.0282 (15)	0.0051 (11)	−0.0023 (12)	0.0055 (11)
C5	0.0223 (14)	0.0197 (13)	0.0261 (15)	0.0057 (11)	−0.0014 (12)	0.0016 (11)
C6	0.0218 (14)	0.0201 (14)	0.0282 (16)	0.0005 (11)	−0.0008 (12)	0.0034 (12)
C7	0.0178 (14)	0.0264 (14)	0.0267 (15)	0.0070 (11)	−0.0023 (11)	−0.0021 (12)

C8	0.0222 (15)	0.0199 (14)	0.0274 (15)	0.0055 (11)	−0.0071 (12)	−0.0024 (11)
C9	0.0236 (15)	0.0192 (14)	0.0279 (16)	0.0042 (11)	−0.0055 (12)	0.0010 (11)
C10	0.0236 (15)	0.0190 (13)	0.0293 (16)	0.0048 (11)	−0.0039 (12)	0.0052 (12)
C11	0.0181 (14)	0.0154 (13)	0.0258 (15)	−0.0006 (10)	−0.0044 (11)	0.0044 (11)
C12	0.0214 (14)	0.0232 (14)	0.0196 (14)	0.0046 (11)	−0.0030 (11)	0.0024 (11)
C13	0.0215 (14)	0.0209 (14)	0.0250 (15)	0.0036 (11)	−0.0048 (11)	0.0005 (11)
C14	0.0215 (15)	0.0186 (14)	0.053 (2)	0.0004 (12)	0.0022 (14)	0.0097 (13)
C15	0.0215 (16)	0.0272 (16)	0.047 (2)	−0.0033 (12)	0.0021 (14)	0.0131 (14)
C16	0.0160 (14)	0.0255 (14)	0.0314 (16)	0.0026 (11)	0.0001 (12)	0.0129 (12)
C17	0.0211 (15)	0.0347 (16)	0.0377 (18)	0.0049 (12)	0.0038 (13)	0.0157 (14)
C18	0.0221 (16)	0.0402 (18)	0.0419 (19)	0.0040 (13)	−0.0002 (14)	0.0191 (15)
C19	0.0303 (16)	0.0207 (14)	0.0205 (15)	0.0035 (12)	0.0020 (12)	0.0081 (11)
C20	0.0298 (17)	0.0441 (19)	0.0340 (18)	0.0118 (15)	0.0021 (14)	−0.0065 (15)
C21	0.0198 (14)	0.0143 (12)	0.0263 (15)	0.0027 (10)	0.0021 (11)	0.0060 (11)
C22	0.0191 (14)	0.0151 (12)	0.0280 (15)	0.0013 (10)	0.0000 (11)	0.0083 (11)
C23	0.0210 (14)	0.0177 (13)	0.0258 (15)	−0.0005 (11)	0.0010 (11)	0.0061 (11)
C24	0.0228 (15)	0.0317 (16)	0.0287 (16)	0.0005 (12)	0.0027 (12)	0.0049 (13)
C25	0.036 (2)	0.068 (3)	0.041 (2)	−0.0003 (18)	−0.0023 (16)	0.0271 (19)
C26A	0.050 (5)	0.042 (5)	0.023 (4)	0.010 (4)	0.004 (3)	0.002 (3)
C27A	0.037 (4)	0.049 (4)	0.034 (4)	0.002 (3)	−0.005 (3)	0.019 (3)
C28A	0.074 (7)	0.080 (8)	0.038 (5)	−0.001 (6)	−0.022 (5)	0.020 (5)
C29A	0.048 (5)	0.080 (6)	0.067 (6)	0.030 (4)	0.023 (4)	0.033 (5)
C26B	0.032 (4)	0.040 (4)	0.024 (4)	0.013 (3)	0.009 (3)	0.003 (3)
C27B	0.032 (4)	0.033 (4)	0.032 (4)	0.013 (3)	−0.001 (3)	0.001 (3)
C28B	0.046 (5)	0.054 (6)	0.035 (5)	0.017 (4)	0.001 (4)	0.004 (4)
C29B	0.051 (5)	0.064 (5)	0.026 (4)	0.019 (4)	0.006 (3)	0.007 (3)
C30	0.0285 (17)	0.0382 (17)	0.0303 (17)	0.0109 (14)	0.0020 (13)	−0.0001 (13)

Geometric parameters (Å, °)

O1—C11	1.369 (3)	C20—H20A	0.9600
O1—C12	1.375 (3)	C20—H20B	0.9600
O2—C19	1.379 (3)	C20—H20C	0.9600
O2—C7	1.404 (3)	C21—C22	1.502 (4)
O3—C19	1.192 (3)	C21—H21A	0.9700
O4—C9	1.247 (3)	C21—H21B	0.9700
O5—C1	1.352 (3)	C22—C23	1.323 (4)
O5—H1O5	0.8912	C22—H22A	0.9300
O6—C3	1.361 (3)	C23—C30	1.505 (4)
O6—C16	1.469 (3)	C23—C24	1.506 (4)
C1—C2	1.389 (4)	C24—C25	1.530 (4)
C1—C10	1.409 (4)	C24—H24A	0.9700
C2—C3	1.406 (4)	C24—H24B	0.9700
C2—C14	1.457 (4)	C25—C26B	1.440 (8)
C3—C4	1.396 (4)	C25—C26A	1.636 (8)
C4—C11	1.387 (4)	C25—H25A	0.9601
C4—C21	1.514 (3)	C25—H25B	0.9600
C5—C6	1.383 (4)	C25—H25C	0.9600
C5—C12	1.385 (4)	C25—H25D	0.9600

C5—H5A	0.9300	C26A—C27A	1.315 (11)
C6—C7	1.392 (4)	C26A—H26A	0.9300
C6—H6A	0.9300	C27A—C29A	1.492 (11)
C7—C8	1.363 (4)	C27A—C28A	1.505 (12)
C8—C13	1.398 (4)	C28A—H28A	0.9600
C8—H8A	0.9300	C28A—H28B	0.9600
C9—C10	1.444 (4)	C28A—H28C	0.9600
C9—C13	1.464 (4)	C29A—H29A	0.9600
C10—C11	1.409 (4)	C29A—H29B	0.9600
C12—C13	1.389 (4)	C29A—H29C	0.9600
C14—C15	1.341 (4)	C26B—C27B	1.324 (10)
C14—H14A	0.9300	C26B—H26B	0.9300
C15—C16	1.500 (4)	C27B—C29B	1.488 (10)
C15—H15A	0.9300	C27B—C28B	1.524 (11)
C16—C17	1.516 (4)	C28B—H28D	0.9600
C16—C18	1.520 (4)	C28B—H28E	0.9600
C17—H17A	0.9600	C28B—H28F	0.9600
C17—H17B	0.9600	C29B—H29D	0.9600
C17—H17C	0.9600	C29B—H29E	0.9600
C18—H18A	0.9600	C29B—H29F	0.9600
C18—H18B	0.9600	C30—H30A	0.9600
C18—H18C	0.9600	C30—H30B	0.9600
C19—C20	1.487 (4)	C30—H30C	0.9600
C11—O1—C12	119.8 (2)	C19—C20—H20B	109.5
C19—O2—C7	119.2 (2)	H20A—C20—H20B	109.5
C1—O5—H1O5	100.7	C19—C20—H20C	109.5
C3—O6—C16	116.5 (2)	H20A—C20—H20C	109.5
O5—C1—C2	118.1 (2)	H20B—C20—H20C	109.5
O5—C1—C10	120.4 (3)	C22—C21—C4	111.2 (2)
C2—C1—C10	121.5 (2)	C22—C21—H21A	109.4
C1—C2—C3	117.5 (3)	C4—C21—H21A	109.4
C1—C2—C14	123.7 (3)	C22—C21—H21B	109.4
C3—C2—C14	118.8 (3)	C4—C21—H21B	109.4
O6—C3—C4	116.0 (2)	H21A—C21—H21B	108.0
O6—C3—C2	120.1 (2)	C23—C22—C21	128.5 (3)
C4—C3—C2	123.8 (3)	C23—C22—H22A	115.7
C11—C4—C3	116.1 (2)	C21—C22—H22A	115.7
C11—C4—C21	122.4 (2)	C22—C23—C30	123.9 (3)
C3—C4—C21	121.4 (2)	C22—C23—C24	120.7 (3)
C6—C5—C12	118.9 (3)	C30—C23—C24	115.3 (2)
C6—C5—H5A	120.5	C23—C24—C25	113.7 (2)
C12—C5—H5A	120.5	C23—C24—H24A	108.8
C5—C6—C7	119.4 (3)	C25—C24—H24A	108.8
C5—C6—H6A	120.3	C23—C24—H24B	108.8
C7—C6—H6A	120.3	C25—C24—H24B	108.8
C8—C7—C6	122.0 (3)	H24A—C24—H24B	107.7
C8—C7—O2	122.0 (3)	C26B—C25—C24	115.7 (4)
C6—C7—O2	115.8 (2)	C24—C25—C26A	108.3 (4)
C7—C8—C13	119.1 (3)	C26B—C25—H25A	126.3

C7—C8—H8A	120.4	C24—C25—H25A	110.2
C13—C8—H8A	120.4	C26A—C25—H25A	110.2
O4—C9—C10	122.4 (3)	C26B—C25—H25B	80.8
O4—C9—C13	121.8 (3)	C24—C25—H25B	109.7
C10—C9—C13	115.9 (2)	C26A—C25—H25B	109.9
C1—C10—C11	117.7 (3)	H25A—C25—H25B	108.6
C1—C10—C9	121.2 (2)	C26B—C25—H25C	108.9
C11—C10—C9	121.2 (3)	C24—C25—H25C	108.7
O1—C11—C4	115.9 (2)	C26A—C25—H25C	86.1
O1—C11—C10	120.8 (2)	H25B—C25—H25C	130.4
C4—C11—C10	123.3 (3)	C26B—C25—H25D	108.2
O1—C12—C5	115.4 (2)	C24—C25—H25D	107.5
O1—C12—C13	123.1 (2)	C26A—C25—H25D	134.7
C5—C12—C13	121.5 (3)	H25A—C25—H25D	81.8
C12—C13—C8	119.1 (3)	H25C—C25—H25D	107.5
C12—C13—C9	119.3 (3)	C27A—C26A—C25	121.8 (8)
C8—C13—C9	121.7 (2)	C27A—C26A—H26A	119.1
C15—C14—C2	118.6 (3)	C25—C26A—H26A	119.1
C15—C14—H14A	120.7	C26A—C27A—C29A	125.8 (8)
C2—C14—H14A	120.7	C26A—C27A—C28A	120.2 (9)
C14—C15—C16	119.6 (3)	C29A—C27A—C28A	114.0 (8)
C14—C15—H15A	120.2	C27B—C26B—C25	124.9 (7)
C16—C15—H15A	120.2	C27B—C26B—H26B	117.5
O6—C16—C15	109.3 (2)	C25—C26B—H26B	117.5
O6—C16—C17	104.6 (2)	C26B—C27B—C29B	125.1 (7)
C15—C16—C17	112.2 (2)	C26B—C27B—C28B	121.5 (7)
O6—C16—C18	108.3 (2)	C29B—C27B—C28B	113.4 (6)
C15—C16—C18	111.3 (2)	C27B—C28B—H28D	109.5
C17—C16—C18	110.8 (3)	C27B—C28B—H28E	109.5
C16—C17—H17A	109.5	H28D—C28B—H28E	109.5
C16—C17—H17B	109.5	C27B—C28B—H28F	109.5
H17A—C17—H17B	109.5	H28D—C28B—H28F	109.5
C16—C17—H17C	109.5	H28E—C28B—H28F	109.5
H17A—C17—H17C	109.5	C27B—C29B—H29D	109.5
H17B—C17—H17C	109.5	C27B—C29B—H29E	109.5
C16—C18—H18A	109.5	H29D—C29B—H29E	109.5
C16—C18—H18B	109.5	C27B—C29B—H29F	109.5
H18A—C18—H18B	109.5	H29D—C29B—H29F	109.5
C16—C18—H18C	109.5	H29E—C29B—H29F	109.5
H18A—C18—H18C	109.5	C23—C30—H30A	109.5
H18B—C18—H18C	109.5	C23—C30—H30B	109.5
O3—C19—O2	123.1 (3)	H30A—C30—H30B	109.5
O3—C19—C20	127.6 (3)	C23—C30—H30C	109.5
O2—C19—C20	109.3 (2)	H30A—C30—H30C	109.5
C19—C20—H20A	109.5	H30B—C30—H30C	109.5
O5—C1—C2—C3	−178.9 (2)	C6—C5—C12—O1	−177.8 (2)
C10—C1—C2—C3	1.5 (4)	C6—C5—C12—C13	1.5 (4)
O5—C1—C2—C14	4.1 (4)	O1—C12—C13—C8	177.6 (2)
C10—C1—C2—C14	−175.5 (3)	C5—C12—C13—C8	−1.7 (4)

C16—O6—C3—C4	155.6 (2)	O1—C12—C13—C9	−2.6 (4)
C16—O6—C3—C2	−28.0 (3)	C5—C12—C13—C9	178.1 (3)
C1—C2—C3—O6	179.8 (2)	C7—C8—C13—C12	0.5 (4)
C14—C2—C3—O6	−3.0 (4)	C7—C8—C13—C9	−179.3 (3)
C1—C2—C3—C4	−4.1 (4)	O4—C9—C13—C12	−177.7 (3)
C14—C2—C3—C4	173.1 (3)	C10—C9—C13—C12	2.0 (4)
O6—C3—C4—C11	179.7 (2)	O4—C9—C13—C8	2.1 (4)
C2—C3—C4—C11	3.4 (4)	C10—C9—C13—C8	−178.2 (3)
O6—C3—C4—C21	−3.7 (4)	C1—C2—C14—C15	−171.4 (3)
C2—C3—C4—C21	−179.9 (3)	C3—C2—C14—C15	11.6 (4)
C12—C5—C6—C7	−0.2 (4)	C2—C14—C15—C16	11.3 (4)
C5—C6—C7—C8	−0.9 (4)	C3—O6—C16—C15	47.3 (3)
C5—C6—C7—O2	173.6 (2)	C3—O6—C16—C17	167.6 (2)
C19—O2—C7—C8	−60.2 (4)	C3—O6—C16—C18	−74.1 (3)
C19—O2—C7—C6	125.3 (3)	C14—C15—C16—O6	−39.3 (4)
C6—C7—C8—C13	0.8 (4)	C14—C15—C16—C17	−155.0 (3)
O2—C7—C8—C13	−173.4 (2)	C14—C15—C16—C18	80.2 (3)
O5—C1—C10—C11	−178.1 (3)	C7—O2—C19—O3	−2.5 (4)
C2—C1—C10—C11	1.5 (4)	C7—O2—C19—C20	177.3 (2)
O5—C1—C10—C9	2.6 (4)	C11—C4—C21—C22	92.4 (3)
C2—C1—C10—C9	−177.8 (3)	C3—C4—C21—C22	−84.1 (3)
O4—C9—C10—C1	−1.0 (4)	C4—C21—C22—C23	119.2 (3)
C13—C9—C10—C1	179.3 (3)	C21—C22—C23—C30	1.6 (4)
O4—C9—C10—C11	179.7 (3)	C21—C22—C23—C24	179.9 (2)
C13—C9—C10—C11	0.0 (4)	C22—C23—C24—C25	107.2 (3)
C12—O1—C11—C4	−177.6 (2)	C30—C23—C24—C25	−74.4 (3)
C12—O1—C11—C10	1.3 (4)	C23—C24—C25—C26B	−166.2 (5)
C3—C4—C11—O1	178.7 (2)	C23—C24—C25—C26A	163.1 (4)
C21—C4—C11—O1	2.1 (4)	C26B—C25—C26A—C27A	9.2 (7)
C3—C4—C11—C10	−0.2 (4)	C24—C25—C26A—C27A	119.4 (7)
C21—C4—C11—C10	−176.8 (2)	C25—C26A—C27A—C29A	0.6 (12)
C1—C10—C11—O1	179.0 (2)	C25—C26A—C27A—C28A	−177.7 (7)
C9—C10—C11—O1	−1.7 (4)	C24—C25—C26B—C27B	−143.2 (7)
C1—C10—C11—C4	−2.2 (4)	C26A—C25—C26B—C27B	−61.7 (9)
C9—C10—C11—C4	177.1 (3)	C25—C26B—C27B—C29B	−8.0 (12)
C11—O1—C12—C5	−179.8 (2)	C25—C26B—C27B—C28B	174.2 (7)
C11—O1—C12—C13	0.9 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H1O5···O4	0.89	1.73	2.576 (3)	157
C5—H5A···O5^{i}	0.93	2.58	3.275 (3)	132
C20—H20A···O4^{ii}	0.96	2.47	3.285 (4)	143
C17—H17A···Cg3^{iii}	0.97	2.74	3.694 (3)	171

Symmetry codes: (i) *x*−1, *y*−1, *z*; (ii) −*x*, −*y*+1, −*z*+1; (iii) *x*+1, *y*, *z*.

Fig. 1

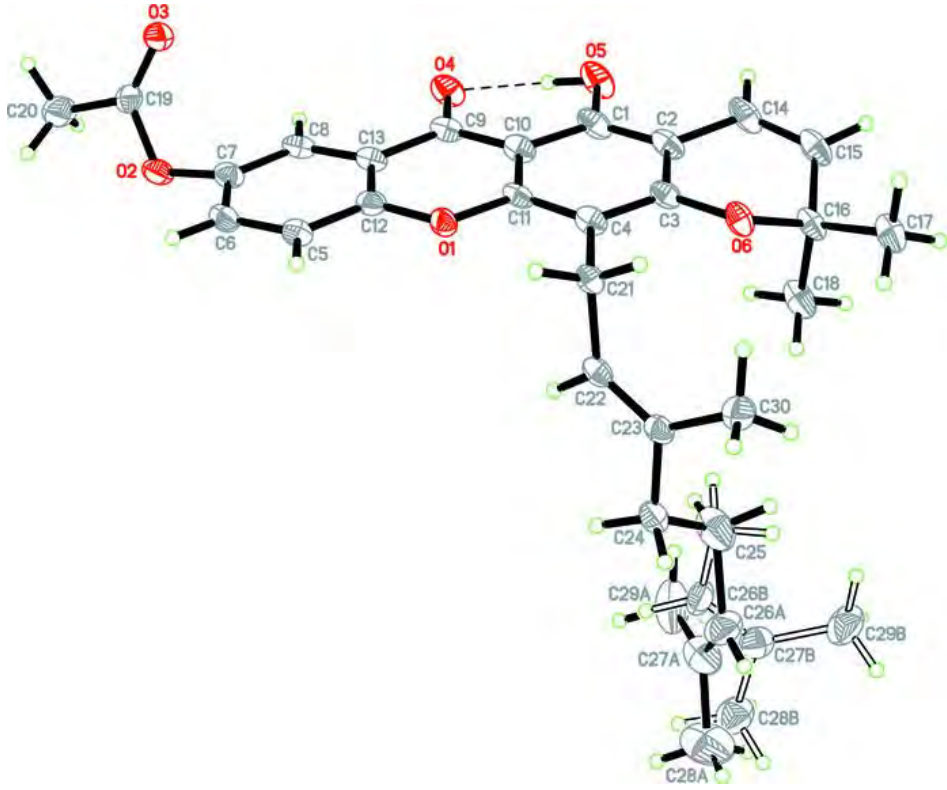
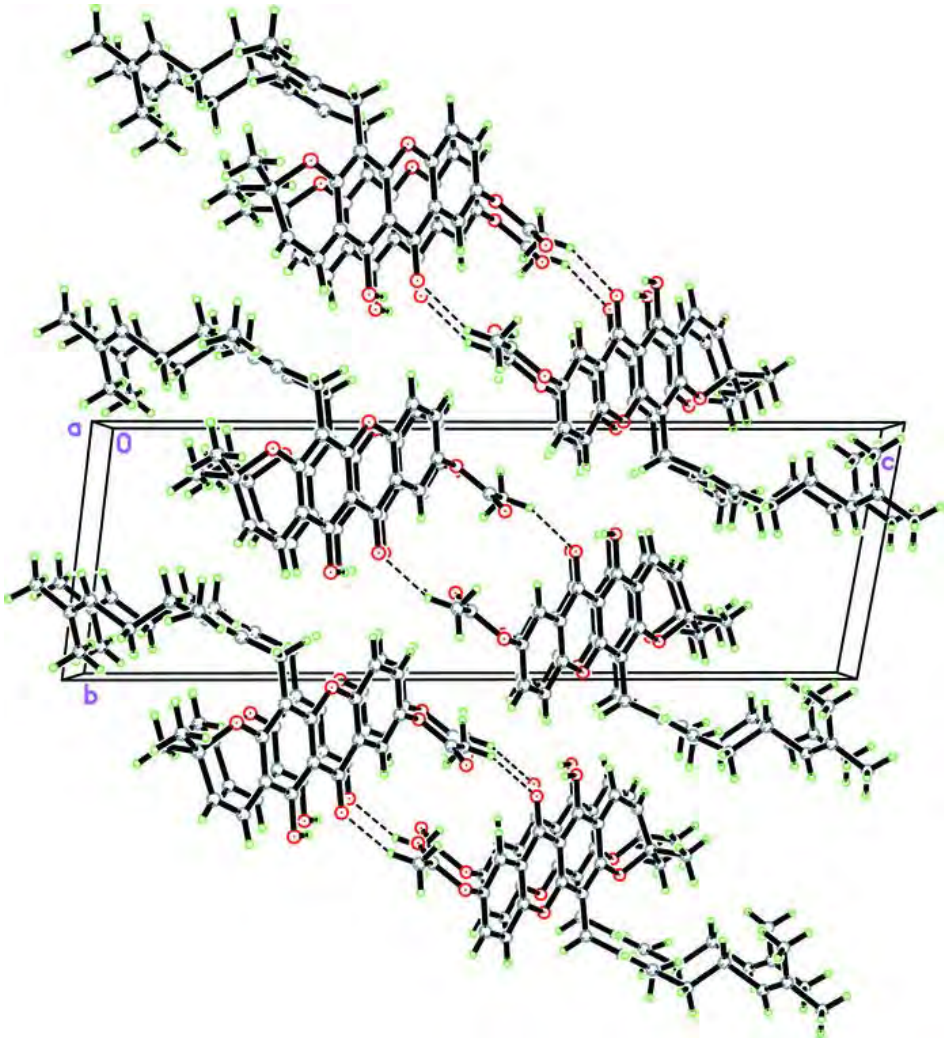


Fig. 2

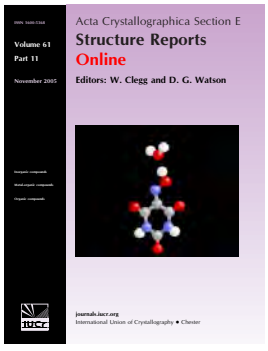


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-1-(4-Bromophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

Thitipone Suwunwong, Suchada Chantrapromma and Hoong-Kun Fun

Acta Cryst. (2009). E**65**, o120



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(4-Bromophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one[1]

Thitipone Suwunwong,[a] Suchada Chantrapromma[a]* and Hoong-Kun Fun[b]§

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

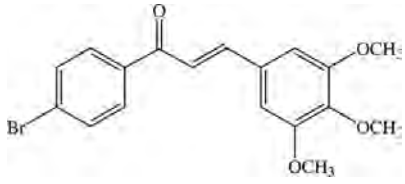
Received 6 November 2008; accepted 9 December 2008

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.097; data-to-parameter ratio = 45.9.

In the title compound, $C_{18}H_{17}BrO_4$, the dihedral angle between the 4-bromophenyl and 3,4,5-trimethoxyphenyl rings is 44.18 (6)°. In the crystal structure, the molecules are linked by C—H···O and C—H···π interactions.

Related literature

For background and applications to chalcones, see: Jung *et al.* (2008); Patil *et al.* (2007); Patil & Dharmaprakash (2008); Prasad *et al.* (2008); Schlogl & Egger (1963). For related structures, see: Ng *et al.* (2006); Patil *et al.* (2006; 2007). For on hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{18}H_{17}BrO_4$
M_r = 377.22
Tetragonal, $P4_2/n$
a = 26.6517 (3) Å
c = 4.4238 (1) Å
V = 3142.28 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.63 mm^{-1}
T = 100.0 (1) K
0.55 × 0.12 × 0.12 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.320, T_{max} = 0.726
142737 measured reflections
9693 independent reflections
6638 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.097
S = 1.07
9693 reflections
211 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O1^{i}	0.93	2.52	3.4391 (16)	170
C17—H17C···O3^{ii}	0.96	2.52	3.2789 (16)	136
C16—H16B···$Cg1^{iii}$	0.96	2.97	3.8080 (14)	147

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $x, y, z-1$; (iii) $x, y, z+1$. $Cg1$ is the centroid of the C10–C15 ring.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

The authors thank the Thailand Research Fund (TRF) and Prince of Songkla University for financial support and also thank Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2134).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jung, Y. J., Son, K. I., Oh, Y. E. & Noh, D. Y. (2008). *Polyhedron*, **27**, 861–867.
Ng, S.-L., Shettigar, V., Razak, I. A., Fun, H.-K., Patil, P. S. & Dharmaprakash, S. M. (2006). *Acta Cryst.* E**62**, o1570–o1572.
Patil, P. S., Chantrapromma, S., Fun, H.-K., Dharmaprakash, S. M. & Babu, H. B. R. (2007). *Acta Cryst.* E**63**, o2612.
Patil, P. S. & Dharmaprakash, S. M. (2008). *Mater. Lett.* **62**, 451–453.
Patil, P. S., Rosli, M. M., Fun, H.-K., Razak, I. A., Puranik, V. G. & Dharmaprakash, S. M. (2006). *Acta Cryst.* E**62**, o4798–o4799.
Prasad, Y. R., Kumar, P. R., Smile, D. J. & Babu, P. A. (2008). *ARKIVOC*, **11**, 266–276.
Schlogl, K. & Egger, H. (1963). *Monatsh. Chem.* **94**, 376–392.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

[1] This paper is dedicated to the late Her Royal Highness Princess Galyani Vadhana Krom Luang Naradhiwas Rajanagarindra for her patronage of Science in Thailand.
§ Additional correspondence author, email: hkfun@usm.my.

supplementary materials

Acta Cryst. (2009). E**65**, o120 [**doi:10.1107/S1600536808041780**]

(*E*)-1-(4-Bromophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

T. Suwunwong, S. Chantrapromma and H.-K. Fun

Comment

Chalcones are compounds in a family of aromatic ketones with two aromatic groups bridged by an enone linkage (Ar-COCH=CH—Ar) (Schlogl & Egger, 1963). They have a wide range of applications covering non-linear optical (NLO) (Patil & Dharmaprakash, 2008) and electro-active fluorescent materials (Jung *et al.*, 2008) to materials with various biological activities. As an example, 1-(4-hydroxyphenyl)-3-(3,4,5-trimethoxyphenyl)-propenone was found to be able to inhibit growth of some bacteria (Prasad *et al.*, 2008). These interesting properties of chalcones led us to synthesize the title compound so as to study for its antibacterial and cytotoxic activities.

The molecule of the title chalcone derivative (Fig. 1) exists in an *E* configuration with respect to the C8?C9 double bond [1.3428 (17) Å] with torsion angle C7–C8–C9–C10 = -173.04 (12)°. The whole molecule is not planar as the interplanar angle between 4-bromophenyl and 3,4,5-trimethoxyphenyl rings is 44.18 (6)°. The propenone unit (C7—C9/O1) is nearly planar with the torsion angle O1–C7–C8–C9 = 3.4 (2)°. Atoms O1, C6, C7, C8 and C9 lie on the same plane with the most deviation of -0.018 (1) Å for atom C8. The mean plane through O1/C6/C7/C8/C9 makes interplanar angles of 30.82 (7)° and 13.37 (7)° with the planes of 4-bromophenyl and 3,4,5-trimethoxyphenyl rings, respectively. The three methoxy groups of 3,4,5-trimethoxyphenyl unit have three difference orientations: one methoxy group (at atom C14 position) is co-planar with the attached benzene ring with torsion angle C18–O4–C14–C15 = 0.71 (17)° whereas the one at atom C12 position is twisted with the torsion angle C16–O2–C12–C11 = 10.38 (16)° and one is (+)-*syn*-clinally attached at atom C13 with the torsion angle C17–O3–C13—C14 = 74.48 (14)°. The bond distances are of normal values (Allen *et al.*, 1987) and are comparable with the closely related structures (Ng *et al.*, 2006; Patil *et al.*, 2006; 2007).

In the crystal packing (Fig. 2), the molecules are linked by weak C11—H11A···O1 intermolcular interactions (Table 1) into cyclic centrosymmetric $R^2_2(14)$ dimers (Bernstein *et al.*, 1995). These dimers are stacked along the *c* axis (Fig. 2) and molecules within the stacks are interlinked by weak C17—H17C···O3 intermolecular interactions. The crystal is stabilized by weak C—H···O interactions (Table 1) and a C—H···π interaction (C16—H16B···Cg_1 = 3.8080 (14) Å), where Cg_1 is the centroid of the C10–C15 ring.

Experimental

The title compound was synthesized by the condensation of 3,4,5-trimethoxybenzaldehyde (0.4 g, 2 mmol) with 4-bromoacetophenone (0.4 g, 2 mmol) in ethanol (50 ml) in the presence of 30% NaOH(aq) (10 ml). After stirring for 4 h, the resulting pale yellow solid appeared and was then collected by filtration, washed with distilled water, dried and purified by repeated recrystallization from acetone. Colorless block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystalized from acetone/methanol (1:1 *v*/*v*) by the slow evaporation of the solvent at room temperature over several days, Mp. 403–404 K.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93 Å, U_{iso} = 1.2U_{eq}(C) for aromatic and CH and C—H = 0.96 Å, U_{iso} = 1.5U_{eq}(C) for CH_3 atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.64 Å from C12 and the deepest hole is located at 0.24 Å from Br1.

Figures

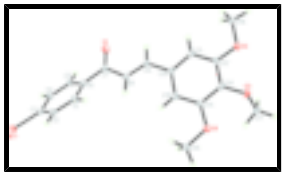


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

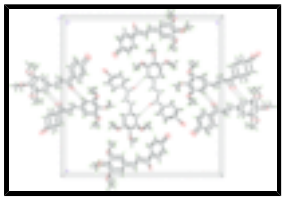


Fig. 2. The crystal packing of the title compound, showing dimers stacked along the *c* axis. Hydrogen bonds are shown as dashed lines.

(*E*)-1-(4-Bromophenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{18}H_{17}BrO_4$	Z = 8
M_r = 377.22	F_{000} = 1536
Tetragonal, $P4_2/n$	D_x = 1.595 Mg m^{-3}
Hall symbol: -P 4bc	Melting point = 403–404 K
a = 26.6517 (3) Å	Mo $K\alpha$ radiation λ = 0.71073 Å
b = 26.6517 (3) Å	Cell parameters from 9693 reflections
c = 4.4238 (1) Å	θ = 1.1–40.0º
α = 90º	μ = 2.63 mm^{-1}
β = 90º	T = 100.0 (1) K
γ = 90º	Block, colorless
V = 3142.28 (9) Å^3	0.55 × 0.12 × 0.12 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	9693 independent reflections
Radiation source: fine-focus sealed tube	6638 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.062
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 40.0º
T = 100.0(1) K	θ_{min} = 1.1º
ω scans	h = −41→48

Absorption correction: multi-scan (SADABS; Bruker, 2005)

T_{min} = 0.320, T_{max} = 0.726

142737 measured reflections

k = −48→44

l = −7→7

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.033

$wR(F^2)$ = 0.097

S = 1.07

9693 reflections

211 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0455P)^2 + 0.774P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.003

$\Delta\rho_{max}$ = 0.71 e Å^{-3}

$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Extinction correction: none

Special details

Experimental. The low-temperature data was collected with the Oxford Cryosystem Cobra low-temperature attachment.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger. Data up to 2theta = 80 degrees is used in the final refinement

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.332148 (4)	0.255150 (5)	−0.02867 (3)	0.01727 (4)
O1	0.44846 (4)	0.45051 (4)	0.6855 (3)	0.02383 (19)
O2	0.71139 (3)	0.49941 (3)	1.1449 (2)	0.01727 (16)
O3	0.76246 (3)	0.42445 (3)	0.88670 (19)	0.01535 (15)
O4	0.71518 (3)	0.34984 (3)	0.5922 (2)	0.01822 (16)
C1	0.44558 (5)	0.32060 (5)	0.4779 (3)	0.0174 (2)
H1A	0.4729	0.3097	0.5908	0.021*
C2	0.41434 (4)	0.28571 (4)	0.3389 (3)	0.0167 (2)
H2A	0.4201	0.2515	0.3625	0.020*
C3	0.37450 (4)	0.30249 (4)	0.1647 (3)	0.01471 (19)
C4	0.36484 (4)	0.35344 (4)	0.1273 (3)	0.01654 (19)
H4A	0.3383	0.3642	0.0071	0.020*

C5	0.39549 (4)	0.38788 (4)	0.2725 (3)	0.01630 (19)
H5A	0.3890	0.4220	0.2523	0.020*
C6	0.43604 (4)	0.37201 (5)	0.4488 (3)	0.01541 (19)
C7	0.46729 (4)	0.41065 (5)	0.6074 (3)	0.0169 (2)
C8	0.52093 (4)	0.39895 (5)	0.6544 (3)	0.0179 (2)
H8A	0.5337	0.3689	0.5811	0.022*
C9	0.55163 (4)	0.43071 (5)	0.8004 (3)	0.0167 (2)
H9A	0.5369	0.4586	0.8901	0.020*
C10	0.60602 (4)	0.42570 (4)	0.8320 (3)	0.01485 (19)
C11	0.63128 (4)	0.46298 (4)	0.9952 (3)	0.01516 (19)
H11A	0.6132	0.4878	1.0950	0.018*
C12	0.68354 (4)	0.46278 (4)	1.0079 (2)	0.01394 (18)
C13	0.71100 (4)	0.42475 (4)	0.8662 (3)	0.01360 (18)
C14	0.68527 (4)	0.38623 (4)	0.7139 (3)	0.01435 (18)
C15	0.63325 (4)	0.38689 (4)	0.6936 (3)	0.01565 (19)
H15A	0.6165	0.3617	0.5887	0.019*
C16	0.68428 (5)	0.53482 (5)	1.3242 (3)	0.0180 (2)
H16A	0.7073	0.5587	1.4098	0.027*
H16B	0.6670	0.5175	1.4837	0.027*
H16C	0.6604	0.5521	1.1996	0.027*
C17	0.78718 (5)	0.43783 (5)	0.6083 (3)	0.0191 (2)
H17A	0.8228	0.4337	0.6311	0.029*
H17B	0.7799	0.4722	0.5601	0.029*
H17C	0.7754	0.4165	0.4485	0.029*
C18	0.69070 (5)	0.31032 (5)	0.4311 (3)	0.0198 (2)
H18A	0.7153	0.2866	0.3617	0.030*
H18B	0.6731	0.3241	0.2608	0.030*
H18C	0.6673	0.2938	0.5625	0.030*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01487 (6)	0.01441 (6)	0.02252 (6)	−0.00131 (4)	−0.00146 (4)	−0.00109 (4)
O1	0.0156 (4)	0.0196 (4)	0.0364 (5)	0.0027 (3)	−0.0021 (4)	−0.0084 (4)
O2	0.0142 (4)	0.0161 (4)	0.0215 (4)	−0.0018 (3)	−0.0011 (3)	−0.0040 (3)
O3	0.0099 (3)	0.0223 (4)	0.0138 (3)	0.0010 (3)	−0.0009 (3)	0.0006 (3)
O4	0.0127 (4)	0.0169 (4)	0.0250 (4)	0.0019 (3)	−0.0016 (3)	−0.0061 (3)
C1	0.0148 (5)	0.0171 (5)	0.0204 (5)	0.0026 (4)	−0.0025 (4)	−0.0007 (4)
C2	0.0168 (5)	0.0140 (5)	0.0193 (5)	0.0026 (4)	−0.0012 (4)	0.0000 (4)
C3	0.0123 (4)	0.0145 (5)	0.0174 (5)	−0.0013 (3)	0.0011 (4)	−0.0005 (4)
C4	0.0132 (5)	0.0153 (5)	0.0211 (5)	0.0012 (4)	−0.0019 (4)	0.0017 (4)
C5	0.0131 (5)	0.0137 (5)	0.0221 (5)	0.0012 (4)	−0.0009 (4)	0.0008 (4)
C6	0.0115 (4)	0.0165 (5)	0.0182 (5)	0.0005 (4)	0.0003 (4)	−0.0012 (4)
C7	0.0120 (5)	0.0179 (5)	0.0209 (5)	0.0003 (4)	−0.0006 (4)	−0.0018 (4)
C8	0.0121 (5)	0.0186 (5)	0.0231 (5)	0.0014 (4)	−0.0007 (4)	−0.0040 (4)
C9	0.0122 (5)	0.0158 (5)	0.0220 (5)	0.0004 (4)	0.0002 (4)	−0.0014 (4)
C10	0.0115 (4)	0.0145 (5)	0.0185 (5)	−0.0001 (3)	−0.0003 (3)	0.0001 (4)
C11	0.0124 (4)	0.0141 (5)	0.0190 (5)	−0.0002 (3)	0.0000 (4)	0.0001 (4)

C12	0.0130 (4)	0.0133 (4)	0.0156 (4)	−0.0011 (3)	−0.0005 (3)	0.0007 (3)
C13	0.0110 (4)	0.0156 (5)	0.0143 (4)	0.0000 (3)	−0.0008 (3)	0.0007 (3)
C14	0.0130 (4)	0.0137 (4)	0.0163 (4)	0.0014 (3)	−0.0008 (3)	−0.0003 (4)
C15	0.0131 (5)	0.0147 (5)	0.0191 (5)	0.0000 (4)	−0.0013 (4)	−0.0015 (4)
C16	0.0196 (5)	0.0159 (5)	0.0186 (5)	0.0003 (4)	−0.0014 (4)	−0.0019 (4)
C17	0.0148 (5)	0.0258 (6)	0.0168 (5)	−0.0009 (4)	0.0009 (4)	0.0024 (4)
C18	0.0170 (5)	0.0165 (5)	0.0259 (6)	0.0002 (4)	−0.0004 (4)	−0.0051 (4)

Geometric parameters (Å, °)

Br1—C3	1.8967 (11)	C8—H8A	0.9300
O1—C7	1.2247 (15)	C9—C10	1.4624 (16)
O2—C12	1.3678 (14)	C9—H9A	0.9300
O2—C16	1.4292 (15)	C10—C11	1.4007 (16)
O3—C13	1.3746 (13)	C10—C15	1.4039 (16)
O3—C17	1.4413 (15)	C11—C12	1.3941 (16)
O4—C14	1.3658 (14)	C11—H11A	0.9300
O4—C18	1.4294 (15)	C12—C13	1.3984 (16)
C1—C2	1.3916 (17)	C13—C14	1.4065 (16)
C1—C6	1.3997 (17)	C14—C15	1.3894 (16)
C1—H1A	0.9300	C15—H15A	0.9300
C2—C3	1.3862 (16)	C16—H16A	0.9600
C2—H2A	0.9300	C16—H16B	0.9600
C3—C4	1.3917 (16)	C16—H16C	0.9600
C4—C5	1.3866 (17)	C17—H17A	0.9600
C4—H4A	0.9300	C17—H17B	0.9600
C5—C6	1.3981 (16)	C17—H17C	0.9600
C5—H5A	0.9300	C18—H18A	0.9600
C6—C7	1.4990 (17)	C18—H18B	0.9600
C7—C8	1.4777 (16)	C18—H18C	0.9600
C8—C9	1.3428 (17)		
C12—O2—C16	116.30 (9)	C12—C11—C10	119.88 (11)
C13—O3—C17	113.47 (9)	C12—C11—H11A	120.1
C14—O4—C18	116.97 (9)	C10—C11—H11A	120.1
C2—C1—C6	120.31 (11)	O2—C12—C11	123.86 (11)
C2—C1—H1A	119.8	O2—C12—C13	115.59 (10)
C6—C1—H1A	119.8	C11—C12—C13	120.51 (10)
C3—C2—C1	119.24 (11)	O3—C13—C12	119.79 (10)
C3—C2—H2A	120.4	O3—C13—C14	120.92 (10)
C1—C2—H2A	120.4	C12—C13—C14	119.25 (10)
C2—C3—C4	121.50 (11)	O4—C14—C15	124.47 (10)
C2—C3—Br1	119.46 (9)	O4—C14—C13	115.00 (10)
C4—C3—Br1	119.04 (9)	C15—C14—C13	120.54 (10)
C5—C4—C3	118.81 (11)	C14—C15—C10	119.81 (11)
C5—C4—H4A	120.6	C14—C15—H15A	120.1
C3—C4—H4A	120.6	C10—C15—H15A	120.1
C4—C5—C6	120.89 (11)	O2—C16—H16A	109.5
C4—C5—H5A	119.6	O2—C16—H16B	109.5
C6—C5—H5A	119.6	H16A—C16—H16B	109.5

C5—C6—C1	119.21 (11)	O2—C16—H16C	109.5
C5—C6—C7	118.87 (11)	H16A—C16—H16C	109.5
C1—C6—C7	121.89 (11)	H16B—C16—H16C	109.5
O1—C7—C8	122.66 (11)	O3—C17—H17A	109.5
O1—C7—C6	120.02 (11)	O3—C17—H17B	109.5
C8—C7—C6	117.29 (10)	H17A—C17—H17B	109.5
C9—C8—C7	121.62 (11)	O3—C17—H17C	109.5
C9—C8—H8A	119.2	H17A—C17—H17C	109.5
C7—C8—H8A	119.2	H17B—C17—H17C	109.5
C8—C9—C10	126.33 (11)	O4—C18—H18A	109.5
C8—C9—H9A	116.8	O4—C18—H18B	109.5
C10—C9—H9A	116.8	H18A—C18—H18B	109.5
C11—C10—C15	119.93 (10)	O4—C18—H18C	109.5
C11—C10—C9	117.44 (10)	H18A—C18—H18C	109.5
C15—C10—C9	122.55 (10)	H18B—C18—H18C	109.5
C6—C1—C2—C3	1.71 (18)	C16—O2—C12—C11	10.38 (16)
C1—C2—C3—C4	−0.42 (18)	C16—O2—C12—C13	−171.99 (10)
C1—C2—C3—Br1	179.40 (9)	C10—C11—C12—O2	175.59 (11)
C2—C3—C4—C5	−1.05 (18)	C10—C11—C12—C13	−1.93 (17)
Br1—C3—C4—C5	179.12 (9)	C17—O3—C13—C12	−107.90 (12)
C3—C4—C5—C6	1.25 (18)	C17—O3—C13—C14	74.48 (14)
C4—C5—C6—C1	0.01 (18)	O2—C12—C13—O3	3.79 (15)
C4—C5—C6—C7	−178.49 (11)	C11—C12—C13—O3	−178.50 (10)
C2—C1—C6—C5	−1.51 (18)	O2—C12—C13—C14	−178.55 (10)
C2—C1—C6—C7	176.94 (11)	C11—C12—C13—C14	−0.84 (17)
C5—C6—C7—O1	28.97 (18)	C18—O4—C14—C15	0.71 (17)
C1—C6—C7—O1	−149.50 (13)	C18—O4—C14—C13	−179.04 (11)
C5—C6—C7—C8	−149.21 (12)	O3—C13—C14—O4	−0.08 (16)
C1—C6—C7—C8	32.33 (17)	C12—C13—C14—O4	−177.72 (10)
O1—C7—C8—C9	3.4 (2)	O3—C13—C14—C15	−179.84 (10)
C6—C7—C8—C9	−178.44 (12)	C12—C13—C14—C15	2.53 (17)
C7—C8—C9—C10	−173.04 (12)	O4—C14—C15—C10	178.83 (11)
C8—C9—C10—C11	−179.68 (12)	C13—C14—C15—C10	−1.43 (18)
C8—C9—C10—C15	3.6 (2)	C11—C10—C15—C14	−1.35 (18)
C15—C10—C11—C12	3.03 (17)	C9—C10—C15—C14	175.30 (11)
C9—C10—C11—C12	−173.80 (11)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11A···O1^{i}	0.93	2.52	3.4391 (16)	170
C17—H17C···O3^{ii}	0.96	2.52	3.2789 (16)	136
C16—H16B···Cg1^{iii}	0.96	2.97	3.8080 (14)	147

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) *x*, *y*, *z*−1; (iii) *x*, *y*, *z*+1.

Fig. 1

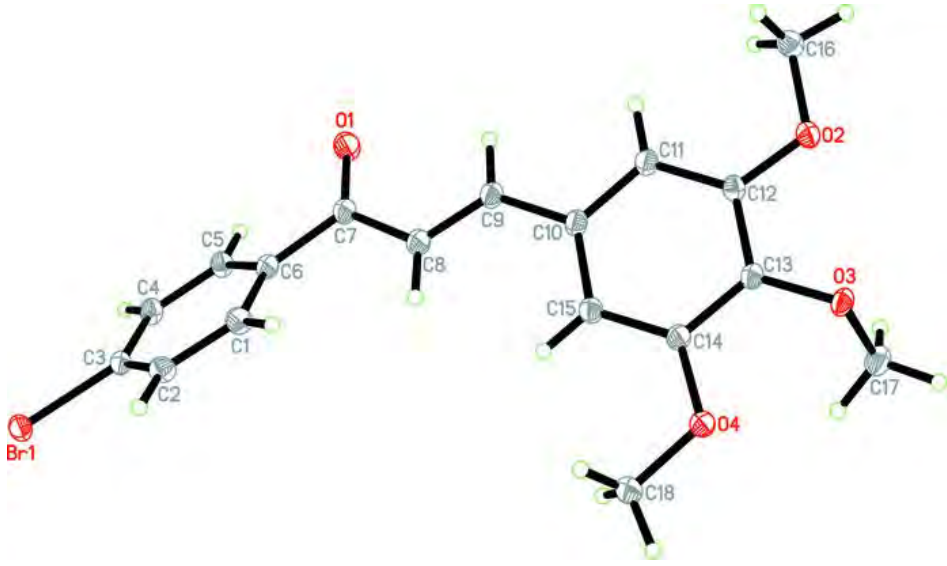
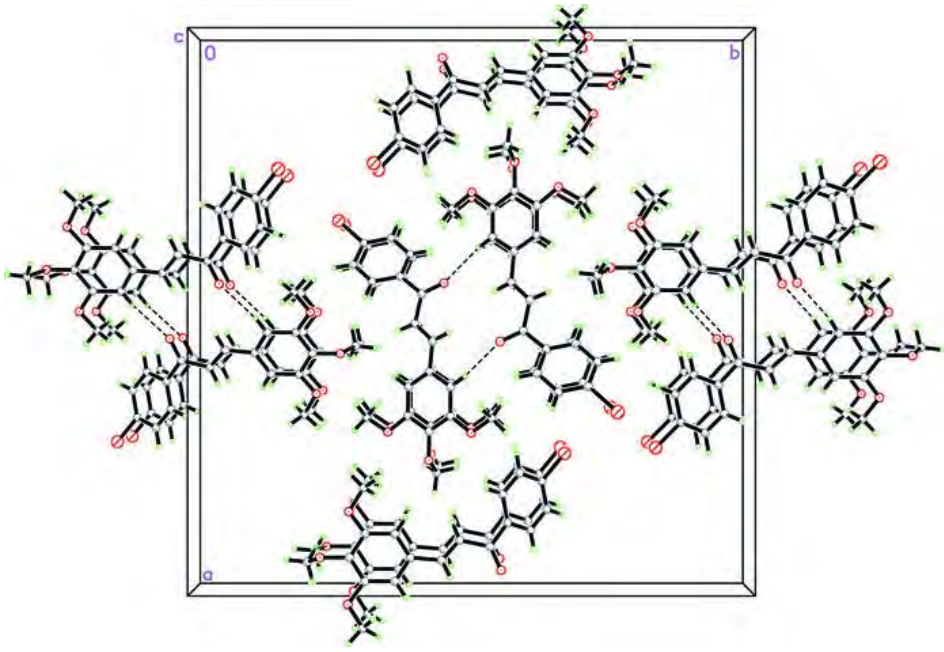


Fig. 2

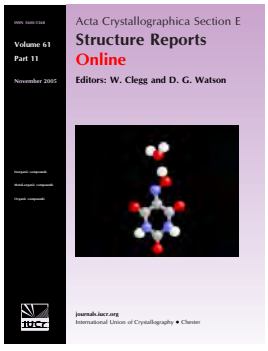


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-3-(Anthracen-9-yl)-1-(4-bromophenyl)prop-2-en-1-one

Thitipone Suwunwong, Suchada Chantrapromma, Chatchanok Karalai, Paradorn Pakdeevanich and Hoong-Kun Fun

Acta Cryst. (2009). E**65**, o420–o421



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-3-(Anthracen-9-yl)-1-(4-bromophenyl)-prop-2-en-1-one[1]

Thitipone Suwunwong,[a] Suchada Chantrapromma,[b]* Chatchanok Karalai,[b] Paradorn Pakdeevanich[c] and Hoong-Kun Fun[d]§

[a]Department of Chemistry and Center of Excellence for Innovation in Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [c]Department of Physics, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [d]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

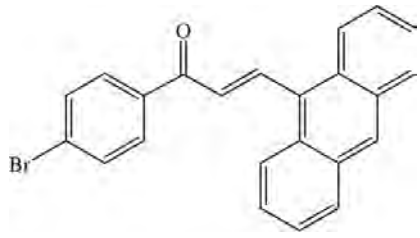
Received 31 December 2008; accepted 26 January 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.076; data-to-parameter ratio = 21.5.

In the title molecule, $C_{23}H_{15}BrO$, the prop-2-en-1-one unit is planar and it makes dihedral angles of 20.9 (1) and 45.8 (1)°, respectively, with the 4-bromophenyl ring and the anthracene ring system. The interplanar angle between the 4-bromophenyl ring and the anthracene ring system is 35.52 (7)°. In the crystal structure, molecules are linked into dimers by C—H···Br hydrogen bonds, and the dimers are linked into a zigzag network parallel to the *bc* plane by weak C—H···O hydrogen bonds and C—H···π interactions involving the central benzene ring of the anthracene ring system.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related structures, see: Ng *et al.* (2006); Patil *et al.* (2006); Patil, Chantrapromma *et al.* (2007); Suwunwong *et al.* (2009). For background and applications of chalcones, see: Jung *et al.* (2008); Patil, Chantrapromma *et al.* (2007); Patil, Dharmaprakash *et al.* (2007); Patil & Dharmaprakash (2008); Prasad *et al.* (2008).



Experimental

Crystal data

$C_{23}H_{15}BrO$
M_r = 387.25
Monoclinic, $P2_1/c$
a = 5.3792 (1) Å
b = 19.1030 (4) Å
c = 16.3005 (4) Å
β = 95.944 (1)°
V = 1666.02 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.47 mm^{-1}
T = 100.0 (1) K
0.57 × 0.27 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.331, T_{max} = 0.714
29994 measured reflections
4866 independent reflections
3803 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.076
S = 1.02
4866 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C18–C23 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8*A*···O1^{i}	0.93	2.42	3.308 (2)	159
C13—H13*A*···O1^{ii}	0.93	2.57	3.288 (2)	135
C21—H21*A*···Br1^{iii}	0.93	2.93	3.4722 (19)	119
C9—H9*A*···*Cg*1^{iv}	0.93	2.83	3.4479 (18)	125

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+2, -y+1, -z+2$; (iii) $-x, -y+1, -z+1$; (iv) $x+1, y, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

Financial support from the Center of Excellence for Innovation in Chemistry (PERCH-CIC), Commision on Higher Education, Ministry of Education, Thailand, is gratefully acknowledged. The authors also thank the Thailand Research Fund (TRF) and the Prince of Songkla University for financial support, and the Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012.

[1] This paper is dedicated to the late Her Royal Highness Princess Galyani Vadhana Krom Luang Naradhiwas Rajanagarindra for her patronage of Science in Thailand.
§ Additional correspondence author, e-mail: hkfun@usm.my.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2756).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Jung, Y. J., Son, K. I., Oh, Y. E. & Noh, D. Y. (2008). *Polyhedron*, **27**, 861–867.

Ng, S.-L., Shettigar, V., Razak, I. A., Fun, H.-K., Patil, P. S. & Dharmaprakash, S. M. (2006). *Acta Cryst.* E**62**, o1570–o1572.

Patil, P. S., Chantrapromma, S., Fun, H.-K., Dharmaprakash, S. M. & Babu, H. B. R. (2007). *Acta Cryst.* E**63**, o2612.

Patil, P. S. & Dharmaprakash, S. M. (2008). *Mater. Lett.* **62**, 451–453.

Patil, P. S., Dharmaprakash, S. M., Ramakrishna, K., Fun, H. K., Sai Santosh Kumar, R. & Narayana Rao, D. (2007). *J. Cryst. Growth*, **303**, 520–524.

Patil, P. S., Rosli, M. M., Fun, H.-K., Razak, I. A., Puranik, V. G. & Dharmaprakash, S. M. (2006). *Acta Cryst.* E**62**, o4798–o4799.

Prasad, Y. R., Kumar, P. R., Smile, D. J. & Babu, P. A. (2008). *Arkivoc*, **11**, 266–276.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Suwunwong, T., Chantrapromma, S. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o120.

supplementary materials

Acta Cryst. (2009). E**65**, o420-o421 [**doi:10.1107/S1600536809003122**]

(*E*)-3-(Anthracen-9-yl)-1-(4-bromophenyl)prop-2-en-1-one

T. Suwunwong, S. Chantrapromma, C. Karalai, P. Pakdeevanich and H.-K. Fun

Comment

Chalcones are compounds which have a wide range of applications covering from non-linear optical (Patil & Dharmaprakash, 2008) and electro-active fluorescent materials (Jung *et al.*, 2008) to materials with various biological activities (Prasad *et al.*, 2008). Our previous work (Patil Dharmaprakash *et al.*, 2007) has reported that 1-(4-bromophenyl)-3-(2,4,5-trimethoxyphenyl)-propenone shows efficient second-order nonlinear optical properties. The various interesting properties of chalcone derivatives lead us to synthesize the title chalcone derivative in order to study its photoluminescence and antimicrobial activities.

The molecule of the title chalcone derivative (Fig. 1) exists in an *E* configuration with respect to the C8═C9 double bond [1.333 (2) Å]. The anthracene ring system is planar, with atom C21 deviating a maximum of 0.147 (2) Å. The molecule is twisted as indicated by the interplanar angle between 4-bromophenyl ring and anthracene ring system of 35.52 (7)°, and torsion angles C5–C6–C7–C8 of 22.9 (1)° and C8–C9–C10–C23 of -50.2 (3)°. The pro-2-en-1-one unit (C7-C9/O1) is planar as evidenced by the torsion angle O1–C7–C8–C9 of 0.1 (3)°. The O1/C6-C9 plane makes dihedral angles of 20.9 (1)° and 45.8 (1)°, respectively, with the 4-bromophenyl ring and anthracene ring system. The bond distances show normal values (Allen *et al.*, 1987) and are comparable with those observed in related structures (Ng *et al.*, 2006; Patil *et al.*, 2006; Patil, Chantrapromma *et al.*, 2007; Suwunwong *et al.*, 2009).

In the crystal packing (Fig. 2), the molecules are linked into dimers by weak C—H···Br interactions (Table 1) and the dimers are further linked into a zigzag network parallel to the *bc* plane by weak C—H···O and C—H···π interactions (Table 1).

Experimental

The title compound was synthesized by the condensation of anthracene-9-carbaldehyde (0.01 mol) with 4-bromoacetophenone (0.01 mol) in ethanol (40 ml) in the presence of NaOH (10 ml, 10%). After stirring for 2 h, a yellow solid appeared and was then collected by filtration, washed with distilled water, dried and purified by repeated recrystallization from acetone. Yellow plate-shaped single crystals of the title compound suitable for *X*-ray structure determination were obtained by slow evaporation of an acetone solution at room temperature after several days.

Refinement

All H atoms were placed in calculated positions, with C-H = 0.93 Å, $U_{iso} = 1.2U_{eq}$(C). The highest residual electron density peak is located at 0.76 Å from Br1 and the deepest hole is located at 0.69 Å from Br1.

Figures

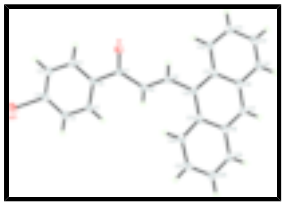


Fig. 1. The molecular structure of the title compound, showing 60% probability displacement ellipsoids and the atom-numbering scheme.

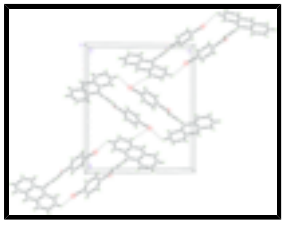


Fig. 2. Part of the crystal packing of the title compound, viewed along the *a* axis, showing hydrogen-bonded (dashed lines) dimers.

(*E*)-3-(Anthracen-9-yl)-1-(4-bromophenyl)prop-2-en-1-one

Crystal data

$C_{23}H_{15}BrO$	F_{000} = 784
M_r = 387.25	D_x = 1.544 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4866 reflections
a = 5.3792 (1) Å	θ = 2.1–30.0º
b = 19.1030 (4) Å	μ = 2.47 mm^{-1}
c = 16.3005 (4) Å	T = 100.0 (1) K
β = 95.944 (1)º	Plate, yellow
V = 1666.02 (6) Å^3	0.57 × 0.27 × 0.15 mm
Z = 4	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	4866 independent reflections
Radiation source: fine-focus sealed tube	3803 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.036
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 30.0º
T = 100.0(1) K	θ_{min} = 2.1º
ω scans	h = −7→7
Absorption correction: multi-scan (SADABS; Bruker, 2005)	k = −26→26
T_{min} = 0.331, T_{max} = 0.714	l = −22→22
29994 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.032$	H-atom parameters constrained
$wR(F^2) = 0.076$	$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2 + 1.1607P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
4866 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}
226 parameters	$\Delta\rho_{min} = -0.54$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.32584 (4)	0.344013 (10)	0.411244 (11)	0.02530 (7)
O1	0.9073 (2)	0.51056 (7)	0.74450 (8)	0.0213 (3)
C1	0.7274 (4)	0.41439 (9)	0.62533 (11)	0.0201 (4)
H1A	0.8800	0.4043	0.6552	0.024*
C2	0.6526 (4)	0.37631 (10)	0.55484 (12)	0.0219 (4)
H2A	0.7530	0.3408	0.5374	0.026*
C3	0.4261 (4)	0.39203 (9)	0.51095 (10)	0.0180 (4)
C4	0.2711 (4)	0.44314 (10)	0.53719 (11)	0.0206 (4)
H4A	0.1176	0.4523	0.5075	0.025*
C5	0.3464 (3)	0.48075 (10)	0.60836 (10)	0.0184 (4)
H5A	0.2419	0.5149	0.6267	0.022*
C6	0.5781 (3)	0.46766 (9)	0.65244 (10)	0.0152 (3)
C7	0.6813 (3)	0.51052 (9)	0.72478 (10)	0.0159 (3)
C8	0.5083 (3)	0.55087 (9)	0.77158 (10)	0.0159 (3)
H8A	0.3374	0.5499	0.7555	0.019*
C9	0.5978 (3)	0.58858 (9)	0.83690 (10)	0.0161 (3)
H9A	0.7707	0.5920	0.8471	0.019*

C10	0.4492 (3)	0.62533 (9)	0.89440 (10)	0.0158 (3)
C11	0.5151 (3)	0.61533 (9)	0.98020 (10)	0.0157 (3)
C12	0.7151 (4)	0.57046 (10)	1.01164 (11)	0.0195 (4)
H12A	0.8015	0.5452	0.9749	0.023*
C13	0.7822 (4)	0.56385 (10)	1.09428 (11)	0.0219 (4)
H13A	0.9149	0.5349	1.1132	0.026*
C14	0.6506 (4)	0.60082 (10)	1.15145 (11)	0.0220 (4)
H14A	0.7002	0.5970	1.2076	0.026*
C15	0.4527 (4)	0.64183 (10)	1.12478 (11)	0.0224 (4)
H15A	0.3652	0.6648	1.1631	0.027*
C16	0.3767 (3)	0.65030 (9)	1.03860 (10)	0.0169 (3)
C17	0.1715 (4)	0.69108 (9)	1.01020 (10)	0.0187 (4)
H17A	0.0769	0.7118	1.0482	0.022*
C18	0.1036 (3)	0.70178 (9)	0.92633 (10)	0.0167 (3)
C19	−0.1040 (4)	0.74534 (9)	0.89840 (11)	0.0196 (4)
H19A	−0.2054	0.7629	0.9365	0.023*
C20	−0.1560 (4)	0.76158 (9)	0.81740 (11)	0.0211 (4)
H20A	−0.2925	0.7898	0.8002	0.025*
C21	−0.0008 (4)	0.73530 (9)	0.75909 (11)	0.0205 (4)
H21A	−0.0309	0.7487	0.7041	0.025*
C22	0.1913 (3)	0.69077 (9)	0.78224 (10)	0.0184 (4)
H22A	0.2862	0.6730	0.7424	0.022*
C23	0.2504 (3)	0.67066 (9)	0.86689 (10)	0.0153 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03907 (13)	0.02093 (10)	0.01575 (9)	−0.00663 (8)	0.00217 (7)	−0.00386 (7)
O1	0.0137 (7)	0.0297 (7)	0.0203 (6)	0.0009 (5)	0.0009 (5)	−0.0031 (5)
C1	0.0181 (10)	0.0199 (9)	0.0219 (9)	0.0037 (7)	0.0000 (7)	−0.0002 (7)
C2	0.0238 (10)	0.0180 (9)	0.0239 (9)	0.0060 (7)	0.0029 (7)	−0.0027 (7)
C3	0.0238 (10)	0.0159 (8)	0.0146 (8)	−0.0052 (7)	0.0032 (6)	−0.0018 (6)
C4	0.0170 (10)	0.0258 (9)	0.0182 (8)	−0.0003 (7)	−0.0010 (7)	−0.0016 (7)
C5	0.0158 (9)	0.0228 (9)	0.0168 (8)	0.0022 (7)	0.0020 (6)	−0.0031 (7)
C6	0.0155 (9)	0.0169 (8)	0.0134 (7)	−0.0013 (6)	0.0024 (6)	0.0007 (6)
C7	0.0156 (9)	0.0176 (8)	0.0148 (7)	−0.0011 (7)	0.0027 (6)	0.0005 (6)
C8	0.0119 (9)	0.0206 (8)	0.0155 (7)	−0.0010 (7)	0.0022 (6)	−0.0008 (6)
C9	0.0133 (9)	0.0179 (8)	0.0175 (8)	−0.0016 (7)	0.0032 (6)	0.0007 (6)
C10	0.0157 (9)	0.0163 (8)	0.0155 (8)	−0.0035 (7)	0.0024 (6)	−0.0019 (6)
C11	0.0163 (9)	0.0155 (8)	0.0154 (8)	−0.0034 (7)	0.0018 (6)	−0.0006 (6)
C12	0.0195 (10)	0.0211 (9)	0.0184 (8)	−0.0005 (7)	0.0034 (7)	−0.0008 (7)
C13	0.0209 (10)	0.0240 (9)	0.0202 (8)	−0.0003 (8)	−0.0003 (7)	0.0026 (7)
C14	0.0292 (11)	0.0223 (9)	0.0140 (8)	−0.0036 (8)	−0.0004 (7)	0.0005 (7)
C15	0.0307 (11)	0.0227 (9)	0.0141 (8)	−0.0017 (8)	0.0042 (7)	−0.0017 (7)
C16	0.0210 (9)	0.0144 (8)	0.0156 (8)	−0.0040 (7)	0.0036 (6)	−0.0015 (6)
C17	0.0225 (10)	0.0168 (8)	0.0175 (8)	−0.0003 (7)	0.0054 (7)	−0.0032 (6)
C18	0.0182 (9)	0.0142 (8)	0.0178 (8)	−0.0032 (7)	0.0021 (6)	−0.0015 (6)
C19	0.0193 (10)	0.0156 (8)	0.0244 (9)	−0.0002 (7)	0.0050 (7)	−0.0024 (7)

C20	0.0200 (10)	0.0160 (8)	0.0266 (9)	−0.0010 (7)	−0.0018 (7)	0.0006 (7)
C21	0.0216 (10)	0.0209 (9)	0.0182 (8)	−0.0033 (7)	−0.0016 (7)	0.0014 (7)
C22	0.0192 (10)	0.0191 (9)	0.0168 (8)	−0.0029 (7)	0.0020 (6)	−0.0020 (7)
C23	0.0155 (9)	0.0154 (8)	0.0150 (7)	−0.0048 (6)	0.0013 (6)	−0.0018 (6)

Geometric parameters (Å, °)

Br1—C3	1.8954 (17)	C12—C13	1.364 (2)
O1—C7	1.225 (2)	C12—H12A	0.93
C1—C2	1.384 (3)	C13—C14	1.416 (3)
C1—C6	1.396 (2)	C13—H13A	0.93
C1—H1A	0.93	C14—C15	1.356 (3)
C2—C3	1.380 (3)	C14—H14A	0.93
C2—H2A	0.93	C15—C16	1.431 (2)
C3—C4	1.380 (3)	C15—H15A	0.93
C4—C5	1.389 (2)	C16—C17	1.391 (3)
C4—H4A	0.93	C17—C18	1.393 (2)
C5—C6	1.395 (2)	C17—H17A	0.93
C5—H5A	0.93	C18—C19	1.429 (3)
C6—C7	1.495 (2)	C18—C23	1.440 (2)
C7—C8	1.480 (2)	C19—C20	1.357 (3)
C8—C9	1.333 (2)	C19—H19A	0.93
C8—H8A	0.93	C20—C21	1.420 (3)
C9—C10	1.472 (2)	C20—H20A	0.93
C9—H9A	0.93	C21—C22	1.361 (3)
C10—C23	1.413 (3)	C21—H21A	0.93
C10—C11	1.420 (2)	C22—C23	1.436 (2)
C11—C12	1.428 (3)	C22—H22A	0.93
C11—C16	1.433 (2)		
C2—C1—C6	121.23 (17)	C11—C12—H12A	119.3
C2—C1—H1A	119.4	C12—C13—C14	120.32 (18)
C6—C1—H1A	119.4	C12—C13—H13A	119.8
C3—C2—C1	118.75 (17)	C14—C13—H13A	119.8
C3—C2—H2A	120.6	C15—C14—C13	120.41 (17)
C1—C2—H2A	120.6	C15—C14—H14A	119.8
C4—C3—C2	121.49 (16)	C13—C14—H14A	119.8
C4—C3—Br1	118.73 (14)	C14—C15—C16	121.00 (17)
C2—C3—Br1	119.78 (14)	C14—C15—H15A	119.5
C3—C4—C5	119.44 (17)	C16—C15—H15A	119.5
C3—C4—H4A	120.3	C17—C16—C15	121.78 (16)
C5—C4—H4A	120.3	C17—C16—C11	119.28 (16)
C4—C5—C6	120.34 (17)	C15—C16—C11	118.94 (17)
C4—C5—H5A	119.8	C16—C17—C18	121.81 (16)
C6—C5—H5A	119.8	C16—C17—H17A	119.1
C5—C6—C1	118.69 (16)	C18—C17—H17A	119.1
C5—C6—C7	123.18 (16)	C17—C18—C19	120.98 (16)
C1—C6—C7	118.04 (16)	C17—C18—C23	119.57 (16)
O1—C7—C8	121.62 (16)	C19—C18—C23	119.41 (15)
O1—C7—C6	119.02 (15)	C20—C19—C18	121.24 (17)

C8—C7—C6	119.35 (15)	C20—C19—H19A	119.4
C9—C8—C7	119.93 (16)	C18—C19—H19A	119.4
C9—C8—H8A	120.0	C19—C20—C21	119.62 (18)
C7—C8—H8A	120.0	C19—C20—H20A	120.2
C8—C9—C10	126.25 (17)	C21—C20—H20A	120.2
C8—C9—H9A	116.9	C22—C21—C20	121.16 (16)
C10—C9—H9A	116.9	C22—C21—H21A	119.4
C23—C10—C11	119.94 (15)	C20—C21—H21A	119.4
C23—C10—C9	122.24 (15)	C21—C22—C23	121.31 (17)
C11—C10—C9	117.80 (16)	C21—C22—H22A	119.3
C10—C11—C12	122.43 (16)	C23—C22—H22A	119.3
C10—C11—C16	119.84 (16)	C10—C23—C22	123.65 (16)
C12—C11—C16	117.72 (15)	C10—C23—C18	119.29 (15)
C13—C12—C11	121.49 (17)	C22—C23—C18	116.99 (16)
C13—C12—H12A	119.3		
C6—C1—C2—C3	0.3 (3)	C13—C14—C15—C16	−1.8 (3)
C1—C2—C3—C4	−1.9 (3)	C14—C15—C16—C17	178.78 (18)
C1—C2—C3—Br1	176.90 (14)	C14—C15—C16—C11	−0.8 (3)
C2—C3—C4—C5	1.4 (3)	C10—C11—C16—C17	3.0 (3)
Br1—C3—C4—C5	−177.43 (14)	C12—C11—C16—C17	−176.19 (16)
C3—C4—C5—C6	0.8 (3)	C10—C11—C16—C15	−177.41 (16)
C4—C5—C6—C1	−2.4 (3)	C12—C11—C16—C15	3.4 (2)
C4—C5—C6—C7	174.05 (17)	C15—C16—C17—C18	177.22 (17)
C2—C1—C6—C5	1.9 (3)	C11—C16—C17—C18	−3.2 (3)
C2—C1—C6—C7	−174.75 (17)	C16—C17—C18—C19	−178.36 (17)
C5—C6—C7—O1	−158.07 (17)	C16—C17—C18—C23	−0.7 (3)
C1—C6—C7—O1	18.4 (2)	C17—C18—C19—C20	173.37 (17)
C5—C6—C7—C8	22.9 (2)	C23—C18—C19—C20	−4.3 (3)
C1—C6—C7—C8	−160.59 (16)	C18—C19—C20—C21	−0.4 (3)
O1—C7—C8—C9	0.1 (3)	C19—C20—C21—C22	3.8 (3)
C6—C7—C8—C9	179.10 (16)	C20—C21—C22—C23	−2.3 (3)
C7—C8—C9—C10	−173.26 (16)	C11—C10—C23—C22	171.79 (16)
C8—C9—C10—C23	−50.2 (3)	C9—C10—C23—C22	−6.7 (3)
C8—C9—C10—C11	131.30 (19)	C11—C10—C23—C18	−5.1 (3)
C23—C10—C11—C12	−179.68 (17)	C9—C10—C23—C18	176.46 (16)
C9—C10—C11—C12	−1.1 (3)	C21—C22—C23—C10	−179.25 (18)
C23—C10—C11—C16	1.1 (3)	C21—C22—C23—C18	−2.3 (3)
C9—C10—C11—C16	179.69 (16)	C17—C18—C23—C10	4.9 (3)
C10—C11—C12—C13	177.21 (17)	C19—C18—C23—C10	−177.42 (16)
C16—C11—C12—C13	−3.6 (3)	C17—C18—C23—C22	−172.15 (16)
C11—C12—C13—C14	1.1 (3)	C19—C18—C23—C22	5.5 (2)
C12—C13—C14—C15	1.6 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8A···O1^{i}	0.93	2.42	3.308 (2)	159
C13—H13A···O1^{ii}	0.93	2.57	3.288 (2)	135

C21—H21A···Br1^{iii}	0.93	2.93	3.4722 (19)	119
C9—H9A···Cg1^{iv}	0.93	2.83	3.4479 (18)	125

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+2, -y+1, -z+2$; (iii) $-x, -y+1, -z+1$; (iv) $x+1, y, z$.

Fig. 1

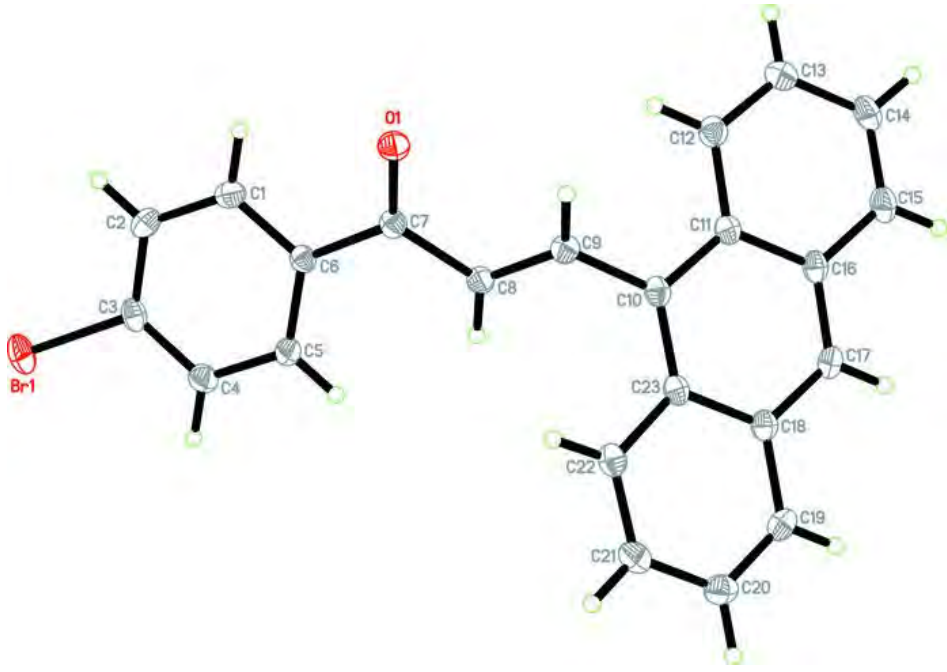
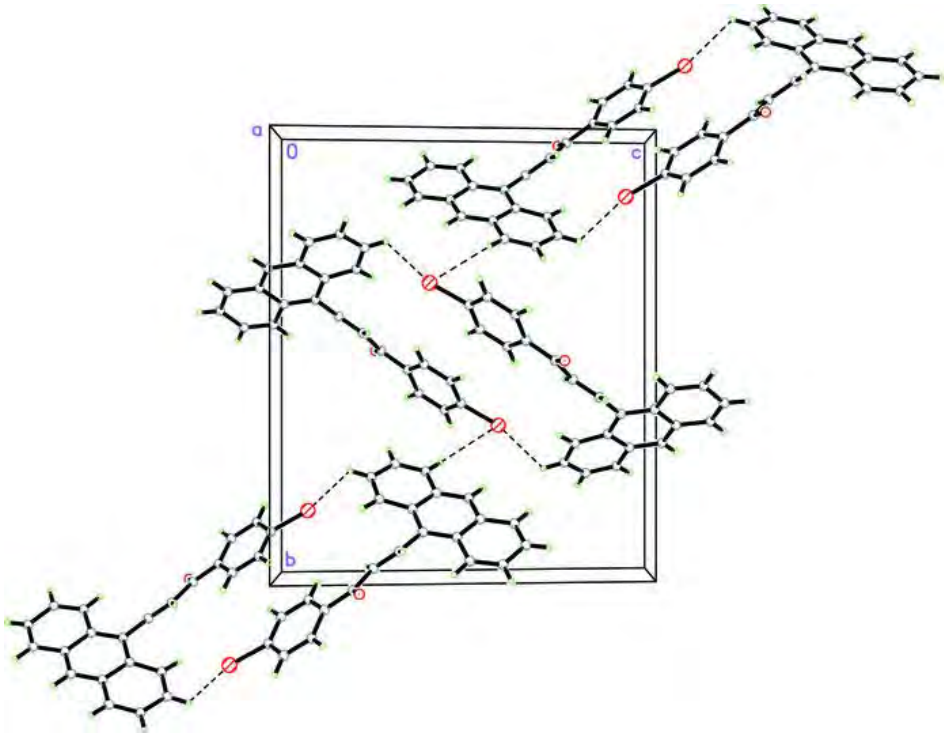


Fig. 2

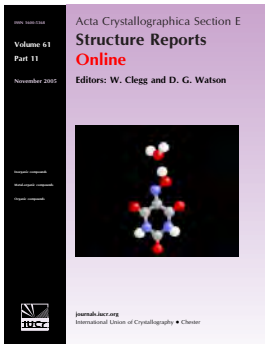


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*Z*)-3-(9-Anthryl)-1-(2-thienyl)prop-2-en-1-one

Hoong-Kun Fun, Thitipone Suwunwong, Nawong Boonnak and Suchada Chantrapromma

Acta Cryst. (2009). E**65**, o2168–o2169



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-3-(9-Anthryl)-1-(2-thienyl)prop-2-en-1-one[1]

Hoong-Kun Fun,[a]*§ Thitipone Suwunwong,[b] Nawong Boonnak[b] and Suchada Chantrapromma[b]¶

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

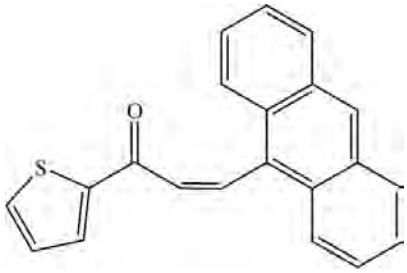
Received 3 August 2009; accepted 12 August 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.006 Å; R factor = 0.065; wR factor = 0.200; data-to-parameter ratio = 17.0.

There are two crystallographically independent molecules in the asymmetric unit of the title heteroaryl chalcone, $C_{21}H_{14}OS$: the dihedral angle between the thiophene and anthracene rings is 75.07 (17)° in one molecule and 76.32 (17)° in the other. The crystal structure is consolidated by short C···O [3.348 (5)–3.394 (5) Å], C···S [3.607 (5)–3.666 (5) Å] and S···O [2.926 (3) Å] contacts, as well as by C—H···π and π–π interactions [Cg···Cg = 3.745 (3) Å].

Related literature

For related structures, see: Chantrapromma *et al.* (2009); Suwunwong *et al.* (2009*a*,*b*). For background to and applications of chalcones, see: Oliveira *et al.* (2007); Patil & Dharmaprakash (2008); Saydam *et al.* (2003); Svetlichny *et al.* (2007). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{21}H_{14}OS$
M_r = 314.39
Orthorhombic, $Pna2_1$
a = 14.6675 (2) Å
b = 5.5096 (1) Å
c = 37.9823 (4) Å
V = 3069.43 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 100 K
0.30 × 0.12 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.939, T_{max} = 0.979
28929 measured reflections
6662 independent reflections
5348 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.200
S = 1.06
6662 reflections
391 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.82 e Å^{-3}
Absolute structure: Flack (1983), 3093 Friedel pairs
Flack parameter: 0.09 (15)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3*A*—H3*AA*···*Cg*3^{i}	0.93	2.99	3.679 (5)	132
C10*A*—H10*A*···*Cg*2^{ii}	0.93	2.95	3.694 (5)	138
C10*B*—H10*B*···*Cg*5^{i}	0.93	2.93	3.594 (5)	129
C15*A*—H15*A*···*Cg*3^{iii}	0.93	2.76	3.550 (5)	143
C15*B*—H15*B*···*Cg*6^{iii}	0.93	2.94	3.689 (5)	139
C19*A*—H19*A*···*Cg*4	0.93	2.72	3.486 (5)	140
C19*B*—H19*B*···*Cg*1^{iii}	0.93	2.72	3.458 (5)	137

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z$; (ii) $x-\frac{1}{2}, -y-\frac{1}{2}, z$; (iii) $x, y+1, z$. *Cg*1, *Cg*2, *Cg*3, *Cg*4, *Cg*5 and *Cg*6 are the centroids of the S1*A*/C18*A*–C21*A*, C1*A*–C6*A*, C8*A*–C13*A*, S1*B*/C18*B*–C21*B*, C1*B*–C6*B* and C8*B*–C13*B* rings, respectively.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for research grant (RSA 5280033) and Prince of Songkla University for financial support through the Crystal Materials Research Unit. The authors also thank Universiti Sains Malaysia for a Research University Golden Goose grant No. 1001/PFIZIK/811012.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2522).

References

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chantrapromma, S., Suwunwong, T., Karalai, C. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o893–o894.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

[1] This paper is dedicated to Her Majesty, Queen Sirikit of Thailand on the occasion of her 77th Birthday Anniversary which fell on August 12th, 2009.
§ Thomson Reuters ResearcherID: A-3561-2009.
¶ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Oliveira, E., Vicente, M., Valencia, L., Macías, A., Bértolo, E., Bastida, R. & Lodeiro, C. (2007). *Inorg. Chim. Acta*, **360**, 2734–2743.
Patil, P. S. & Dharmaprakash, S. M. (2008). *Mater. Lett.* **62**, 451–453.
Saydam, G., Aydin, H. H., Sahin, F., Kucukoglu, O., Erciyas, E., Terzioglu, E., Buyukkececi, F. & Omay, S. B. (2003). *Leuk. Res.* **27**, 57–64.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suwunwong, T., Chantrapromma, S., Karalai, C., Pakdeevanich, P. & Fun, H.-K. (2009*a*). *Acta Cryst.* E**65**, o420–o421.
Suwunwong, T., Chantrapromma, S., Pakdeevanich, P. & Fun, H.-K. (2009*b*). *Acta Cryst.* E**65**, o1575–o1576.
Svetlichny, V. Y., Merola, F., Dobretsov, G. E., Gularyan, S. K. & Syrejshchikova, T. I. (2007). *Chem. Phys. Lipids*, **145**, 13–26.

supplementary materials

Acta Cryst. (2009). E**65**, o2168-o2169 [**doi:10.1107/S1600536809031900**]

(*Z*)-3-(9-Anthryl)-1-(2-thienyl)prop-2-en-1-one

H.-K. Fun, T. Suwunwong, N. Boonnak and S. Chantrapromma

Comment

Chalcones have been studied for their chemical and biological activities for a long time. They have a wide range of applications such as in non-linear optical (NLO) materials (Patil & Dharmaprakash, 2008), fluorescent materials (Svetlichny *et al.*, 2007) and for showing various biological activities (Saydam *et al.*, 2003). The anthracene moieties are well known for their high absorption co-efficients as well as their high fluorescence yields (Oliveira *et al.*, 2007). These interesting properties has lead us to synthesize the title heteroaryl chalcone derivative, (I), which contains the donor sub-unit (anthracene) and fluorophore (thiophene) in order to study its NLO and fluorescent properties. We have previously synthesized and reported the crystal structures of chalcones and heteroaryl chalcone derivatives (Chantrapromma *et al.*, 2009; Suwunwong *et al.*, 2009*a*, *b*) which exist in the *E* configuartion. Herein, we report the crystal structure of the (I) which is in the *Z* configuration. Compound (I) crystallizes in the non-centrosymmetric orthorhombic space group $Pna2_1$ and therefore, it should exhibit second-order nonlinear optical properties. Moreover, (I) also shows interesting fluorescence properties which will be reported elsewhere.

The asymmetric unit of (I) contains two molecules, *A* and *B*, with the same configuration but with slight differences in bond lengths and angles. The molecule of (I)(Fig. 1) exists in an *Z* configuration with respect to the C15=C16 double bond [1.360 (6) Å in molecule *A* and 1.331 (6) Å in molecule *B*]; the C14–C15–C16–C17 torsion angle = -3.7 (7)° in molecule *A* [-4.0 (7)° in molecule *B*]. The anthracene unit is essentially planar with the greatest deviation of 0.089 (5) Å at atom C11A [0.086 (5)Å at atom C3B]. The total molecule is twisted as the interplanar angle between thiophene and anthracene rings is 75.07 (17)° and the mean plane through the prop-2-en-1-one unit (C15–C17/O1) makes interplanar angles of 13.1 (3) and 71.2 (3)° with the thiophene and anthracene rings, respectively [the corresponding values are 76.32 (17), 15.2 (3) and 72.3 (3)° in molecule *B*]. The bond distances are comparable with related structures (Chantrapromma *et al.*, 2009; Suwunwong *et al.*, 2009*a*, *b*).

In the crystal packing, the molecules are connected by short C···O [3.348 (5)–3.394 (5) Å], C···S [3.607 (5)–3.666 (5) Å], and S···O [2.926 (3) Å] contacts. The crystal structure is further stabilized by C—H···π interactions (Table 1) and π–π interactions with the $Cg_1\cdots Cg_4^{i}$ distance being 3.745 (3) Å (i: 1/2 + *x*, -*y* + 1/2, *z*); Cg_1 and Cg_4 are the centroids of the S1A/C18A–C21A and S1B/C18B–C21B rings, respectively.

Experimental

Compound (I) was synthesized by the condensation of anthracene-9-carbaldehyde (2 mmol, 0.41 g) with 2-acetylthiophene (2 mmol, 0.22 ml) in ethanol (30 ml) in the presence of NaOH (5 ml, 30 %). After stirring for 2 h, a yellow solid appeared which was then collected by filtration, washed with distilled water, dried and purified by repeated recrystallization using ethanol/acetone in a 1:5 ratio as solvent. Orange block-shaped crystals of (I) were obtained from hot ethanol by the slow evaporation of the solvent held at room temperature for several days; *M.p.* 391–392 K.

Refinement

All H atoms were placed in calculated positions with C—H = 0.93 Å and U_{iso} = 1.2U_{eq}(C). The highest residual electron density peak was located 0.14 Å from atom C19B and the deepest hole was located 0.48 Å from atom S1B.

Figures

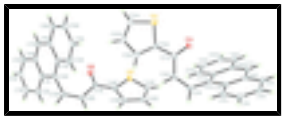


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

(*Z*)-3-(9-Anthryl)-1-(2-thienyl)prop-2-en-1-one

Crystal data

$C_{21}H_{14}OS$	D_x = 1.361 Mg m^{-3}
M_r = 314.39	Melting point = 391–392 K
Orthorhombic, *Pna*2$_1$	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 6662 reflections
a = 14.6675 (2) Å	θ = 1.1–27.5º
b = 5.5096 (1) Å	μ = 0.21 mm^{-1}
c = 37.9823 (4) Å	*T* = 100 K
V = 3069.43 (8) Å^3	Block, orange
Z = 8	0.30 × 0.12 × 0.10 mm
F_{000} = 1312	

Data collection

Bruker APEXII CCD area-detector diffractometer	6662 independent reflections
Radiation source: sealed tube	5348 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.055
T = 100 K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 1.1º
Absorption correction: multi-scan (SADABS; Bruker, 2005)	*h* = −19→18
T_{min} = 0.939, T_{max} = 0.979	*k* = −7→7
28929 measured reflections	*l* = −49→49

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.065	$w = 1/[\sigma^2(F_o^2) + (0.1247P)^2 + 2.1057P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.200$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 1.58$ e Å^{-3}
6662 reflections	$\Delta\rho_{min} = -0.82$ e Å^{-3}
391 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 3093 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.09 (15)
Secondary atom site location: difference Fourier map	

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1A	0.80198 (8)	−0.2591 (2)	0.30703 (3)	0.0253 (3)
O1A	0.7762 (2)	−0.1293 (6)	0.38105 (8)	0.0233 (7)
C1A	0.7530 (3)	0.1889 (8)	0.46578 (11)	0.0157 (8)
C2A	0.8200 (3)	0.3771 (8)	0.46638 (11)	0.0201 (9)
H2AA	0.8239	0.4840	0.4475	0.024*
C3A	0.8784 (3)	0.4033 (9)	0.49401 (12)	0.0252 (10)
H3AA	0.9204	0.5298	0.4942	0.030*
C4A	0.8750 (3)	0.2366 (9)	0.52269 (13)	0.0262 (10)
H4AA	0.9157	0.2533	0.5413	0.031*
C5A	0.8129 (3)	0.0531 (9)	0.52310 (11)	0.0227 (9)
H5AA	0.8121	−0.0544	0.5420	0.027*
C6A	0.7491 (3)	0.0228 (8)	0.49507 (11)	0.0189 (9)
C7A	0.6846 (3)	−0.1604 (8)	0.49469 (11)	0.0200 (9)
H7AA	0.6840	−0.2712	0.5132	0.024*
C8A	0.6203 (3)	−0.1866 (8)	0.46777 (11)	0.0176 (9)
C9A	0.5528 (3)	−0.3732 (9)	0.46845 (13)	0.0240 (10)
H9AA	0.5526	−0.4859	0.4867	0.029*
C10A	0.4888 (3)	−0.3872 (9)	0.44255 (12)	0.0263 (10)
H10A	0.4447	−0.5085	0.4433	0.032*
C11A	0.4888 (3)	−0.2184 (10)	0.41446 (13)	0.0264 (11)
H11A	0.4438	−0.2268	0.3973	0.032*

C12A	0.5540 (3)	−0.0448 (8)	0.41243 (11)	0.0195 (9)
H12A	0.5539	0.0609	0.3933	0.023*
C13A	0.6233 (3)	−0.0193 (8)	0.43891 (10)	0.0173 (8)
C14A	0.6914 (3)	0.1587 (8)	0.43721 (10)	0.0153 (8)
C15A	0.6981 (3)	0.3295 (8)	0.40749 (11)	0.0186 (9)
H15A	0.6851	0.4914	0.4123	0.022*
C16A	0.7215 (3)	0.2743 (8)	0.37378 (12)	0.0172 (9)
H16A	0.7196	0.3990	0.3573	0.021*
C17A	0.7496 (3)	0.0316 (8)	0.36137 (11)	0.0169 (9)
C18A	0.7486 (3)	−0.0075 (8)	0.32292 (11)	0.0156 (8)
C19A	0.7103 (3)	0.1397 (9)	0.29449 (11)	0.0176 (5)
H19A	0.6789	0.2851	0.2973	0.021*
C20A	0.7287 (3)	0.0241 (8)	0.26182 (11)	0.0176 (5)
H20A	0.7094	0.0859	0.2403	0.021*
C21A	0.7784 (3)	−0.1906 (9)	0.26501 (11)	0.0176 (5)
H21A	0.7964	−0.2851	0.2460	0.021*
S1B	0.45484 (8)	0.2512 (2)	0.28505 (3)	0.0222 (3)
O1B	0.4761 (2)	0.3719 (5)	0.20988 (8)	0.0224 (7)
C1B	0.4998 (3)	0.6562 (8)	0.12502 (11)	0.0163 (8)
C2B	0.4294 (3)	0.8335 (9)	0.12167 (11)	0.0201 (9)
H2BA	0.4236	0.9538	0.1387	0.024*
C3B	0.3701 (3)	0.8295 (8)	0.09376 (12)	0.0218 (9)
H3BA	0.3256	0.9490	0.0917	0.026*
C4B	0.3764 (3)	0.6446 (9)	0.06824 (12)	0.0225 (9)
H4BA	0.3353	0.6422	0.0496	0.027*
C5B	0.4412 (3)	0.4706 (8)	0.07042 (11)	0.0198 (9)
H5BA	0.4435	0.3491	0.0534	0.024*
C6B	0.5066 (3)	0.4707 (8)	0.09854 (10)	0.0152 (8)
C7B	0.5750 (3)	0.2983 (8)	0.10025 (11)	0.0181 (9)
H7BA	0.5776	0.1762	0.0834	0.022*
C8B	0.6408 (3)	0.3054 (8)	0.12722 (11)	0.0174 (9)
C9B	0.7149 (3)	0.1350 (8)	0.12850 (12)	0.0217 (9)
H9BA	0.7190	0.0142	0.1115	0.026*
C10B	0.7797 (3)	0.1477 (9)	0.15438 (13)	0.0268 (10)
H10B	0.8272	0.0361	0.1548	0.032*
C11B	0.7745 (3)	0.3299 (9)	0.18054 (12)	0.0230 (10)
H11B	0.8194	0.3384	0.1978	0.028*
C12B	0.7041 (3)	0.4945 (9)	0.18086 (12)	0.0198 (9)
H12B	0.7016	0.6119	0.1984	0.024*
C13B	0.6343 (3)	0.4872 (8)	0.15435 (10)	0.0157 (8)
C14B	0.5611 (3)	0.6550 (8)	0.15328 (11)	0.0150 (8)
C15B	0.5492 (3)	0.8349 (8)	0.18238 (12)	0.0181 (9)
H15B	0.5587	0.9976	0.1770	0.022*
C16B	0.5261 (3)	0.7810 (8)	0.21535 (12)	0.0169 (9)
H16B	0.5247	0.9086	0.2314	0.020*
C17B	0.5024 (3)	0.5369 (8)	0.22900 (11)	0.0163 (8)
C18B	0.5063 (3)	0.5004 (8)	0.26765 (12)	0.0180 (8)
C19B	0.5461 (3)	0.6449 (9)	0.29485 (11)	0.0186 (5)
H19B	0.5774	0.7895	0.2911	0.022*

C20B	0.5319 (3)	0.5402 (8)	0.32761 (11)	0.0186 (5)
H20B	0.5538	0.6075	0.3484	0.022*
C21B	0.4834 (3)	0.3311 (9)	0.32663 (11)	0.0186 (5)
H21B	0.4675	0.2420	0.3465	0.022*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0264 (6)	0.0243 (7)	0.0251 (6)	−0.0017 (5)	0.0031 (5)	−0.0036 (5)
O1A	0.0312 (17)	0.0193 (17)	0.0194 (14)	0.0059 (14)	0.0025 (13)	0.0055 (14)
C1A	0.0149 (19)	0.016 (2)	0.0165 (19)	0.0051 (16)	0.0006 (15)	−0.0002 (18)
C2A	0.022 (2)	0.019 (2)	0.0196 (19)	0.0016 (17)	0.0055 (16)	0.0007 (19)
C3A	0.021 (2)	0.025 (2)	0.030 (2)	−0.0011 (18)	−0.0034 (18)	−0.006 (2)
C4A	0.024 (2)	0.030 (3)	0.024 (2)	0.0028 (19)	−0.0063 (18)	−0.008 (2)
C5A	0.025 (2)	0.027 (2)	0.0158 (18)	0.0093 (19)	−0.0007 (17)	−0.0011 (19)
C6A	0.021 (2)	0.021 (2)	0.0150 (18)	0.0088 (17)	0.0036 (16)	−0.0006 (18)
C7A	0.023 (2)	0.021 (2)	0.0162 (19)	0.0065 (18)	0.0027 (16)	0.0061 (19)
C8A	0.0172 (19)	0.015 (2)	0.021 (2)	0.0031 (16)	0.0082 (16)	−0.0008 (19)
C9A	0.026 (2)	0.017 (2)	0.029 (2)	−0.0006 (17)	0.0103 (18)	0.002 (2)
C10A	0.026 (2)	0.023 (2)	0.030 (2)	−0.0091 (19)	0.0108 (18)	−0.006 (2)
C11A	0.018 (2)	0.036 (3)	0.025 (2)	0.0000 (19)	−0.0004 (17)	−0.010 (2)
C12A	0.024 (2)	0.020 (2)	0.0147 (19)	0.0038 (17)	0.0001 (16)	0.0000 (19)
C13A	0.019 (2)	0.019 (2)	0.0134 (18)	0.0019 (17)	−0.0010 (15)	−0.0045 (18)
C14A	0.023 (2)	0.013 (2)	0.0103 (18)	0.0027 (16)	0.0032 (15)	−0.0018 (17)
C15A	0.027 (2)	0.013 (2)	0.016 (2)	−0.0008 (16)	0.0002 (16)	−0.0055 (19)
C16A	0.022 (2)	0.014 (2)	0.016 (2)	0.0029 (17)	−0.0010 (18)	0.0068 (17)
C17A	0.0174 (19)	0.014 (2)	0.019 (2)	−0.0061 (16)	0.0048 (15)	0.0018 (18)
C18A	0.0151 (18)	0.016 (2)	0.0156 (17)	−0.0043 (15)	0.0055 (15)	−0.0018 (17)
C19A	0.0125 (11)	0.0246 (13)	0.0158 (11)	−0.0094 (10)	0.0020 (9)	−0.0044 (11)
C20A	0.0125 (11)	0.0246 (13)	0.0158 (11)	−0.0094 (10)	0.0020 (9)	−0.0044 (11)
C21A	0.0125 (11)	0.0246 (13)	0.0158 (11)	−0.0094 (10)	0.0020 (9)	−0.0044 (11)
S1B	0.0233 (6)	0.0204 (6)	0.0230 (6)	0.0010 (4)	0.0023 (4)	0.0046 (5)
O1B	0.0306 (17)	0.0165 (16)	0.0200 (14)	−0.0019 (13)	0.0020 (13)	−0.0011 (14)
C1B	0.019 (2)	0.014 (2)	0.0161 (19)	0.0011 (16)	0.0065 (15)	0.0049 (18)
C2B	0.022 (2)	0.022 (2)	0.0166 (19)	0.0012 (18)	0.0014 (16)	0.0026 (18)
C3B	0.019 (2)	0.020 (2)	0.026 (2)	0.0002 (17)	−0.0013 (17)	0.007 (2)
C4B	0.020 (2)	0.028 (3)	0.0185 (19)	−0.0049 (19)	−0.0027 (16)	0.001 (2)
C5B	0.024 (2)	0.021 (2)	0.0141 (18)	−0.0022 (17)	−0.0021 (16)	0.0000 (18)
C6B	0.0152 (19)	0.018 (2)	0.0122 (17)	−0.0054 (15)	0.0011 (15)	−0.0010 (17)
C7B	0.021 (2)	0.020 (2)	0.0133 (18)	−0.0024 (17)	0.0062 (16)	−0.0033 (18)
C8B	0.019 (2)	0.017 (2)	0.016 (2)	0.0006 (17)	0.0050 (16)	0.0041 (18)
C9B	0.020 (2)	0.019 (2)	0.026 (2)	0.0022 (17)	0.0098 (17)	−0.001 (2)
C10B	0.024 (2)	0.026 (3)	0.031 (2)	0.007 (2)	0.0083 (19)	0.008 (2)
C11B	0.020 (2)	0.028 (3)	0.022 (2)	0.0023 (19)	0.0002 (17)	0.009 (2)
C12B	0.020 (2)	0.021 (2)	0.019 (2)	0.0012 (17)	−0.0006 (16)	0.0012 (18)
C13B	0.0178 (19)	0.014 (2)	0.0151 (18)	0.0009 (16)	0.0059 (15)	0.0005 (18)
C14B	0.0174 (19)	0.012 (2)	0.0160 (18)	−0.0009 (16)	0.0016 (15)	0.0015 (18)
C15B	0.0163 (19)	0.014 (2)	0.024 (2)	−0.0019 (15)	0.0031 (16)	−0.002 (2)

C16B	0.023 (2)	0.014 (2)	0.014 (2)	−0.0007 (17)	0.0004 (18)	−0.0044 (17)
C17B	0.0171 (19)	0.017 (2)	0.0147 (18)	−0.0021 (16)	0.0040 (15)	−0.0011 (18)
C18B	0.0157 (18)	0.017 (2)	0.021 (2)	0.0030 (16)	0.0051 (16)	−0.0023 (18)
C19B	0.0145 (11)	0.0243 (14)	0.0171 (11)	0.0086 (10)	0.0017 (9)	0.0021 (11)
C20B	0.0145 (11)	0.0243 (14)	0.0171 (11)	0.0086 (10)	0.0017 (9)	0.0021 (11)
C21B	0.0145 (11)	0.0243 (14)	0.0171 (11)	0.0086 (10)	0.0017 (9)	0.0021 (11)

Geometric parameters (Å, °)

S1A—C21A	1.676 (4)	S1B—C21B	1.692 (5)
S1A—C18A	1.703 (4)	S1B—C18B	1.700 (5)
O1A—C17A	1.224 (5)	O1B—C17B	1.226 (5)
C1A—C14A	1.422 (6)	C1B—C14B	1.400 (6)
C1A—C2A	1.430 (6)	C1B—C2B	1.427 (6)
C1A—C6A	1.442 (6)	C1B—C6B	1.438 (6)
C2A—C3A	1.362 (6)	C2B—C3B	1.372 (6)
C2A—H2AA	0.9300	C2B—H2BA	0.9300
C3A—C4A	1.426 (7)	C3B—C4B	1.409 (7)
C3A—H3AA	0.9300	C3B—H3BA	0.9300
C4A—C5A	1.361 (7)	C4B—C5B	1.353 (6)
C4A—H4AA	0.9300	C4B—H4BA	0.9300
C5A—C6A	1.428 (6)	C5B—C6B	1.435 (5)
C5A—H5AA	0.9300	C5B—H5BA	0.9300
C6A—C7A	1.383 (6)	C6B—C7B	1.383 (6)
C7A—C8A	1.398 (6)	C7B—C8B	1.408 (6)
C7A—H7AA	0.9300	C7B—H7BA	0.9300
C8A—C9A	1.427 (6)	C8B—C9B	1.437 (6)
C8A—C13A	1.433 (6)	C8B—C13B	1.440 (6)
C9A—C10A	1.362 (7)	C9B—C10B	1.368 (7)
C9A—H9AA	0.9300	C9B—H9BA	0.9300
C10A—C11A	1.415 (7)	C10B—C11B	1.415 (7)
C10A—H10A	0.9300	C10B—H10B	0.9300
C11A—C12A	1.354 (7)	C11B—C12B	1.374 (6)
C11A—H11A	0.9300	C11B—H11B	0.9300
C12A—C13A	1.437 (5)	C12B—C13B	1.436 (5)
C12A—H12A	0.9300	C12B—H12B	0.9300
C13A—C14A	1.401 (6)	C13B—C14B	1.417 (6)
C14A—C15A	1.473 (6)	C14B—C15B	1.495 (6)
C15A—C16A	1.360 (6)	C15B—C16B	1.331 (6)
C15A—H15A	0.9300	C15B—H15B	0.9300
C16A—C17A	1.477 (6)	C16B—C17B	1.483 (6)
C16A—H16A	0.9300	C16B—H16B	0.9300
C17A—C18A	1.476 (5)	C17B—C18B	1.483 (6)
C18A—C19A	1.463 (6)	C18B—C19B	1.430 (6)
C19A—C20A	1.421 (6)	C19B—C20B	1.387 (6)
C19A—H19A	0.9300	C19B—H19B	0.9300
C20A—C21A	1.395 (6)	C20B—C21B	1.355 (7)
C20A—H20A	0.9300	C20B—H20B	0.9300
C21A—H21A	0.9300	C21B—H21B	0.9300

C21A—S1A—C18A	93.4 (2)	C21B—S1B—C18B	92.5 (2)
C14A—C1A—C2A	122.3 (4)	C14B—C1B—C2B	122.4 (4)
C14A—C1A—C6A	119.3 (4)	C14B—C1B—C6B	119.2 (4)
C2A—C1A—C6A	118.4 (4)	C2B—C1B—C6B	118.3 (4)
C3A—C2A—C1A	121.5 (4)	C3B—C2B—C1B	121.1 (4)
C3A—C2A—H2AA	119.3	C3B—C2B—H2BA	119.4
C1A—C2A—H2AA	119.3	C1B—C2B—H2BA	119.4
C2A—C3A—C4A	119.9 (4)	C2B—C3B—C4B	120.1 (4)
C2A—C3A—H3AA	120.1	C2B—C3B—H3BA	120.0
C4A—C3A—H3AA	120.1	C4B—C3B—H3BA	120.0
C5A—C4A—C3A	120.7 (4)	C5B—C4B—C3B	121.1 (4)
C5A—C4A—H4AA	119.6	C5B—C4B—H4BA	119.4
C3A—C4A—H4AA	119.6	C3B—C4B—H4BA	119.4
C4A—C5A—C6A	121.2 (4)	C4B—C5B—C6B	121.0 (4)
C4A—C5A—H5AA	119.4	C4B—C5B—H5BA	119.5
C6A—C5A—H5AA	119.4	C6B—C5B—H5BA	119.5
C7A—C6A—C5A	122.8 (4)	C7B—C6B—C5B	121.3 (4)
C7A—C6A—C1A	118.8 (4)	C7B—C6B—C1B	120.4 (4)
C5A—C6A—C1A	118.4 (4)	C5B—C6B—C1B	118.3 (4)
C6A—C7A—C8A	122.9 (4)	C6B—C7B—C8B	120.8 (4)
C6A—C7A—H7AA	118.5	C6B—C7B—H7BA	119.6
C8A—C7A—H7AA	118.5	C8B—C7B—H7BA	119.6
C7A—C8A—C9A	121.9 (4)	C7B—C8B—C9B	121.7 (4)
C7A—C8A—C13A	118.2 (4)	C7B—C8B—C13B	119.6 (4)
C9A—C8A—C13A	119.9 (4)	C9B—C8B—C13B	118.7 (4)
C10A—C9A—C8A	120.4 (4)	C10B—C9B—C8B	121.1 (4)
C10A—C9A—H9AA	119.8	C10B—C9B—H9BA	119.5
C8A—C9A—H9AA	119.8	C8B—C9B—H9BA	119.5
C9A—C10A—C11A	120.5 (4)	C9B—C10B—C11B	120.2 (4)
C9A—C10A—H10A	119.8	C9B—C10B—H10B	119.9
C11A—C10A—H10A	119.8	C11B—C10B—H10B	119.9
C12A—C11A—C10A	120.5 (4)	C12B—C11B—C10B	121.0 (4)
C12A—C11A—H11A	119.8	C12B—C11B—H11B	119.5
C10A—C11A—H11A	119.8	C10B—C11B—H11B	119.5
C11A—C12A—C13A	121.9 (4)	C11B—C12B—C13B	120.7 (4)
C11A—C12A—H12A	119.0	C11B—C12B—H12B	119.6
C13A—C12A—H12A	119.0	C13B—C12B—H12B	119.6
C14A—C13A—C8A	120.5 (4)	C14B—C13B—C12B	122.8 (4)
C14A—C13A—C12A	122.7 (4)	C14B—C13B—C8B	118.9 (4)
C8A—C13A—C12A	116.8 (4)	C12B—C13B—C8B	118.3 (4)
C13A—C14A—C1A	120.0 (4)	C1B—C14B—C13B	120.8 (4)
C13A—C14A—C15A	122.0 (4)	C1B—C14B—C15B	119.2 (4)
C1A—C14A—C15A	117.9 (4)	C13B—C14B—C15B	120.0 (4)
C16A—C15A—C14A	126.6 (4)	C16B—C15B—C14B	125.3 (4)
C16A—C15A—H15A	116.7	C16B—C15B—H15B	117.4
C14A—C15A—H15A	116.7	C14B—C15B—H15B	117.4
C15A—C16A—C17A	125.0 (4)	C15B—C16B—C17B	126.2 (4)
C15A—C16A—H16A	117.5	C15B—C16B—H16B	116.9
C17A—C16A—H16A	117.5	C17B—C16B—H16B	116.9

O1A—C17A—C18A	120.1 (4)	O1B—C17B—C18B	119.9 (4)
O1A—C17A—C16A	123.4 (4)	O1B—C17B—C16B	122.6 (4)
C18A—C17A—C16A	116.4 (4)	C18B—C17B—C16B	117.4 (4)
C19A—C18A—C17A	130.8 (4)	C19B—C18B—C17B	131.0 (4)
C19A—C18A—S1A	111.5 (3)	C19B—C18B—S1B	110.5 (3)
C17A—C18A—S1A	117.7 (3)	C17B—C18B—S1B	118.5 (3)
C20A—C19A—C18A	108.9 (4)	C20B—C19B—C18B	110.8 (4)
C20A—C19A—H19A	125.6	C20B—C19B—H19B	124.6
C18A—C19A—H19A	125.6	C18B—C19B—H19B	124.6
C21A—C20A—C19A	113.8 (4)	C21B—C20B—C19B	114.1 (4)
C21A—C20A—H20A	123.1	C21B—C20B—H20B	123.0
C19A—C20A—H20A	123.1	C19B—C20B—H20B	123.0
C20A—C21A—S1A	112.4 (3)	C20B—C21B—S1B	112.1 (3)
C20A—C21A—H21A	123.8	C20B—C21B—H21B	123.9
S1A—C21A—H21A	123.8	S1B—C21B—H21B	123.9
C14A—C1A—C2A—C3A	179.9 (4)	C14B—C1B—C2B—C3B	−179.7 (4)
C6A—C1A—C2A—C3A	−0.9 (6)	C6B—C1B—C2B—C3B	−0.7 (6)
C1A—C2A—C3A—C4A	1.8 (7)	C1B—C2B—C3B—C4B	1.7 (7)
C2A—C3A—C4A—C5A	−1.1 (7)	C2B—C3B—C4B—C5B	−0.9 (7)
C3A—C4A—C5A—C6A	−0.4 (7)	C3B—C4B—C5B—C6B	−0.9 (7)
C4A—C5A—C6A—C7A	−179.6 (4)	C4B—C5B—C6B—C7B	−177.7 (4)
C4A—C5A—C6A—C1A	1.2 (6)	C4B—C5B—C6B—C1B	1.9 (6)
C14A—C1A—C6A—C7A	−0.7 (6)	C14B—C1B—C6B—C7B	−2.4 (6)
C2A—C1A—C6A—C7A	−179.8 (4)	C2B—C1B—C6B—C7B	178.5 (4)
C14A—C1A—C6A—C5A	178.6 (4)	C14B—C1B—C6B—C5B	178.0 (4)
C2A—C1A—C6A—C5A	−0.6 (6)	C2B—C1B—C6B—C5B	−1.1 (6)
C5A—C6A—C7A—C8A	178.2 (4)	C5B—C6B—C7B—C8B	177.6 (4)
C1A—C6A—C7A—C8A	−2.6 (6)	C1B—C6B—C7B—C8B	−2.0 (6)
C6A—C7A—C8A—C9A	−178.3 (4)	C6B—C7B—C8B—C9B	−177.0 (4)
C6A—C7A—C8A—C13A	1.7 (6)	C6B—C7B—C8B—C13B	3.0 (6)
C7A—C8A—C9A—C10A	177.2 (4)	C7B—C8B—C9B—C10B	178.4 (4)
C13A—C8A—C9A—C10A	−2.7 (6)	C13B—C8B—C9B—C10B	−1.6 (6)
C8A—C9A—C10A—C11A	0.6 (7)	C8B—C9B—C10B—C11B	0.1 (7)
C9A—C10A—C11A—C12A	1.8 (7)	C9B—C10B—C11B—C12B	1.0 (7)
C10A—C11A—C12A—C13A	−2.1 (7)	C10B—C11B—C12B—C13B	−0.6 (7)
C7A—C8A—C13A—C14A	2.6 (6)	C11B—C12B—C13B—C14B	−179.1 (4)
C9A—C8A—C13A—C14A	−177.4 (4)	C11B—C12B—C13B—C8B	−0.8 (6)
C7A—C8A—C13A—C12A	−177.5 (4)	C7B—C8B—C13B—C14B	0.3 (6)
C9A—C8A—C13A—C12A	2.4 (6)	C9B—C8B—C13B—C14B	−179.7 (4)
C11A—C12A—C13A—C14A	179.8 (4)	C7B—C8B—C13B—C12B	−178.1 (4)
C11A—C12A—C13A—C8A	−0.1 (6)	C9B—C8B—C13B—C12B	1.9 (6)
C8A—C13A—C14A—C1A	−5.9 (6)	C2B—C1B—C14B—C13B	−175.3 (4)
C12A—C13A—C14A—C1A	174.3 (4)	C6B—C1B—C14B—C13B	5.7 (6)
C8A—C13A—C14A—C15A	178.3 (4)	C2B—C1B—C14B—C15B	4.2 (6)
C12A—C13A—C14A—C15A	−1.5 (6)	C6B—C1B—C14B—C15B	−174.8 (4)
C2A—C1A—C14A—C13A	−176.0 (4)	C12B—C13B—C14B—C1B	173.6 (4)
C6A—C1A—C14A—C13A	4.8 (6)	C8B—C13B—C14B—C1B	−4.7 (6)
C2A—C1A—C14A—C15A	0.0 (6)	C12B—C13B—C14B—C15B	−5.9 (6)
C6A—C1A—C14A—C15A	−179.2 (4)	C8B—C13B—C14B—C15B	175.9 (4)

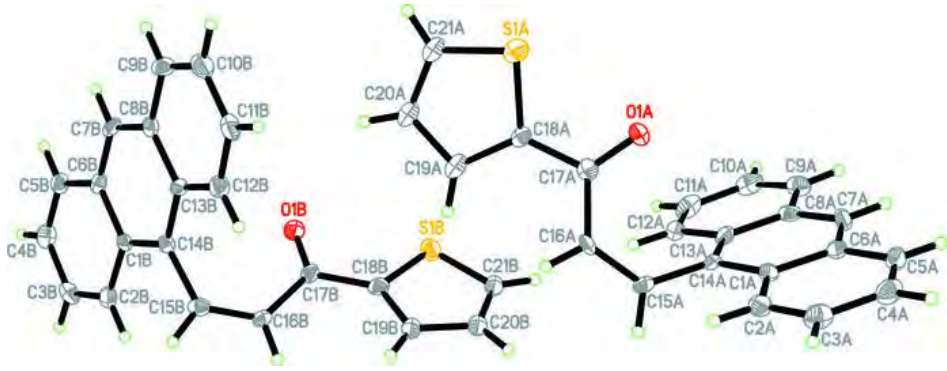
C13A—C14A—C15A—C16A	−68.5 (6)	C1B—C14B—C15B—C16B	112.9 (5)
C1A—C14A—C15A—C16A	115.5 (5)	C13B—C14B—C15B—C16B	−67.7 (6)
C14A—C15A—C16A—C17A	−3.7 (7)	C14B—C15B—C16B—C17B	−4.0 (7)
C15A—C16A—C17A—O1A	−18.5 (7)	C15B—C16B—C17B—O1B	−21.8 (7)
C15A—C16A—C17A—C18A	164.3 (4)	C15B—C16B—C17B—C18B	161.7 (4)
O1A—C17A—C18A—C19A	171.3 (4)	O1B—C17B—C18B—C19B	169.3 (4)
C16A—C17A—C18A—C19A	−11.4 (6)	C16B—C17B—C18B—C19B	−14.0 (7)
O1A—C17A—C18A—S1A	−10.4 (5)	O1B—C17B—C18B—S1B	−11.8 (5)
C16A—C17A—C18A—S1A	166.9 (3)	C16B—C17B—C18B—S1B	164.8 (3)
C21A—S1A—C18A—C19A	−0.1 (3)	C21B—S1B—C18B—C19B	0.3 (3)
C21A—S1A—C18A—C17A	−178.7 (3)	C21B—S1B—C18B—C17B	−178.8 (3)
C17A—C18A—C19A—C20A	179.1 (4)	C17B—C18B—C19B—C20B	179.2 (4)
S1A—C18A—C19A—C20A	0.7 (4)	S1B—C18B—C19B—C20B	0.3 (4)
C18A—C19A—C20A—C21A	−1.1 (5)	C18B—C19B—C20B—C21B	−1.0 (5)
C19A—C20A—C21A—S1A	1.0 (4)	C19B—C20B—C21B—S1B	1.2 (5)
C18A—S1A—C21A—C20A	−0.5 (3)	C18B—S1B—C21B—C20B	−0.8 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3A—H3AA···Cg3^{i}	0.93	2.99	3.679 (5)	132
C10A—H10A···Cg2^{ii}	0.93	2.95	3.694 (5)	138
C10B—H10B···Cg5^{i}	0.93	2.93	3.594 (5)	129
C15A—H15A···Cg3^{iii}	0.93	2.76	3.550 (5)	143
C15B—H15B···Cg6^{iii}	0.93	2.94	3.689 (5)	139
C19A—H19A···Cg4	0.93	2.72	3.486 (5)	140
C19B—H19B···Cg1^{iii}	0.93	2.72	3.458 (5)	137

Symmetry codes: (i) *x*+1/2, −*y*+1/2, *z*; (ii) *x*−1/2, −*y*−1/2, *z*; (iii) *x*, *y*+1, *z*.

Fig. 1

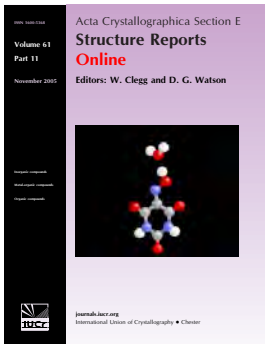


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*Z*)-3-(9-Anthryl)-1-(4-methoxyphenyl)prop-2-en-1-one

Suchada Chantrapromma, Jirapa Horkaew, Thitipone Suwunwong and Hoong-Kun Fun

Acta Cryst. (2009). E**65**, o2673–o2674



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-3-(9-Anthryl)-1-(4-methoxyphenyl)-prop-2-en-1-one[1]

Suchada Chantrapromma,[a]*§ Jirapa Horkaew,[a] Thitipone Suwunwong[a] and Hoong-Kun Fun[b]¶

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

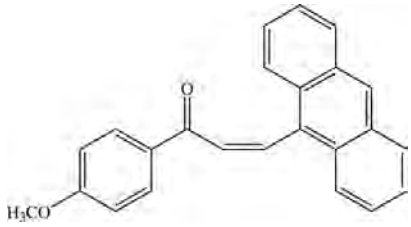
Received 19 September 2009; accepted 24 September 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.087; data-to-parameter ratio = 7.3.

The title chalcone derivative, $C_{24}H_{18}O_2$, which consists of the substituted 4-methoxyphenyl and anthracene rings bridged by the prop-2-en-1-one unit, exists in a *cis* configuration. The molecule is twisted, the interplanar angle between the benzene and anthracene rings being 69.50 (10)°. The methoxy group is coplanar with the attached benzene ring [C—O—C—C angle = 2.9 (3)°]. In the crystal structure, molecules are linked into chains along the *a* axis by a weak C—H···O(enone) interaction. The chains are stacked along the *c* axis. A C—H···π interaction involving the benzene ring is observed.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related structures, see: Fun *et al.* (2009); Suwunwong *et al.* (2009). For background to and applications of chalcones, see: Patil & Dharmaprakash (2008); Saydam *et al.* (2003); Svetlichny *et al.* (2007). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{24}H_{18}O_2$
M_r = 338.38
Monoclinic, *Cc*
a = 5.5018 (2) Å
b = 19.9215 (8) Å
c = 16.0500 (7) Å
β = 95.072 (2)°
V = 1752.26 (12) Å^3
Z = 4
Mo *K*α radiation
μ = 0.08 mm^{-1}
T = 293 K
0.54 × 0.27 × 0.09 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.958, T_{max} = 0.993
7966 measured reflections
1721 independent reflections
1545 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.087
S = 1.07
1721 reflections
236 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O1^{i}	0.93	2.47	3.290 (3)	147
C24—H24*A*···O1^{ii}	0.96	2.59	3.176 (4)	120
C17—H17*A*···*Cg*1^{iii}	0.93	2.89	3.694 (3)	145

Symmetry codes: (i) $x-1, y, z$; (ii) $x-\frac{1}{2}, y-\frac{1}{2}, z$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$. *Cg*1 is the centroid of the C1–C6 ring.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

Financial support from the Thailand Research Fund (TRF) and Prince of Songkla University are gratefully acknowledged. The authors also thank Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2659).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Fun, H.-K., Suwunwong, T., Boonnak, N. & Chantrapromma, S. (2009). *Acta Cryst.* E**65**, o2168–o2169.
Patil, P. S. & Dharmaprakash, S. M. (2008). *Mater. Lett.* **62**, 451–453.
Saydam, G., Aydin, H. H., Sahin, F., Kucukoglu, O., Erciyas, E., Terzioglu, E., Buyukkececi, F. & Omay, S. B. (2003). *Leuk. Res.* **27**, 57–64.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

[1] This paper is dedicated to the late His Royal Highness Prince Mahidol of Songkla for his contributions to the development of medical education in Thailand on the occasion of Mahidol Day which falls on the 24th September.
§ Thomson Reuters ResearcherID: A-5085-2009.
¶ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Suwunwong, T., Chantrapromma, S., Karalai, C., Pakdeevanich, P. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o420–o421.

Svetlichny, V. Y., Merola, F., Dobretsov, G. E., Gularyan, S. K. & Syrejshchikova, T. I. (2007). *Chem. Phys. Lipids*, **145**, 13–26.

supplementary materials

Acta Cryst. (2009). E**65**, o2673-o2674 [**doi:10.1107/S1600536809038665**]

(*Z*)-3-(9-Anthryl)-1-(4-methoxyphenyl)prop-2-en-1-one

S. Chantrapromma, J. Horkaew, T. Suwunwong and H.-K. Fun

Comment

Chalcones have been studied for their wide range of applications such as non-linear optical (Patil & Dharmaprakash, 2008) and fluorescent properties (Svetlichny *et al.*, 2007) and biological activities (Saydam *et al.*, 2003). We have previously reported crystal structures of chalcone derivatives containing the anthracene moiety which exist in both the *E* (Suwunwong *et al.*, 2009) and *Z* configurations (Fun *et al.*, 2009). The title compound was synthesized to study its fluorescent properties in addition to its antibacterial activity. The title compound shows interesting fluorescence properties which will be reported elsewhere. The crystal structure of the title compound was studied in order to elucidate its conformation which may affect the fluorescence properties.

The molecule of the title chalcone derivative, $C_{24}H_{18}O_2$, (Fig. 1) exists in a *Z* configuration with respect to the C8=C9 ethenyl bond with the torsion angle C7–C8–C9–C10 being 3.6 (5)°. The anthracene ring system (C10–C23) is essentially planar with the root mean deviation of 0.050 (3) Å. The molecule is twisted as shown by the interplanar angle between the 4-methoxyphenyl and anthracene rings being 69.50 (10)°. The substituted methoxy group is coplanar with the phenyl ring with the torsion angle C24–O2–C3–C2 being 2.9 (3)°. The prop-2-en-1-one unit (C7—C9/O1) is twisted with the torsion angle O1–C7–C8–C9 of 44.5 (4)°. The orientation of the prop-2-en-1-one unit with respect to the 4-methoxyphenyl and anthracene rings is indicated by the torsion angles C1–C6–C7–C8 = 15.6 (4) and C7–C8–C9–C10 = 3.6 (5) °. The bond distances (Allen *et al.*, 1987) and angles are normal and comparable to those found in closely related structures (Fun *et al.*, 2009; Suwunwong *et al.*, 2009).

In the crystal packing, the molecules are linked into chains along the *a* axis through the enone unit by a weak C8—H8A···O1 interaction (Fig. 2, Table 1). These chains are stacked along the *c* axis involving a C—H···π interaction (Table 1); Cg_1 is the centroid of the C1–C6 ring.

Experimental

The title compound was synthesized by condensation of anthracene-9-carbaldehyde (0.41 g, 2 mmol) with 4-methoxyacetophenone (0.30 g, 2 mmol) in ethanol (30 ml) in the presence of 30% aqueous NaOH (5 ml) at room temperature. After stirring for 3 hr, a yellow solid appeared and was then collected by filtration, washed with acetone and dried in air. Yellow block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystalized from ethanol by the slow evaporation of the solvent at room temperature after several days, Mp. 440–441 K.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93 Å, U_{iso} = 1.2U_{eq}(C) for aromatic and CH and C—H = 0.96 Å, U_{iso} = 1.5U_{eq}(C) for CH_3 atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.73 Å from C18 and the deepest hole is located at 1.39 Å from C17. A total of 1128

Friedel pairs were merged before final refinement as there is no large anomalous dispersion for the determination of the absolute structure.

Figures

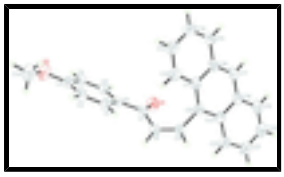


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

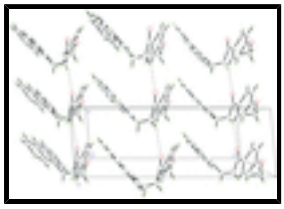


Fig. 2. The crystal packing of the title compound viewed along the *b* axis, showing chains running along the *a* axis. Weak C—H···O interactions are shown as dashed lines.

(*Z*)-3-(9-Anthryl)-1-(4-methoxyphenyl)prop-2-en-1-one

Crystal data

$C_{24}H_{18}O_2$	F_{000} = 712
M_r = 338.38	D_x = 1.283 Mg m^{-3}
Monoclinic, *Cc*	Melting point = 440–441 K
Hall symbol: C -2yc	Mo *K*α radiation, λ = 0.71073 Å
a = 5.5018 (2) Å	Cell parameters from 1721 reflections
b = 19.9215 (8) Å	θ = 2.0–26.0º
c = 16.0500 (7) Å	μ = 0.08 mm^{-1}
β = 95.072 (2)º	*T* = 293 K
V = 1752.26 (12) Å^3	Plate, yellow
Z = 4	0.54 × 0.27 × 0.09 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	1721 independent reflections
Radiation source: fine-focus sealed tube	1545 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.023
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 26.0º
T = 293 K	θ_{min} = 2.0º
ω scans	*h* = −6→6
Absorption correction: multi-scan (SADABS; Bruker, 2005)	*k* = −24→23
T_{min} = 0.958, T_{max} = 0.993	*l* = −19→19
7966 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.087$	$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.4575P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
1721 reflections	$\Delta\rho_{max} = 0.14$ e Å^{-3}
236 parameters	$\Delta\rho_{min} = -0.14$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.7379 (3)	0.26049 (10)	0.44324 (14)	0.0584 (5)
O2	0.5958 (4)	−0.04663 (10)	0.52663 (15)	0.0663 (6)
C1	0.3633 (5)	0.11209 (14)	0.43620 (17)	0.0469 (7)
H1A	0.2261	0.1294	0.4055	0.056*
C2	0.3691 (5)	0.04454 (15)	0.45676 (18)	0.0514 (7)
H2A	0.2382	0.0169	0.4394	0.062*
C3	0.5709 (5)	0.01859 (14)	0.50326 (17)	0.0469 (6)
C4	0.7641 (5)	0.06087 (14)	0.52870 (18)	0.0498 (7)
H4A	0.8992	0.0437	0.5607	0.060*
C5	0.7586 (4)	0.12742 (14)	0.50729 (17)	0.0448 (6)
H5A	0.8907	0.1548	0.5243	0.054*
C6	0.5565 (4)	0.15470 (13)	0.46010 (15)	0.0394 (6)
C7	0.5525 (4)	0.22645 (13)	0.43565 (16)	0.0413 (6)
C8	0.3147 (4)	0.25664 (13)	0.40406 (18)	0.0454 (6)
H8A	0.1795	0.2456	0.4322	0.054*
C9	0.2783 (5)	0.29786 (13)	0.33936 (17)	0.0451 (6)
H9A	0.1214	0.3150	0.3286	0.054*

C10	0.4614 (4)	0.31959 (13)	0.28220 (16)	0.0411 (6)
C11	0.5862 (5)	0.27224 (13)	0.23652 (16)	0.0428 (6)
C12	0.5348 (6)	0.20192 (15)	0.23660 (18)	0.0527 (7)
H12A	0.4126	0.1859	0.2678	0.063*
C13	0.6601 (6)	0.15829 (16)	0.1923 (2)	0.0634 (9)
H13A	0.6205	0.1129	0.1927	0.076*
C14	0.8495 (7)	0.18016 (18)	0.1455 (2)	0.0670 (9)
H14A	0.9371	0.1492	0.1167	0.080*
C15	0.9040 (6)	0.24607 (17)	0.14224 (18)	0.0580 (8)
H15A	1.0296	0.2601	0.1112	0.070*
C16	0.7721 (5)	0.29480 (14)	0.18577 (15)	0.0460 (6)
C17	0.8194 (5)	0.36309 (15)	0.17973 (16)	0.0491 (7)
H17A	0.9427	0.3773	0.1478	0.059*
C18	0.6874 (5)	0.41081 (14)	0.22015 (16)	0.0463 (7)
C19	0.7276 (6)	0.48094 (15)	0.21182 (19)	0.0559 (8)
H19A	0.8496	0.4957	0.1796	0.067*
C20	0.5933 (6)	0.52656 (16)	0.2495 (2)	0.0622 (8)
H20A	0.6211	0.5721	0.2422	0.075*
C21	0.4106 (6)	0.50528 (17)	0.2999 (2)	0.0616 (8)
H21A	0.3169	0.5370	0.3251	0.074*
C22	0.3704 (5)	0.43918 (16)	0.31209 (18)	0.0529 (7)
H22A	0.2533	0.4263	0.3473	0.063*
C23	0.5028 (5)	0.38878 (13)	0.27229 (16)	0.0426 (6)
C24	0.4045 (7)	−0.09228 (16)	0.4985 (3)	0.0761 (10)
H24A	0.4477	−0.1368	0.5173	0.114*
H24B	0.3820	−0.0916	0.4385	0.114*
H24C	0.2557	−0.0790	0.5209	0.114*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0426 (10)	0.0571 (12)	0.0750 (14)	−0.0105 (10)	0.0021 (9)	0.0098 (11)
O2	0.0777 (15)	0.0442 (12)	0.0741 (15)	−0.0021 (11)	−0.0085 (12)	0.0055 (11)
C1	0.0362 (13)	0.0538 (17)	0.0499 (15)	−0.0038 (12)	−0.0007 (11)	0.0081 (13)
C2	0.0466 (15)	0.0532 (17)	0.0532 (16)	−0.0111 (13)	−0.0017 (13)	0.0015 (13)
C3	0.0513 (15)	0.0464 (16)	0.0435 (15)	0.0022 (12)	0.0071 (12)	0.0015 (12)
C4	0.0450 (15)	0.0519 (17)	0.0514 (16)	0.0072 (13)	−0.0026 (12)	0.0011 (13)
C5	0.0340 (13)	0.0535 (16)	0.0471 (15)	−0.0037 (12)	0.0041 (11)	−0.0032 (12)
C6	0.0337 (12)	0.0493 (16)	0.0365 (13)	−0.0027 (11)	0.0101 (10)	0.0015 (11)
C7	0.0372 (13)	0.0498 (15)	0.0387 (13)	−0.0049 (12)	0.0132 (11)	0.0020 (12)
C8	0.0357 (13)	0.0479 (15)	0.0543 (16)	−0.0020 (12)	0.0133 (11)	0.0064 (13)
C9	0.0363 (13)	0.0449 (16)	0.0542 (15)	0.0019 (11)	0.0042 (11)	0.0035 (13)
C10	0.0381 (13)	0.0470 (16)	0.0377 (13)	−0.0006 (11)	−0.0003 (10)	0.0045 (11)
C11	0.0459 (14)	0.0439 (15)	0.0376 (13)	0.0007 (11)	−0.0018 (11)	0.0044 (12)
C12	0.0589 (17)	0.0486 (16)	0.0505 (16)	−0.0023 (14)	0.0040 (14)	0.0033 (14)
C13	0.084 (2)	0.0470 (17)	0.0592 (18)	0.0052 (16)	0.0048 (17)	−0.0025 (15)
C14	0.084 (2)	0.063 (2)	0.0557 (18)	0.0208 (18)	0.0146 (17)	−0.0049 (16)
C15	0.0646 (19)	0.068 (2)	0.0430 (16)	0.0071 (16)	0.0165 (14)	0.0037 (14)

C16	0.0495 (16)	0.0547 (17)	0.0335 (13)	0.0020 (13)	0.0015 (11)	0.0041 (12)
C17	0.0483 (15)	0.0615 (19)	0.0384 (14)	−0.0058 (13)	0.0083 (12)	0.0093 (13)
C18	0.0499 (15)	0.0505 (17)	0.0370 (13)	−0.0056 (13)	−0.0043 (12)	0.0073 (12)
C19	0.0635 (19)	0.0537 (19)	0.0496 (16)	−0.0158 (15)	0.0000 (14)	0.0084 (14)
C20	0.079 (2)	0.0415 (17)	0.063 (2)	−0.0042 (16)	−0.0104 (17)	0.0035 (15)
C21	0.070 (2)	0.0494 (19)	0.0640 (19)	0.0076 (15)	−0.0005 (16)	−0.0029 (15)
C22	0.0531 (16)	0.0531 (19)	0.0520 (17)	0.0021 (14)	0.0027 (13)	0.0015 (14)
C23	0.0438 (14)	0.0436 (15)	0.0390 (13)	−0.0005 (12)	−0.0037 (11)	0.0029 (11)
C24	0.093 (3)	0.052 (2)	0.082 (2)	−0.0170 (18)	−0.003 (2)	0.0025 (18)

Geometric parameters (Å, °)

O1—C7	1.222 (3)	C12—H12A	0.9300
O2—C3	1.356 (3)	C13—C14	1.407 (5)
O2—C24	1.433 (4)	C13—H13A	0.9300
C1—C2	1.385 (4)	C14—C15	1.349 (5)
C1—C6	1.387 (4)	C14—H14A	0.9300
C1—H1A	0.9300	C15—C16	1.431 (4)
C2—C3	1.382 (4)	C15—H15A	0.9300
C2—H2A	0.9300	C16—C17	1.390 (4)
C3—C4	1.389 (4)	C17—C18	1.391 (4)
C4—C5	1.369 (4)	C17—H17A	0.9300
C4—H4A	0.9300	C18—C19	1.423 (4)
C5—C6	1.399 (4)	C18—C23	1.440 (4)
C5—H5A	0.9300	C19—C20	1.348 (5)
C6—C7	1.482 (4)	C19—H19A	0.9300
C7—C8	1.488 (4)	C20—C21	1.411 (5)
C8—C9	1.325 (4)	C20—H20A	0.9300
C8—H8A	0.9300	C21—C22	1.352 (5)
C9—C10	1.486 (4)	C21—H21A	0.9300
C9—H9A	0.9300	C22—C23	1.425 (4)
C10—C23	1.408 (4)	C22—H22A	0.9300
C10—C11	1.410 (4)	C24—H24A	0.9600
C11—C12	1.429 (4)	C24—H24B	0.9600
C11—C16	1.435 (4)	C24—H24C	0.9600
C12—C13	1.350 (4)		
C3—O2—C24	117.9 (2)	C12—C13—H13A	119.4
C2—C1—C6	121.8 (3)	C14—C13—H13A	119.4
C2—C1—H1A	119.1	C15—C14—C13	120.0 (3)
C6—C1—H1A	119.1	C15—C14—H14A	120.0
C3—C2—C1	119.5 (3)	C13—C14—H14A	120.0
C3—C2—H2A	120.2	C14—C15—C16	121.2 (3)
C1—C2—H2A	120.2	C14—C15—H15A	119.4
O2—C3—C2	124.4 (3)	C16—C15—H15A	119.4
O2—C3—C4	116.4 (2)	C17—C16—C15	121.6 (3)
C2—C3—C4	119.3 (3)	C17—C16—C11	119.5 (2)
C5—C4—C3	120.9 (2)	C15—C16—C11	118.9 (3)
C5—C4—H4A	119.5	C16—C17—C18	121.9 (2)
C3—C4—H4A	119.5	C16—C17—H17A	119.1

C4—C5—C6	120.7 (2)	C18—C17—H17A	119.1
C4—C5—H5A	119.6	C17—C18—C19	122.4 (3)
C6—C5—H5A	119.6	C17—C18—C23	119.1 (2)
C1—C6—C5	117.7 (2)	C19—C18—C23	118.5 (3)
C1—C6—C7	121.6 (2)	C20—C19—C18	121.6 (3)
C5—C6—C7	120.7 (2)	C20—C19—H19A	119.2
O1—C7—C6	121.1 (2)	C18—C19—H19A	119.2
O1—C7—C8	120.7 (2)	C19—C20—C21	120.1 (3)
C6—C7—C8	118.2 (2)	C19—C20—H20A	119.9
C9—C8—C7	125.8 (2)	C21—C20—H20A	119.9
C9—C8—H8A	117.1	C22—C21—C20	120.6 (3)
C7—C8—H8A	117.1	C22—C21—H21A	119.7
C8—C9—C10	126.9 (2)	C20—C21—H21A	119.7
C8—C9—H9A	116.5	C21—C22—C23	121.7 (3)
C10—C9—H9A	116.5	C21—C22—H22A	119.2
C23—C10—C11	120.3 (2)	C23—C22—H22A	119.2
C23—C10—C9	118.7 (2)	C10—C23—C22	123.0 (2)
C11—C10—C9	120.9 (2)	C10—C23—C18	119.6 (2)
C10—C11—C12	123.4 (2)	C22—C23—C18	117.4 (2)
C10—C11—C16	119.3 (2)	O2—C24—H24A	109.5
C12—C11—C16	117.3 (2)	O2—C24—H24B	109.5
C13—C12—C11	121.4 (3)	H24A—C24—H24B	109.5
C13—C12—H12A	119.3	O2—C24—H24C	109.5
C11—C12—H12A	119.3	H24A—C24—H24C	109.5
C12—C13—C14	121.2 (3)	H24B—C24—H24C	109.5
C6—C1—C2—C3	0.8 (4)	C12—C13—C14—C15	−2.0 (5)
C24—O2—C3—C2	2.6 (4)	C13—C14—C15—C16	0.0 (5)
C24—O2—C3—C4	−177.4 (3)	C14—C15—C16—C17	−177.0 (3)
C1—C2—C3—O2	−179.8 (3)	C14—C15—C16—C11	2.8 (4)
C1—C2—C3—C4	0.1 (4)	C10—C11—C16—C17	−2.6 (4)
O2—C3—C4—C5	179.0 (3)	C12—C11—C16—C17	176.3 (2)
C2—C3—C4—C5	−1.0 (4)	C10—C11—C16—C15	177.6 (2)
C3—C4—C5—C6	0.8 (4)	C12—C11—C16—C15	−3.4 (3)
C2—C1—C6—C5	−1.0 (4)	C15—C16—C17—C18	178.3 (2)
C2—C1—C6—C7	178.0 (2)	C11—C16—C17—C18	−1.5 (4)
C4—C5—C6—C1	0.1 (4)	C16—C17—C18—C19	−177.5 (3)
C4—C5—C6—C7	−178.9 (2)	C16—C17—C18—C23	2.4 (4)
C1—C6—C7—O1	−166.3 (3)	C17—C18—C19—C20	178.0 (3)
C5—C6—C7—O1	12.6 (4)	C23—C18—C19—C20	−1.8 (4)
C1—C6—C7—C8	15.6 (4)	C18—C19—C20—C21	1.2 (5)
C5—C6—C7—C8	−165.4 (2)	C19—C20—C21—C22	0.9 (5)
O1—C7—C8—C9	44.5 (4)	C20—C21—C22—C23	−2.5 (5)
C6—C7—C8—C9	−137.5 (3)	C11—C10—C23—C22	175.5 (2)
C7—C8—C9—C10	3.6 (5)	C9—C10—C23—C22	−2.5 (4)
C8—C9—C10—C23	−124.0 (3)	C11—C10—C23—C18	−4.8 (3)
C8—C9—C10—C11	58.1 (4)	C9—C10—C23—C18	177.2 (2)
C23—C10—C11—C12	−173.1 (2)	C21—C22—C23—C10	−178.5 (3)
C9—C10—C11—C12	4.8 (4)	C21—C22—C23—C18	1.8 (4)
C23—C10—C11—C16	5.7 (4)	C17—C18—C23—C10	0.8 (3)

C9—C10—C11—C16	−176.3 (2)	C19—C18—C23—C10	−179.3 (2)
C10—C11—C12—C13	−179.6 (3)	C17—C18—C23—C22	−179.5 (2)
C16—C11—C12—C13	1.5 (4)	C19—C18—C23—C22	0.4 (3)
C11—C12—C13—C14	1.2 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8A···O1^{i}	0.93	2.47	3.290 (3)	147
C24—H24A···O1^{ii}	0.96	2.59	3.176 (4)	120
C17—H17A···Cg1^{iii}	0.93	2.89	3.694 (3)	145

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*−1/2, *y*−1/2, *z*; (iii) *x*+1/2, −*y*+1/2, *z*−1/2.

Fig. 1

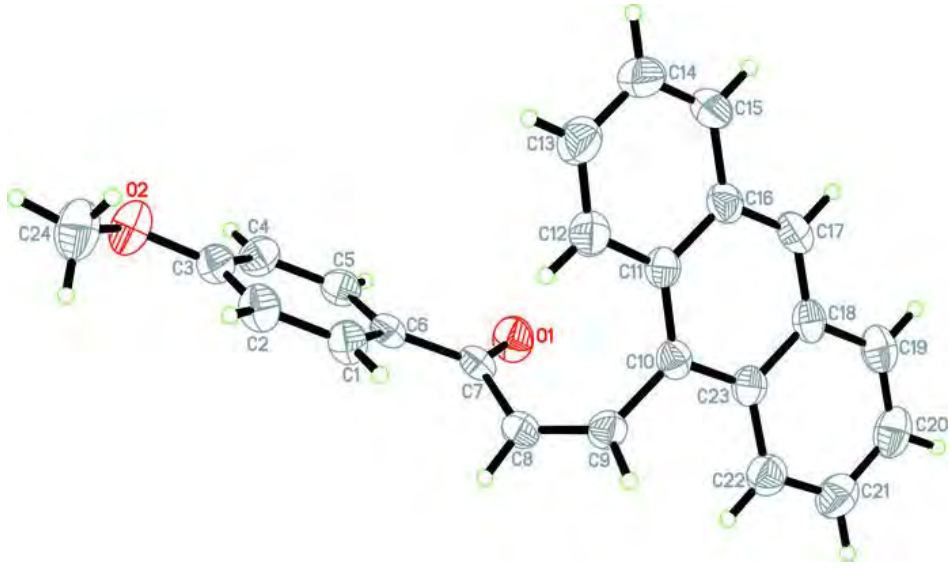
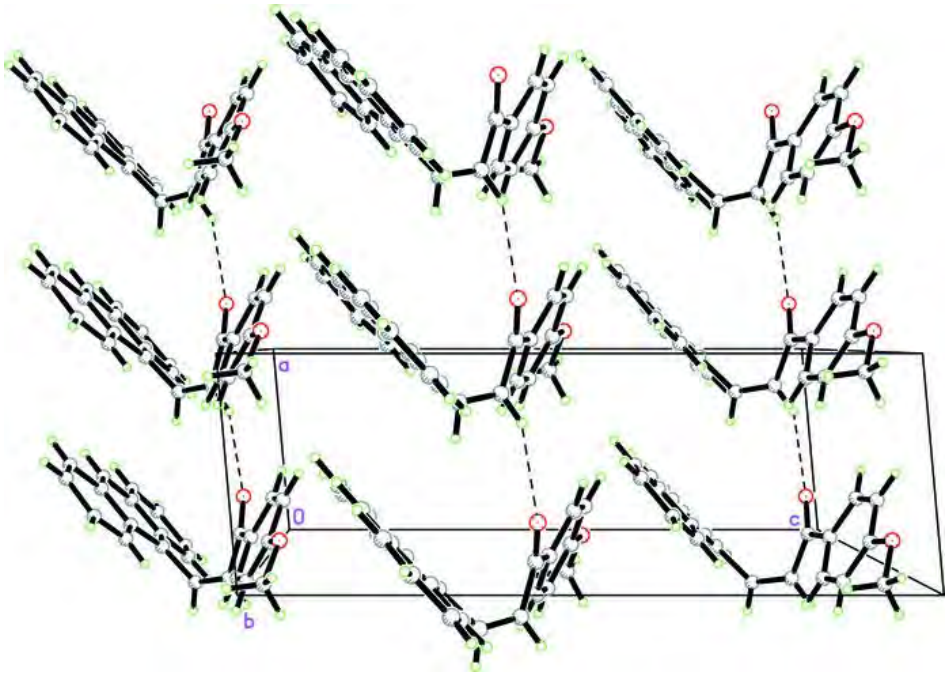


Fig. 2

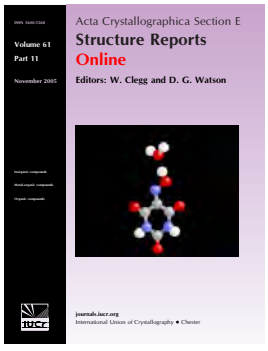


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

6-(4-Bromophenyl)-2-ethoxy-4-(2,4,6-trimethoxyphenyl)nicotinonitrile

Suchada Chantrapromma, Hoong-Kun Fun, Thitipone Suwunwong, Mahesh Padaki and Arun M. Isloor

Acta Cryst. (2009). E**65**, o2914–o2915



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-(4-Bromophenyl)-2-ethoxy-4-(2,4,6-trimethoxyphenyl)nicotinonitrile[1]

Suchada Chantrapromma,[a]*§ Hoong-Kun Fun,[b]¶ Thitipone Suwunwong,[a] Mahesh Padaki[c] and Arun M. Isloor[c]

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [c]Department of Chemistry, National Institute of Technology–Karnataka, Surathkal, Mangalore 575 025, India
Correspondence e-mail: suchada.c@psu.ac.th

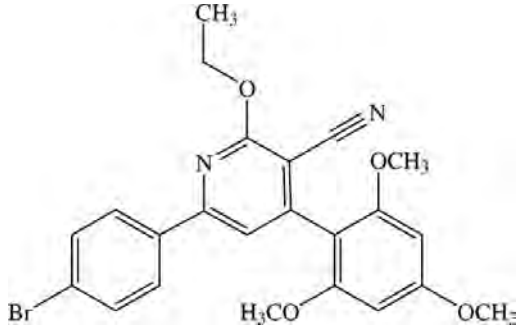
Received 11 October 2009; accepted 23 October 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.095; data-to-parameter ratio = 34.1.

In the asymmetric unit of the title nicotinonitrile derivative, $C_{23}H_{21}BrN_2O_4$, there are two non-planar independent molecules. The central pyridine ring makes dihedral angles of 9.05 (7) and 77.06 (7)°, respectively, with the 4-bromophenyl and 2,4,6-trimethoxyphenyl rings in one molecule, whereas the corresponding values are 5.96 (7) and 82.37 (7)° in the other. All the three methoxy groups are essentially in the plane of the attached benzene ring [C—O—C—C angles = 2.99 (19), 4.8 (2) and −6.2 (2)° in one molecule, and 2.69 (18), 176.73 (15) and 1.3 (2)° in the other]. The ethoxy group is slightly twisted in one molecule [C—C—O—C = 173.84 (12)°], whereas it is coplanar with the pyridine ring in the other [C—C—O—C = −177.23 (13)°]. Weak intramolecular C—H···N interactions generate S(5) ring motifs. In the crystal structure, the molecules are linked by weak intermolecular C—H···N and C—H···O interactions into a supramolecular three-dimensional network in such a way that the nicotinonitrile units of neighboring molecules are stacked in an antiparallel manner along the c axis. The crystal is further stabilized by C—H···π interactions.

Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the synthesis and applications of nicotinonitrile derivatives, see: Abdel-Aziz (2007); Borgna *et al.* (1993); Chantrapromma *et al.* (2009); Goda *et al.* (2004); Raghukumar *et al.* (2003). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{23}H_{21}BrN_2O_4$
M_r = 469.32
Monoclinic, $P2_1/c$
a = 14.1799 (2) Å
b = 18.0877 (3) Å
c = 16.6881 (2) Å
β = 95.081 (1)°
V = 4263.38 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.96 mm^{-1}
T = 100 K
0.51 × 0.49 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.437, T_{max} = 0.669
85168 measured reflections
18727 independent reflections
12072 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.095
S = 1.01
18727 reflections
549 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1A—H1AA···N1A	0.93	2.45	2.7872 (19)	101
C1A—H1AA···O4B^{i}	0.93	2.60	3.4813 (18)	159
C8A—H8AA···O2B	0.93	2.44	3.3391 (16)	162
C1B—H1BA···N1B	0.93	2.43	2.7751 (19)	102
C8B—H8BA···O2A	0.93	2.39	3.2889 (16)	162
C18A—H18B···O4B^{i}	0.97	2.57	3.3360 (18)	136
C20A—H20B···O2A^{ii}	0.96	2.58	3.2869 (17)	131
C20B—H20E···O2B^{iii}	0.96	2.55	3.2169 (17)	126
C21A—H21B···O3B^{iv}	0.96	2.57	3.154 (2)	119
C22A—H22B···N2B^{iii}	0.96	2.58	3.479 (2)	155
C18B—H18D···$Cg1^{iv}$	0.97	2.93	3.7798 (16)	147
C20A—H20C···$Cg3$	0.96	2.60	3.5075 (15)	157
C20B—H20F···$Cg2$	0.96	2.51	3.3845 (15)	152

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+2, -y, -z+1$; (iii) $-x+1, -y, -z+1$; (iv) $x, -y-\frac{1}{2}, z-\frac{1}{2}$. $Cg1$, $Cg2$ and $Cg3$ are the centroids of the C7A–C11A/N1A, C12A–C17A and C12B–C17B rings, respectively.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine

[1] This paper is dedicated to the late His Royal Highness King Chulalongkorn (King Rama V) of Thailand for his numerous reforms to modernize the country on the occasion of Chulalongkorn Day (Piyamaharaj Day) which fell on the 23rd October.

§ Thomson Reuters ResearcherID: A-5085-2009.
¶ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for research grant (RSA5280033) and Prince of Songkla University for financial support. AMI is grateful to the Director, NITK-Surathkal, India, for providing research facilities and the Head of the Department of Chemistry and Dean R&D, NITK Surathkal, for their encouragement. The authors also thank Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2473).

References

Abdel-Aziz, A. A.-M. (2007). *Tetrahedron Lett.* **48**, 2861–2865.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Borgna, P., Pregnolato, M., Gamba, I. A. & Mellerio, G. (1993). *J. Heterocycl. Chem.* **30**, 1079–1084.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chantrapromma, S., Suwunwong, T., Karalai, C. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o893–o894.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Goda, F. E., Abdel-Aziz, A. A.-M. & Attef, O. A. (2004). *Bioorg. Med. Chem.* **12**, 1845–1852.
Raghukumar, V., Thrirumalai, D., Ramakrishman, V. T., Karunakara, V. & Ramamurthy, P. (2003). *Tetrahedron*, **59**, 3761–3768.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2009). E**65**, o2914-o2915 [**doi:10.1107/S1600536809043943**]

6-(4-Bromophenyl)-2-ethoxy-4-(2,4,6-trimethoxyphenyl)nicotinonitrile

S. Chantrapromma, H.-K. Fun, T. Suwunwong, M. Padaki and A. M. Isloor

Comment

The pyridine ring is among the most common heterocyclic compound found in the naturally occurring heterocycles and in various therapeutic agents. The substituted pyridine derivatives have been claimed to have several biological activities (Borgna *et al.*, 1993; Goda *et al.*, 2004) and non-linear optical properties (Raghukumar *et al.*, 2003). The title nicotinonitrile derivative is a compound containing a pyridine ring which was synthesized by cyclization of chalcone derivative (Chantrapromma *et al.*, 2009) and malononitrile in order to be tested as antibacterial agents. It was tested against both Gram-positive bacteria *i.e. Staphyrococcus aureus*, *Bacillus subtilis*, *Enterococcus faecalis*, Methicillin-Resistant *Staphyrococcus aureus* and Vancomycin-Resistant *Enterococcus faecalis*, and Gram-negative bacteria *i.e. Pseudomonas aeruginosa*, *Salmonella typhi* and *Shigella sonnei*. Our results showed that the title compound has no antibacterial action against these pathogens, having the same results as its starting chalcone derivative (Chantrapromma *et al.*, 2009). Herein we report the crystal structure of the title compound (I).

There are two crystallographic independent molecules *A* and *B* in the asymmetric unit of (I) (Fig. 1) with slight differences in bond angles and in the conformation of the middle methoxy group in 2,4,6-trimethoxyphenyl unit between the two molecules. The molecular structure of (I), $C_{23}H_{21}BrN_2O_4$ is not planar. The central pyridine ring is nearly planar with the 4-bromophenyl ring with the dihedral angles of 9.05 (7)° [5.96 (7)° in molecule *B*] whereas is inclined to the 2,4,6-trimethoxyphenyl unit with the torsion angle of 77.06 (7)° [82.37 (7)° in molecule *B*] due to the steric effect between the methoxy and cyano groups. All the three methoxy groups are nearly co-planar to the attached benzene ring with the torsion angles C20–O2–C13–C14 = 2.99 (19)°, C21–O3–C15–C16 = 4.8 (2)° and C22–O4–C17–C16 = -6.2 (2)° in molecule *A* and the corresponding values are 2.69 (18), 176.73 (15) and 1.3 (2)° in molecule *B*. However these values show that the middle methoxy group is in different orientation in which it tilts to the methoxy group at C17 in molecule *A* but tilts to the methoxy group at C13 in molecule *B*. The ethoxy group in molecule *A* is slightly twisted with respect to the pyridine ring as indicated by the torsion angles C11–O1–C18–C19 of 173.84 (12)° and N1–C11–O1–C18 = 7.48 (19)° whereas it is co-planar in molecule *B* as shown by the corresponding values of -177.23 (13) and 0.12 (19)°. Intramolecular C1A—H1AA···N1A and C1B–H1BA–N1B interactions generate S(5) ring motifs (Bernstein *et al.*, 1995). The bond distances agree with the literature values (Allen *et al.*, 1987).

In the crystal structure (Fig. 2), the molecules are linked by intermolecular C—H···N and C—H···O weak interactions (Table 1) into a supramolecular three-dimensional network in such a way that the nicotinonitrile moiety of the neighbouring molecules are stacked in an antiparallel manner along the *c* axis. The crystal is further stabilized by C—H···π interactions (Table 1); Cg_1, Cg_2 and Cg_3 are the centroids of C7A–C11A/N1A, C12A–C17A and C12B–C17B rings, respectively.

Experimental

E-1-(4-Bromophenyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one which was synthesized according to the previous procedure (Chantrapromma *et al.*, 2009) (0.57 g, 0.0015 mol) were added with continuous stirring to a freshly prepared sodium alkoxide (0.0014 mol of sodium in 100 ml of ethanol). Malononitrile (1.30 g, 0.02 mol) was then added with continuous

stirring at room temperature until the precipitate was separated out. The resulting solid was filtered (yield 72%). Colorless block-shaped single crystals of the title compound suitable for *X*-ray structure determination were recrystalized from ethanol by the slow evaporation of the solvent at room temperature after several days (m.p. 423–424 K).

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C—H) = 0.93 Å for aromatic, 0.97 Å for CH_2 and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.69 Å from C16B and the deepest hole is located at 0.41 Å from Br1B.

Figures

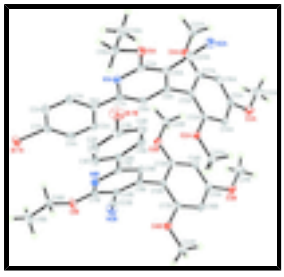


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. For clarity, aromatic H atoms are not shown.

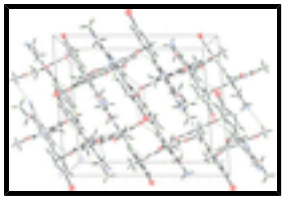


Fig. 2. The crystal packing of the title compound viewed along the *b* axis, showing supra-molecular three-dimensional network. Hydrogen bonds are shown as dashed lines.

6-(4-Bromophenyl)-2-ethoxy-4-(2,4,6-trimethoxyphenyl)nicotinonitrile

Crystal data

$C_{23}H_{21}BrN_2O_4$	F_{000} = 1920
M_r = 469.32	D_x = 1.462 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point = 423–424 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 14.1799 (2) Å	Cell parameters from 18727 reflections
b = 18.0877 (3) Å	θ = 2.1–35.0º
c = 16.6881 (2) Å	μ = 1.96 mm^{-1}
β = 95.081 (1)º	T = 100 K
V = 4263.38 (11) Å^3	Block, colorless
Z = 8	0.51 × 0.49 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	18727 independent reflections

Radiation source: sealed tube	12072 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.044$
$T = 100$ K	$\theta_{max} = 35.0^o$
φ and ω scans	$\theta_{min} = 2.1^o$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -22 \rightarrow 22$
$T_{min} = 0.437$, $T_{max} = 0.669$	$k = -29 \rightarrow 27$
85168 measured reflections	$l = -26 \rightarrow 25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.095$	$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2 + 1.2763P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.003$
18727 reflections	$\Delta\rho_{max} = 0.50$ e Å^{-3}
549 parameters	$\Delta\rho_{min} = -0.45$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1A	0.415133 (12)	0.399816 (9)	0.519413 (10)	0.02931 (5)
O1A	0.93118 (7)	0.24881 (5)	0.28179 (6)	0.0205 (2)
O2A	0.88259 (7)	0.02891 (5)	0.50720 (6)	0.01749 (19)
O3A	0.79300 (9)	−0.21035 (6)	0.41508 (6)	0.0279 (2)
O4A	0.74715 (8)	0.00621 (5)	0.24317 (6)	0.0216 (2)
N1A	0.79667 (8)	0.25126 (6)	0.35033 (7)	0.0163 (2)
N2A	0.99990 (10)	0.06683 (8)	0.24974 (8)	0.0274 (3)

C1A	0.64576 (11)	0.33207 (8)	0.40150 (9)	0.0213 (3)
H1AA	0.6868	0.3535	0.3677	0.026*
C2A	0.57443 (11)	0.37470 (8)	0.42979 (10)	0.0244 (3)
H2AA	0.5670	0.4240	0.4146	0.029*
C3A	0.51453 (10)	0.34262 (8)	0.48100 (9)	0.0212 (3)
C4A	0.52353 (10)	0.26904 (8)	0.50332 (9)	0.0210 (3)
H4AA	0.4827	0.2483	0.5377	0.025*
C5A	0.59440 (10)	0.22651 (8)	0.47372 (8)	0.0186 (3)
H5AA	0.6002	0.1769	0.4878	0.022*
C6A	0.65723 (10)	0.25764 (7)	0.42278 (8)	0.0166 (2)
C7A	0.73374 (10)	0.21320 (7)	0.39098 (8)	0.0153 (2)
C8A	0.74013 (10)	0.13710 (7)	0.40206 (8)	0.0163 (2)
H8AA	0.6982	0.1130	0.4330	0.020*
C9A	0.80937 (10)	0.09694 (7)	0.36680 (8)	0.0159 (2)
C10A	0.87236 (10)	0.13553 (7)	0.32258 (8)	0.0166 (2)
C11A	0.86418 (10)	0.21338 (7)	0.31897 (8)	0.0169 (3)
C12A	0.81270 (9)	0.01486 (7)	0.37548 (8)	0.0157 (2)
C13A	0.84560 (10)	−0.01815 (7)	0.44856 (8)	0.0161 (2)
C14A	0.84073 (10)	−0.09404 (7)	0.45904 (9)	0.0181 (3)
H14A	0.8641	−0.1157	0.5073	0.022*
C15A	0.80025 (11)	−0.13720 (7)	0.39600 (9)	0.0199 (3)
C16A	0.76879 (10)	−0.10705 (8)	0.32189 (9)	0.0195 (3)
H16A	0.7434	−0.1367	0.2799	0.023*
C17A	0.77663 (10)	−0.03050 (7)	0.31265 (8)	0.0173 (3)
C18A	0.93260 (11)	0.32872 (8)	0.28667 (9)	0.0208 (3)
H18A	0.9353	0.3446	0.3423	0.025*
H18B	0.8761	0.3493	0.2580	0.025*
C19A	1.01910 (12)	0.35363 (9)	0.24895 (10)	0.0294 (4)
H19A	1.0228	0.4066	0.2506	0.044*
H19B	1.0156	0.3372	0.1941	0.044*
H19C	1.0744	0.3330	0.2780	0.044*
C20A	0.92176 (10)	−0.00406 (8)	0.58126 (8)	0.0194 (3)
H20A	0.9507	0.0336	0.6158	0.029*
H20B	0.9685	−0.0401	0.5700	0.029*
H20C	0.8722	−0.0277	0.6075	0.029*
C21A	0.75721 (18)	−0.25954 (9)	0.35314 (11)	0.0464 (5)
H21A	0.7560	−0.3089	0.3741	0.070*
H21B	0.7973	−0.2580	0.3097	0.070*
H21C	0.6942	−0.2449	0.3338	0.070*
C22A	0.71499 (11)	−0.03616 (8)	0.17402 (9)	0.0233 (3)
H22A	0.6994	−0.0036	0.1294	0.035*
H22B	0.6598	−0.0639	0.1850	0.035*
H22C	0.7641	−0.0695	0.1611	0.035*
C23A	0.94346 (11)	0.09810 (8)	0.28221 (9)	0.0200 (3)
Br1B	1.103317 (12)	0.395272 (9)	0.476239 (10)	0.02990 (5)
O1B	0.57485 (7)	0.27568 (5)	0.71164 (6)	0.0202 (2)
O2B	0.62698 (7)	0.02194 (5)	0.51718 (6)	0.01751 (19)
O3B	0.73304 (9)	−0.19538 (6)	0.66989 (7)	0.0289 (3)
O4B	0.76063 (8)	0.04510 (5)	0.78231 (6)	0.0212 (2)

N1B	0.71233 (8)	0.26852 (6)	0.64794 (7)	0.0165 (2)
N2B	0.47997 (10)	0.09979 (8)	0.73356 (9)	0.0300 (3)
C1B	0.86314 (11)	0.34248 (8)	0.58904 (9)	0.0224 (3)
H1BA	0.8191	0.3678	0.6168	0.027*
C2B	0.93700 (12)	0.38133 (8)	0.55887 (10)	0.0255 (3)
H2BA	0.9428	0.4321	0.5666	0.031*
C3B	1.00145 (11)	0.34315 (8)	0.51734 (9)	0.0211 (3)
C4B	0.99424 (10)	0.26760 (8)	0.50491 (9)	0.0212 (3)
H4BA	1.0382	0.2429	0.4765	0.025*
C5B	0.92041 (10)	0.22931 (8)	0.53546 (9)	0.0197 (3)
H5BA	0.9150	0.1786	0.5274	0.024*
C6B	0.85397 (10)	0.26631 (7)	0.57832 (8)	0.0161 (2)
C7B	0.77575 (9)	0.22614 (7)	0.61271 (8)	0.0154 (2)
C8B	0.76838 (10)	0.14930 (7)	0.61027 (8)	0.0159 (2)
H8BA	0.8128	0.1216	0.5855	0.019*
C9B	0.69472 (10)	0.11389 (7)	0.64472 (8)	0.0152 (2)
C10B	0.62731 (10)	0.15791 (7)	0.67798 (8)	0.0163 (2)
C11B	0.64068 (10)	0.23539 (7)	0.67824 (8)	0.0164 (2)
C12B	0.69364 (9)	0.03151 (7)	0.65008 (8)	0.0156 (2)
C13B	0.66365 (9)	−0.01382 (7)	0.58520 (8)	0.0151 (2)
C14B	0.67260 (10)	−0.09074 (7)	0.58966 (9)	0.0181 (3)
H14B	0.6503	−0.1205	0.5466	0.022*
C15B	0.71545 (11)	−0.12171 (8)	0.65961 (9)	0.0205 (3)
C16B	0.74559 (11)	−0.07822 (7)	0.72598 (9)	0.0194 (3)
H16B	0.7735	−0.0998	0.7728	0.023*
C17B	0.73323 (10)	−0.00229 (7)	0.72081 (8)	0.0165 (3)
C18B	0.58732 (11)	0.35528 (7)	0.71198 (9)	0.0212 (3)
H18C	0.5843	0.3740	0.6574	0.025*
H18D	0.6483	0.3684	0.7392	0.025*
C19B	0.50878 (13)	0.38711 (9)	0.75552 (11)	0.0322 (4)
H19D	0.5140	0.4400	0.7567	0.048*
H19E	0.5130	0.3685	0.8096	0.048*
H19F	0.4490	0.3732	0.7282	0.048*
C20B	0.59292 (10)	−0.02279 (8)	0.44918 (9)	0.0196 (3)
H20D	0.5672	0.0086	0.4063	0.029*
H20E	0.5446	−0.0557	0.4646	0.029*
H20F	0.6443	−0.0511	0.4313	0.029*
C21B	0.70011 (16)	−0.24452 (9)	0.60668 (11)	0.0396 (5)
H21D	0.7188	−0.2941	0.6210	0.059*
H21E	0.7270	−0.2306	0.5581	0.059*
H21F	0.6323	−0.2419	0.5984	0.059*
C22B	0.80585 (12)	0.01372 (9)	0.85449 (9)	0.0252 (3)
H22D	0.8217	0.0523	0.8928	0.038*
H22E	0.8625	−0.0115	0.8425	0.038*
H22F	0.7637	−0.0207	0.8766	0.038*
C23B	0.54596 (11)	0.12606 (8)	0.70986 (9)	0.0203 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1A	0.02877 (9)	0.03090 (8)	0.02875 (9)	0.01345 (6)	0.00520 (6)	−0.00364 (7)
O1A	0.0216 (5)	0.0169 (5)	0.0241 (5)	−0.0004 (4)	0.0076 (4)	0.0033 (4)
O2A	0.0201 (5)	0.0157 (4)	0.0159 (5)	0.0020 (4)	−0.0026 (4)	−0.0004 (4)
O3A	0.0481 (7)	0.0126 (5)	0.0226 (5)	−0.0021 (5)	0.0006 (5)	−0.0012 (4)
O4A	0.0311 (6)	0.0173 (5)	0.0156 (5)	0.0008 (4)	−0.0028 (4)	−0.0009 (4)
N1A	0.0176 (5)	0.0148 (5)	0.0165 (5)	0.0008 (4)	0.0013 (4)	0.0010 (4)
N2A	0.0293 (7)	0.0286 (7)	0.0250 (7)	0.0085 (6)	0.0069 (5)	0.0033 (5)
C1A	0.0248 (7)	0.0159 (6)	0.0235 (7)	0.0014 (5)	0.0039 (6)	0.0008 (5)
C2A	0.0286 (8)	0.0165 (6)	0.0285 (8)	0.0051 (6)	0.0044 (6)	−0.0014 (6)
C3A	0.0199 (7)	0.0230 (7)	0.0202 (7)	0.0063 (5)	−0.0006 (5)	−0.0048 (5)
C4A	0.0194 (7)	0.0254 (7)	0.0183 (7)	0.0040 (5)	0.0020 (5)	0.0016 (5)
C5A	0.0186 (7)	0.0180 (6)	0.0190 (7)	0.0024 (5)	0.0009 (5)	0.0021 (5)
C6A	0.0174 (6)	0.0156 (6)	0.0167 (6)	0.0012 (5)	0.0006 (5)	−0.0002 (5)
C7A	0.0169 (6)	0.0144 (6)	0.0144 (6)	0.0007 (5)	−0.0003 (5)	−0.0002 (5)
C8A	0.0178 (6)	0.0145 (6)	0.0167 (6)	0.0002 (5)	0.0022 (5)	−0.0001 (5)
C9A	0.0171 (6)	0.0137 (6)	0.0165 (6)	0.0012 (5)	−0.0005 (5)	−0.0006 (5)
C10A	0.0170 (6)	0.0170 (6)	0.0158 (6)	0.0022 (5)	0.0013 (5)	0.0008 (5)
C11A	0.0183 (6)	0.0164 (6)	0.0159 (6)	−0.0001 (5)	0.0012 (5)	0.0022 (5)
C12A	0.0160 (6)	0.0133 (6)	0.0181 (6)	0.0009 (5)	0.0028 (5)	−0.0002 (5)
C13A	0.0159 (6)	0.0146 (6)	0.0180 (6)	0.0013 (5)	0.0020 (5)	−0.0028 (5)
C14A	0.0212 (7)	0.0143 (6)	0.0187 (6)	0.0027 (5)	0.0009 (5)	0.0005 (5)
C15A	0.0246 (7)	0.0135 (6)	0.0222 (7)	0.0013 (5)	0.0046 (6)	−0.0011 (5)
C16A	0.0226 (7)	0.0162 (6)	0.0199 (7)	−0.0008 (5)	0.0017 (5)	−0.0049 (5)
C17A	0.0180 (6)	0.0167 (6)	0.0173 (6)	0.0025 (5)	0.0018 (5)	−0.0003 (5)
C18A	0.0240 (7)	0.0164 (6)	0.0221 (7)	−0.0024 (5)	0.0017 (6)	0.0029 (5)
C19A	0.0315 (9)	0.0280 (8)	0.0295 (8)	−0.0077 (7)	0.0068 (7)	0.0046 (7)
C20A	0.0203 (7)	0.0207 (6)	0.0168 (6)	0.0033 (5)	−0.0005 (5)	−0.0003 (5)
C21A	0.0914 (17)	0.0167 (7)	0.0298 (9)	−0.0114 (9)	−0.0025 (10)	−0.0053 (7)
C22A	0.0291 (8)	0.0252 (7)	0.0156 (7)	−0.0035 (6)	0.0007 (6)	−0.0022 (5)
C23A	0.0227 (7)	0.0186 (6)	0.0188 (6)	0.0022 (5)	0.0022 (5)	0.0036 (5)
Br1B	0.03256 (9)	0.03036 (8)	0.02790 (8)	−0.01565 (7)	0.00899 (7)	−0.00084 (6)
O1B	0.0215 (5)	0.0148 (4)	0.0251 (5)	0.0019 (4)	0.0066 (4)	−0.0036 (4)
O2B	0.0203 (5)	0.0152 (4)	0.0164 (5)	0.0000 (4)	−0.0017 (4)	0.0008 (4)
O3B	0.0466 (7)	0.0111 (4)	0.0266 (6)	−0.0007 (4)	−0.0092 (5)	0.0014 (4)
O4B	0.0309 (6)	0.0161 (5)	0.0159 (5)	0.0000 (4)	−0.0021 (4)	−0.0010 (4)
N1B	0.0180 (6)	0.0140 (5)	0.0174 (5)	0.0003 (4)	0.0013 (4)	−0.0017 (4)
N2B	0.0264 (7)	0.0298 (7)	0.0349 (8)	−0.0051 (6)	0.0099 (6)	−0.0027 (6)
C1B	0.0265 (8)	0.0163 (6)	0.0255 (7)	−0.0029 (5)	0.0077 (6)	−0.0017 (5)
C2B	0.0346 (9)	0.0158 (6)	0.0271 (8)	−0.0078 (6)	0.0072 (7)	−0.0023 (6)
C3B	0.0229 (7)	0.0207 (7)	0.0197 (7)	−0.0074 (5)	0.0018 (5)	0.0029 (5)
C4B	0.0198 (7)	0.0203 (7)	0.0238 (7)	−0.0013 (5)	0.0038 (6)	0.0014 (5)
C5B	0.0205 (7)	0.0158 (6)	0.0231 (7)	−0.0005 (5)	0.0031 (5)	0.0015 (5)
C6B	0.0185 (6)	0.0145 (6)	0.0152 (6)	−0.0010 (5)	−0.0001 (5)	0.0012 (5)
C7B	0.0172 (6)	0.0145 (6)	0.0146 (6)	−0.0004 (5)	0.0011 (5)	−0.0002 (5)

C8B	0.0169 (6)	0.0131 (5)	0.0178 (6)	0.0014 (5)	0.0022 (5)	0.0000 (5)
C9B	0.0166 (6)	0.0131 (6)	0.0157 (6)	−0.0002 (4)	0.0004 (5)	0.0009 (5)
C10B	0.0172 (6)	0.0154 (6)	0.0163 (6)	−0.0008 (5)	0.0017 (5)	−0.0005 (5)
C11B	0.0174 (6)	0.0151 (6)	0.0165 (6)	0.0014 (5)	0.0010 (5)	−0.0028 (5)
C12B	0.0163 (6)	0.0120 (5)	0.0189 (6)	−0.0007 (4)	0.0035 (5)	0.0002 (5)
C13B	0.0133 (6)	0.0151 (6)	0.0171 (6)	−0.0005 (4)	0.0022 (5)	0.0013 (5)
C14B	0.0202 (7)	0.0142 (6)	0.0196 (7)	−0.0022 (5)	0.0005 (5)	−0.0004 (5)
C15B	0.0246 (7)	0.0130 (6)	0.0238 (7)	−0.0016 (5)	0.0024 (6)	0.0021 (5)
C16B	0.0251 (7)	0.0146 (6)	0.0182 (6)	−0.0012 (5)	−0.0007 (5)	0.0033 (5)
C17B	0.0185 (6)	0.0141 (6)	0.0171 (6)	−0.0027 (5)	0.0022 (5)	−0.0007 (5)
C18B	0.0252 (7)	0.0143 (6)	0.0241 (7)	0.0021 (5)	0.0015 (6)	−0.0029 (5)
C19B	0.0352 (9)	0.0256 (8)	0.0371 (9)	0.0069 (7)	0.0102 (7)	−0.0078 (7)
C20B	0.0190 (7)	0.0203 (6)	0.0191 (7)	−0.0007 (5)	−0.0010 (5)	−0.0019 (5)
C21B	0.0672 (14)	0.0138 (7)	0.0347 (10)	−0.0012 (8)	−0.0124 (9)	−0.0030 (7)
C22B	0.0341 (9)	0.0239 (7)	0.0168 (7)	0.0015 (6)	−0.0025 (6)	0.0000 (6)
C23B	0.0219 (7)	0.0170 (6)	0.0221 (7)	0.0001 (5)	0.0030 (5)	−0.0034 (5)

Geometric parameters (Å, °)

Br1A—C3A	1.9047 (14)	Br1B—C3B	1.9029 (14)
O1A—C11A	1.3426 (16)	O1B—C11B	1.3436 (16)
O1A—C18A	1.4478 (17)	O1B—C18B	1.4505 (17)
O2A—C13A	1.3662 (16)	O2B—C13B	1.3684 (16)
O2A—C20A	1.4389 (17)	O2B—C20B	1.4418 (17)
O3A—C15A	1.3668 (17)	O3B—C15B	1.3637 (17)
O3A—C21A	1.423 (2)	O3B—C21B	1.4259 (19)
O4A—C17A	1.3690 (17)	O4B—C17B	1.3669 (16)
O4A—C22A	1.4262 (17)	O4B—C22B	1.4310 (18)
N1A—C11A	1.3223 (17)	N1B—C11B	1.3185 (17)
N1A—C7A	1.3555 (17)	N1B—C7B	1.3550 (17)
N2A—C23A	1.1536 (19)	N2B—C23B	1.1504 (19)
C1A—C2A	1.387 (2)	C1B—C2B	1.392 (2)
C1A—C6A	1.3982 (19)	C1B—C6B	1.3940 (19)
C1A—H1AA	0.9300	C1B—H1BA	0.9300
C2A—C3A	1.385 (2)	C2B—C3B	1.380 (2)
C2A—H2AA	0.9300	C2B—H2BA	0.9300
C3A—C4A	1.385 (2)	C3B—C4B	1.385 (2)
C4A—C5A	1.3907 (19)	C4B—C5B	1.3894 (19)
C4A—H4AA	0.9300	C4B—H4BA	0.9300
C5A—C6A	1.4026 (19)	C5B—C6B	1.4021 (19)
C5A—H5AA	0.9300	C5B—H5BA	0.9300
C6A—C7A	1.4856 (19)	C6B—C7B	1.4833 (18)
C7A—C8A	1.3908 (18)	C7B—C8B	1.3942 (18)
C8A—C9A	1.3929 (18)	C8B—C9B	1.3921 (18)
C8A—H8AA	0.9300	C8B—H8BA	0.9300
C9A—C10A	1.3952 (19)	C9B—C10B	1.3962 (18)
C9A—C12A	1.4919 (18)	C9B—C12B	1.4930 (18)
C10A—C11A	1.4137 (19)	C10B—C11B	1.4141 (18)
C10A—C23A	1.4314 (19)	C10B—C23B	1.4333 (19)

C12A—C17A	1.3924 (19)	C12B—C13B	1.3943 (19)
C12A—C13A	1.4002 (19)	C12B—C17B	1.4017 (19)
C13A—C14A	1.3863 (18)	C13B—C14B	1.3985 (18)
C14A—C15A	1.392 (2)	C14B—C15B	1.386 (2)
C14A—H14A	0.9300	C14B—H14B	0.9300
C15A—C16A	1.389 (2)	C15B—C16B	1.394 (2)
C16A—C17A	1.3987 (19)	C16B—C17B	1.3862 (19)
C16A—H16A	0.9300	C16B—H16B	0.9300
C18A—C19A	1.497 (2)	C18B—C19B	1.498 (2)
C18A—H18A	0.9700	C18B—H18C	0.9700
C18A—H18B	0.9700	C18B—H18D	0.9700
C19A—H19A	0.9600	C19B—H19D	0.9600
C19A—H19B	0.9600	C19B—H19E	0.9600
C19A—H19C	0.9600	C19B—H19F	0.9600
C20A—H20A	0.9600	C20B—H20D	0.9600
C20A—H20B	0.9600	C20B—H20E	0.9600
C20A—H20C	0.9600	C20B—H20F	0.9600
C21A—H21A	0.9600	C21B—H21D	0.9600
C21A—H21B	0.9600	C21B—H21E	0.9600
C21A—H21C	0.9600	C21B—H21F	0.9600
C22A—H22A	0.9600	C22B—H22D	0.9600
C22A—H22B	0.9600	C22B—H22E	0.9600
C22A—H22C	0.9600	C22B—H22F	0.9600
C11A—O1A—C18A	117.22 (11)	C11B—O1B—C18B	116.81 (11)
C13A—O2A—C20A	116.84 (11)	C13B—O2B—C20B	117.63 (10)
C15A—O3A—C21A	117.74 (12)	C15B—O3B—C21B	118.15 (12)
C17A—O4A—C22A	118.45 (11)	C17B—O4B—C22B	117.36 (11)
C11A—N1A—C7A	117.82 (11)	C11B—N1B—C7B	118.22 (11)
C2A—C1A—C6A	121.37 (14)	C2B—C1B—C6B	121.14 (14)
C2A—C1A—H1AA	119.3	C2B—C1B—H1BA	119.4
C6A—C1A—H1AA	119.3	C6B—C1B—H1BA	119.4
C3A—C2A—C1A	118.86 (14)	C3B—C2B—C1B	118.81 (13)
C3A—C2A—H2AA	120.6	C3B—C2B—H2BA	120.6
C1A—C2A—H2AA	120.6	C1B—C2B—H2BA	120.6
C2A—C3A—C4A	121.46 (13)	C2B—C3B—C4B	121.72 (13)
C2A—C3A—Br1A	119.52 (11)	C2B—C3B—Br1B	119.39 (11)
C4A—C3A—Br1A	118.99 (11)	C4B—C3B—Br1B	118.90 (11)
C3A—C4A—C5A	119.23 (13)	C3B—C4B—C5B	119.01 (14)
C3A—C4A—H4AA	120.4	C3B—C4B—H4BA	120.5
C5A—C4A—H4AA	120.4	C5B—C4B—H4BA	120.5
C4A—C5A—C6A	120.73 (13)	C4B—C5B—C6B	120.76 (13)
C4A—C5A—H5AA	119.6	C4B—C5B—H5BA	119.6
C6A—C5A—H5AA	119.6	C6B—C5B—H5BA	119.6
C1A—C6A—C5A	118.34 (13)	C1B—C6B—C5B	118.55 (13)
C1A—C6A—C7A	120.16 (12)	C1B—C6B—C7B	119.93 (12)
C5A—C6A—C7A	121.49 (12)	C5B—C6B—C7B	121.52 (12)
N1A—C7A—C8A	122.09 (12)	N1B—C7B—C8B	121.72 (12)
N1A—C7A—C6A	115.98 (11)	N1B—C7B—C6B	116.01 (11)
C8A—C7A—C6A	121.94 (12)	C8B—C7B—C6B	122.26 (12)

C7A—C8A—C9A	120.00 (12)	C9B—C8B—C7B	120.24 (12)
C7A—C8A—H8AA	120.0	C9B—C8B—H8BA	119.9
C9A—C8A—H8AA	120.0	C7B—C8B—H8BA	119.9
C8A—C9A—C10A	118.08 (12)	C8B—C9B—C10B	117.84 (12)
C8A—C9A—C12A	119.70 (12)	C8B—C9B—C12B	119.73 (12)
C10A—C9A—C12A	122.20 (12)	C10B—C9B—C12B	122.30 (12)
C9A—C10A—C11A	117.83 (12)	C9B—C10B—C11B	118.04 (12)
C9A—C10A—C23A	121.53 (12)	C9B—C10B—C23B	121.34 (12)
C11A—C10A—C23A	120.63 (12)	C11B—C10B—C23B	120.62 (12)
N1A—C11A—O1A	120.15 (12)	N1B—C11B—O1B	119.99 (12)
N1A—C11A—C10A	123.97 (12)	N1B—C11B—C10B	123.84 (12)
O1A—C11A—C10A	115.87 (12)	O1B—C11B—C10B	116.16 (12)
C17A—C12A—C13A	118.45 (12)	C13B—C12B—C17B	117.99 (12)
C17A—C12A—C9A	120.45 (12)	C13B—C12B—C9B	122.99 (12)
C13A—C12A—C9A	120.94 (12)	C17B—C12B—C9B	118.71 (12)
O2A—C13A—C14A	123.15 (13)	O2B—C13B—C12B	115.69 (11)
O2A—C13A—C12A	115.80 (11)	O2B—C13B—C14B	122.72 (12)
C14A—C13A—C12A	121.05 (13)	C12B—C13B—C14B	121.58 (13)
C13A—C14A—C15A	118.79 (13)	C15B—C14B—C13B	118.55 (13)
C13A—C14A—H14A	120.6	C15B—C14B—H14B	120.7
C15A—C14A—H14A	120.6	C13B—C14B—H14B	120.7
O3A—C15A—C16A	124.25 (13)	O3B—C15B—C14B	124.23 (13)
O3A—C15A—C14A	113.65 (13)	O3B—C15B—C16B	114.31 (13)
C16A—C15A—C14A	122.08 (13)	C14B—C15B—C16B	121.46 (13)
C15A—C16A—C17A	117.68 (13)	C17B—C16B—C15B	118.77 (13)
C15A—C16A—H16A	121.2	C17B—C16B—H16B	120.6
C17A—C16A—H16A	121.2	C15B—C16B—H16B	120.6
O4A—C17A—C12A	114.60 (12)	O4B—C17B—C16B	123.28 (13)
O4A—C17A—C16A	123.52 (13)	O4B—C17B—C12B	115.13 (12)
C12A—C17A—C16A	121.85 (13)	C16B—C17B—C12B	121.57 (13)
O1A—C18A—C19A	106.54 (12)	O1B—C18B—C19B	106.73 (12)
O1A—C18A—H18A	110.4	O1B—C18B—H18C	110.4
C19A—C18A—H18A	110.4	C19B—C18B—H18C	110.4
O1A—C18A—H18B	110.4	O1B—C18B—H18D	110.4
C19A—C18A—H18B	110.4	C19B—C18B—H18D	110.4
H18A—C18A—H18B	108.6	H18C—C18B—H18D	108.6
C18A—C19A—H19A	109.5	C18B—C19B—H19D	109.5
C18A—C19A—H19B	109.5	C18B—C19B—H19E	109.5
H19A—C19A—H19B	109.5	H19D—C19B—H19E	109.5
C18A—C19A—H19C	109.5	C18B—C19B—H19F	109.5
H19A—C19A—H19C	109.5	H19D—C19B—H19F	109.5
H19B—C19A—H19C	109.5	H19E—C19B—H19F	109.5
O2A—C20A—H20A	109.5	O2B—C20B—H20D	109.5
O2A—C20A—H20B	109.5	O2B—C20B—H20E	109.5
H20A—C20A—H20B	109.5	H20D—C20B—H20E	109.5
O2A—C20A—H20C	109.5	O2B—C20B—H20F	109.5
H20A—C20A—H20C	109.5	H20D—C20B—H20F	109.5
H20B—C20A—H20C	109.5	H20E—C20B—H20F	109.5
O3A—C21A—H21A	109.5	O3B—C21B—H21D	109.5

O3A—C21A—H21B	109.5	O3B—C21B—H21E	109.5
H21A—C21A—H21B	109.5	H21D—C21B—H21E	109.5
O3A—C21A—H21C	109.5	O3B—C21B—H21F	109.5
H21A—C21A—H21C	109.5	H21D—C21B—H21F	109.5
H21B—C21A—H21C	109.5	H21E—C21B—H21F	109.5
O4A—C22A—H22A	109.5	O4B—C22B—H22D	109.5
O4A—C22A—H22B	109.5	O4B—C22B—H22E	109.5
H22A—C22A—H22B	109.5	H22D—C22B—H22E	109.5
O4A—C22A—H22C	109.5	O4B—C22B—H22F	109.5
H22A—C22A—H22C	109.5	H22D—C22B—H22F	109.5
H22B—C22A—H22C	109.5	H22E—C22B—H22F	109.5
N2A—C23A—C10A	178.83 (16)	N2B—C23B—C10B	178.29 (17)
C6A—C1A—C2A—C3A	0.9 (2)	C6B—C1B—C2B—C3B	0.4 (2)
C1A—C2A—C3A—C4A	−1.0 (2)	C1B—C2B—C3B—C4B	0.2 (2)
C1A—C2A—C3A—Br1A	−179.19 (12)	C1B—C2B—C3B—Br1B	−179.62 (12)
C2A—C3A—C4A—C5A	0.1 (2)	C2B—C3B—C4B—C5B	−0.3 (2)
Br1A—C3A—C4A—C5A	178.29 (11)	Br1B—C3B—C4B—C5B	179.45 (11)
C3A—C4A—C5A—C6A	1.0 (2)	C3B—C4B—C5B—C6B	0.0 (2)
C2A—C1A—C6A—C5A	0.1 (2)	C2B—C1B—C6B—C5B	−0.7 (2)
C2A—C1A—C6A—C7A	179.46 (14)	C2B—C1B—C6B—C7B	178.65 (14)
C4A—C5A—C6A—C1A	−1.1 (2)	C4B—C5B—C6B—C1B	0.5 (2)
C4A—C5A—C6A—C7A	179.61 (13)	C4B—C5B—C6B—C7B	−178.82 (13)
C11A—N1A—C7A—C8A	1.8 (2)	C11B—N1B—C7B—C8B	−2.1 (2)
C11A—N1A—C7A—C6A	−178.00 (12)	C11B—N1B—C7B—C6B	178.93 (12)
C1A—C6A—C7A—N1A	8.23 (19)	C1B—C6B—C7B—N1B	5.09 (19)
C5A—C6A—C7A—N1A	−172.45 (13)	C5B—C6B—C7B—N1B	−175.57 (13)
C1A—C6A—C7A—C8A	−171.55 (13)	C1B—C6B—C7B—C8B	−173.91 (14)
C5A—C6A—C7A—C8A	7.8 (2)	C5B—C6B—C7B—C8B	5.4 (2)
N1A—C7A—C8A—C9A	−3.9 (2)	N1B—C7B—C8B—C9B	−0.2 (2)
C6A—C7A—C8A—C9A	175.86 (13)	C6B—C7B—C8B—C9B	178.75 (13)
C7A—C8A—C9A—C10A	1.6 (2)	C7B—C8B—C9B—C10B	2.8 (2)
C7A—C8A—C9A—C12A	−176.91 (13)	C7B—C8B—C9B—C12B	−173.05 (13)
C8A—C9A—C10A—C11A	2.4 (2)	C8B—C9B—C10B—C11B	−3.1 (2)
C12A—C9A—C10A—C11A	−179.11 (13)	C12B—C9B—C10B—C11B	172.60 (13)
C8A—C9A—C10A—C23A	−177.84 (13)	C8B—C9B—C10B—C23B	175.86 (13)
C12A—C9A—C10A—C23A	0.6 (2)	C12B—C9B—C10B—C23B	−8.4 (2)
C7A—N1A—C11A—O1A	−176.51 (12)	C7B—N1B—C11B—O1B	−178.12 (12)
C7A—N1A—C11A—C10A	2.6 (2)	C7B—N1B—C11B—C10B	1.7 (2)
C18A—O1A—C11A—N1A	7.48 (19)	C18B—O1B—C11B—N1B	0.12 (19)
C18A—O1A—C11A—C10A	−171.71 (12)	C18B—O1B—C11B—C10B	−179.68 (12)
C9A—C10A—C11A—N1A	−4.7 (2)	C9B—C10B—C11B—N1B	1.0 (2)
C23A—C10A—C11A—N1A	175.50 (13)	C23B—C10B—C11B—N1B	−178.04 (13)
C9A—C10A—C11A—O1A	174.41 (12)	C9B—C10B—C11B—O1B	−179.24 (12)
C23A—C10A—C11A—O1A	−5.3 (2)	C23B—C10B—C11B—O1B	1.8 (2)
C8A—C9A—C12A—C17A	101.45 (16)	C8B—C9B—C12B—C13B	−79.57 (18)
C10A—C9A—C12A—C17A	−77.02 (18)	C10B—C9B—C12B—C13B	104.76 (16)
C8A—C9A—C12A—C13A	−73.84 (18)	C8B—C9B—C12B—C17B	93.84 (16)
C10A—C9A—C12A—C13A	107.69 (16)	C10B—C9B—C12B—C17B	−81.82 (17)
C20A—O2A—C13A—C14A	2.99 (19)	C20B—O2B—C13B—C12B	−178.53 (11)

C20A—O2A—C13A—C12A	−176.57 (11)	C20B—O2B—C13B—C14B	2.69 (18)
C17A—C12A—C13A—O2A	178.24 (12)	C17B—C12B—C13B—O2B	−178.96 (11)
C9A—C12A—C13A—O2A	−6.38 (19)	C9B—C12B—C13B—O2B	−5.51 (19)
C17A—C12A—C13A—C14A	−1.3 (2)	C17B—C12B—C13B—C14B	−0.2 (2)
C9A—C12A—C13A—C14A	174.04 (13)	C9B—C12B—C13B—C14B	173.29 (13)
O2A—C13A—C14A—C15A	178.92 (12)	O2B—C13B—C14B—C15B	176.43 (12)
C12A—C13A—C14A—C15A	−1.5 (2)	C12B—C13B—C14B—C15B	−2.3 (2)
C21A—O3A—C15A—C16A	4.8 (2)	C21B—O3B—C15B—C14B	−4.1 (2)
C21A—O3A—C15A—C14A	−176.65 (16)	C21B—O3B—C15B—C16B	176.73 (15)
C13A—C14A—C15A—O3A	−175.49 (13)	C13B—C14B—C15B—O3B	−176.30 (13)
C13A—C14A—C15A—C16A	3.1 (2)	C13B—C14B—C15B—C16B	2.8 (2)
O3A—C15A—C16A—C17A	176.75 (13)	O3B—C15B—C16B—C17B	178.31 (13)
C14A—C15A—C16A—C17A	−1.7 (2)	C14B—C15B—C16B—C17B	−0.9 (2)
C22A—O4A—C17A—C12A	175.92 (12)	C22B—O4B—C17B—C16B	1.3 (2)
C22A—O4A—C17A—C16A	−6.2 (2)	C22B—O4B—C17B—C12B	−176.93 (12)
C13A—C12A—C17A—O4A	−179.24 (12)	C15B—C16B—C17B—O4B	−179.75 (13)
C9A—C12A—C17A—O4A	5.36 (18)	C15B—C16B—C17B—C12B	−1.7 (2)
C13A—C12A—C17A—C16A	2.8 (2)	C13B—C12B—C17B—O4B	−179.59 (12)
C9A—C12A—C17A—C16A	−172.59 (13)	C9B—C12B—C17B—O4B	6.66 (18)
C15A—C16A—C17A—O4A	−179.09 (13)	C13B—C12B—C17B—C16B	2.2 (2)
C15A—C16A—C17A—C12A	−1.3 (2)	C9B—C12B—C17B—C16B	−171.56 (13)
C11A—O1A—C18A—C19A	173.84 (12)	C11B—O1B—C18B—C19B	−177.23 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1A—H1AA···N1A	0.93	2.45	2.7872 (19)	101
C1A—H1AA···O4B^{i}	0.93	2.60	3.4813 (18)	159
C8A—H8AA···O2B	0.93	2.44	3.3391 (16)	162
C1B—H1BA···N1B	0.93	2.43	2.7751 (19)	102
C8B—H8BA···O2A	0.93	2.39	3.2889 (16)	162
C18A—H18B···O4B^{i}	0.97	2.57	3.3360 (18)	136
C20A—H20B···O2A^{ii}	0.96	2.58	3.2869 (17)	131
C20B—H20E···O2B^{iii}	0.96	2.55	3.2169 (17)	126
C21A—H21B···O3B^{iv}	0.96	2.57	3.154 (2)	119
C22A—H22B···N2B^{iii}	0.96	2.58	3.479 (2)	155
C18B—H18D···Cg1^{iv}	0.97	2.93	3.7798 (16)	147
C20A—H20C···Cg3	0.96	2.60	3.5075 (15)	157
C20B—H20F···Cg2	0.96	2.51	3.3845 (15)	152

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) −*x*+2, −*y*, −*z*+1; (iii) −*x*+1, −*y*, −*z*+1; (iv) *x*, −*y*−1/2, *z*−1/2.

Fig. 1

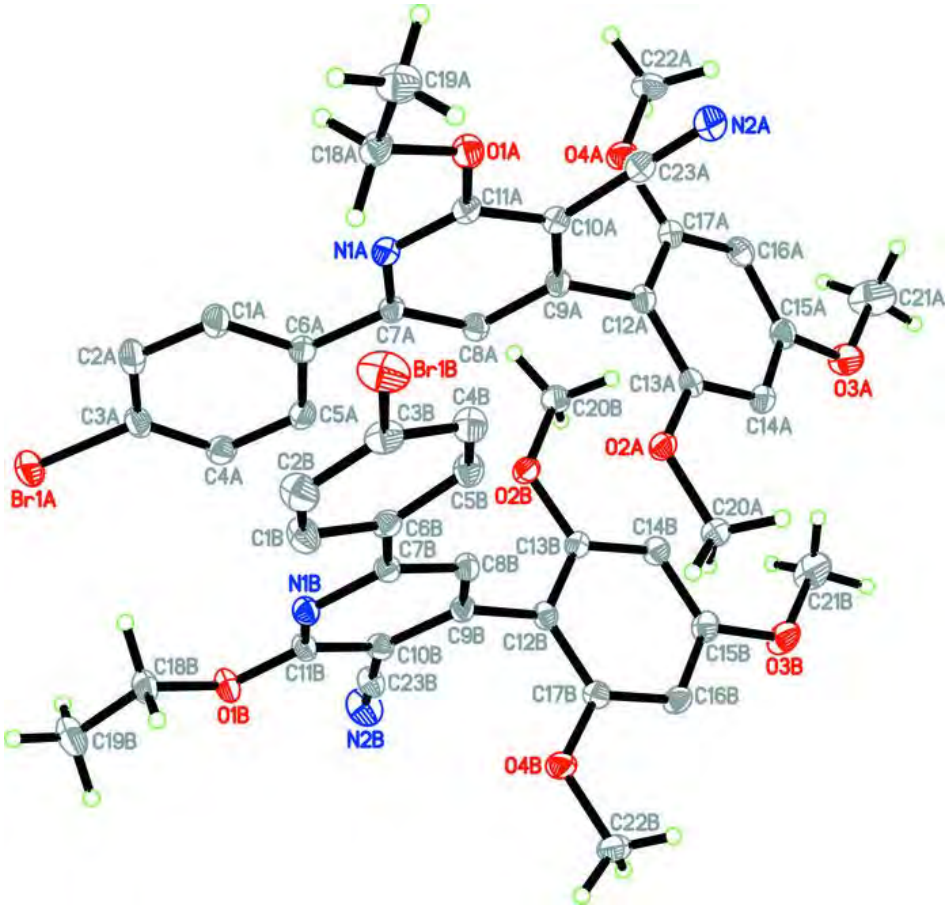
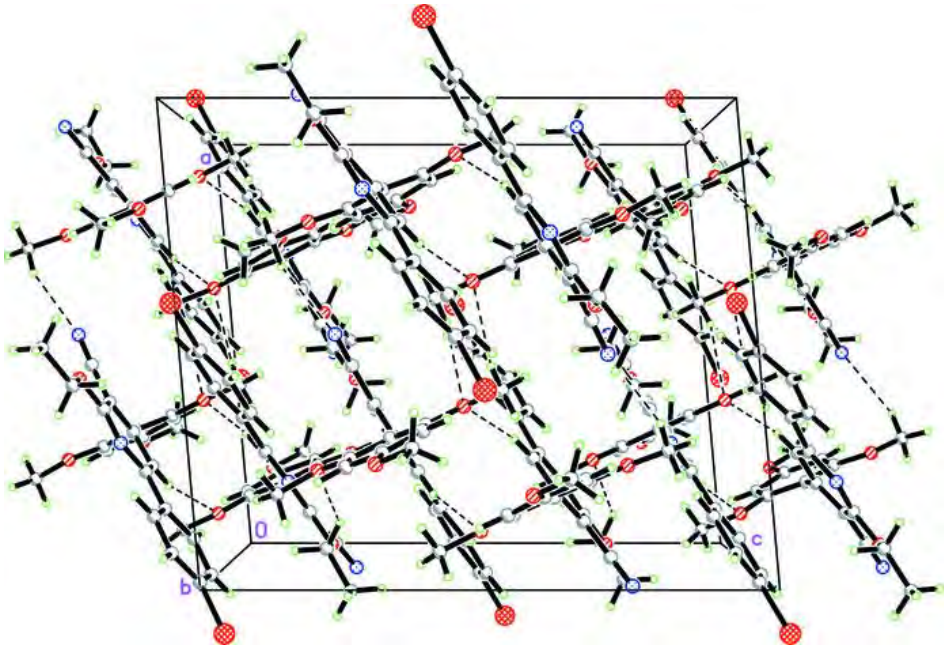


Fig. 2

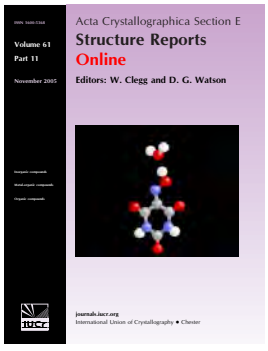


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

6-(4-Bromophenyl)-2-ethoxy-4-(4-ethoxyphenyl)nicotinonitrile

Suchada Chantrapromma, Hoong-Kun Fun, Thitipone Suwunwong, Mahesh Padaki and Arun M. Isloor

Acta Cryst. (2010). E**66**, o79–o80



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-(4-Bromophenyl)-2-ethoxy-4-(4-ethoxyphenyl)nicotinonitrile

Suchada Chantrapromma,[a]*‡ Hoong-Kun Fun,[b]§ Thitipone Suwunwong,[a] Mahesh Padaki[c] and Arun M. Isloor[c]

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [c]Department of Chemistry, National Institute of Technology-Karnataka, Surathkal, Mangalore 575 025, India
Correspondence e-mail: suchada.c@psu.ac.th

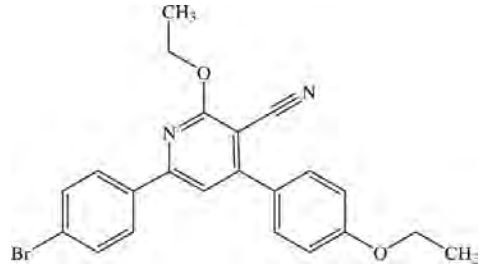
Received 12 November 2009; accepted 2 December 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.084; data-to-parameter ratio = 22.3.

The molecule of the title nicotinonitrile derivative, $C_{22}H_{19}BrN_2O_2$, is non-planar, the central pyridine ring making dihedral angles of 7.34 (14) and 43.56 (15)° with the 4-bromophenyl and 4-ethoxyphenyl rings, respectively. The ethoxy group of the 4-ethoxyphenyl is slightly twisted from the attached benzene ring [C—O—C—C = 174.2 (3)°], whereas the ethoxy group attached to the pyridine ring is in a (+)*syn*-clinal conformation [C—O—C—C = 83.0 (3)°]. A weak intramolecular C—H···N interaction generates an S(5) ring motif. In the crystal structure, the molecules are linked by weak intermolecular C—H···N interactions into screw chains along the b axis. These chains stacked along the a axis. π–π interactions with centroid–centroid distances of 3.8724 (16) and 3.8727 (16) Å are also observed.

Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the synthesis and applications of nicotinonitrile derivatives, see: Borgna *et al.* (1993); Fun *et al.* (2008); Goda *et al.* (2004); Kamal *et al.* (2007); Malinka *et al.* (1998). For related structures, see: Chantrapromma *et al.* (2009). For the stability of the temperature controller used in the data collection, see Cosier & Glazer (1986).



Experimental

Crystal data

$C_{22}H_{19}BrN_2O_2$
M_r = 423.29
Orthorhombic, $P2_12_12_1$
a = 4.3414 (2) Å
b = 14.7392 (6) Å
c = 29.4409 (13) Å
V = 1883.89 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.20 mm^{-1}
T = 100 K
0.57 × 0.05 × 0.03 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.368, T_{max} = 0.931
18389 measured reflections
5477 independent reflections
4081 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.084
S = 0.99
5477 reflections
246 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}
Absolute structure: Flack (1983), 2269 Friedel pairs
Flack parameter: 0.008 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···N1	0.93	2.41	2.758 (4)	102
C5—H5A···N2^{i}	0.93	2.58	3.446 (4)	156
C13—H13A···N2^{ii}	0.93	2.53	3.206 (4)	130

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for a research grant (RSA 5280033) and Prince of Songkla University for financial support. AMI is grateful to the Director, NITK-Surathkal, India, for providing research facilities and the Head of the Department of Chemistry and Dean R&D, NITK-Surathkal, for their encouragement. The authors also thank the Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012.

‡ Thomson Reuters ResearcherID: A-5085-2009.
§ Thomson Reuters ResearcherID: A-3561-2009. Additional correspondence author, e-mail: hkfun@usm.my.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2683).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Borgna, P., Pregnolato, M., Gamba, I. A. & Mellerio, G. (1993). *J. Heterocycl. Chem.* **30**, 1079–1084.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chantrapromma, S., Fun, H.-K., Suwunwong, T., Padaki, M. & Isloor, A. M. (2009). *Acta Cryst.* E**65**, o2914–o2915.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Fun, H.-K., Patil, P. S., Dharmaprakash, S. M. & Chantrapromma, S. (2008). *Acta Cryst.* E**64**, o1540–o1541.

Goda, F. E., Abdel-Aziz, A. A.-M. & Attef, O. A. (2004). *Bioorg. Med. Chem.* **12**, 1845–1852.

Kamal, A., Khan, M. N. A., Reddy, K. S. & Rohini, K. (2007). *Bioorg. Med. Chem.* **15**, 1004–1013.

Malinka, W., Ryng, S., Sieklucka-Dziuba, M., Rajtar, G., Głowniak, A. & Kleinrok, Z. (1998). *Farmaco*. **53**, 504–512.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2010). E**66**, o79-o80 [**doi:10.1107/S1600536809051861**]

6-(4-Bromophenyl)-2-ethoxy-4-(4-ethoxyphenyl)nicotinonitrile

S. Chantrapromma, H.-K. Fun, T. Suwunwong, M. Padaki and A. M. Isloor

Comment

A large number of substituted pyridines have been claimed to exhibit biological activities in a number of areas (Borgna *et al.*, 1993; Goda *et al.*, 2004; Kamal *et al.*, 2007; Malinka *et al.*, 1998). The pyridine ring is among the most common heterocyclic compounds found in the naturally occurring heterocycles and in various therapeutic agents. Our research is aimed at the synthesis and preliminary pharmacological screening (*in vivo*) of the nicotinonitrile derivatives. Therefore the title nicotinonitrile derivative, which is a substituted pyridine compound, was synthesized by cyclization of a chalcone derivative (Fun *et al.*, 2008) and malononitrile in order to investigate its analgesic and anti-inflammatory activities. Our results of these pharmacological studies showed that the title compound is a promising candidate for analgesic and anti-inflammatory activities. The analgesic and anti-inflammatory profiles of the title compound together with some other related nicotinonitrile derivatives will be reported elsewhere.

The title compound (I), $C_{22}H_{19}BrN_2O_2$ is a non-planar molecule (Fig. 1). The central pyridine ring is nearly coplanar with the 4-bromophenyl ring with the dihedral angle of 7.34 (14)° whereas it is inclined to the 4-ethoxyphenyl unit with the dihedral angle of 43.56 (15)°. The ethoxy substituent of the 4-ethoxyphenyl is slightly twisted from the mean plane of the attached benzene ring with the torsion angle C15–O2–C20–C21 = 174.2 (3)° whereas the ethoxy group attached to the pyridine ring is in a (+)*syn*-clinal conformation with a C11–O1–C18–C19 torsion angle of 83.0 (3)°. The orientation of the cyano group can be indicated by the torsion angle C8–C9–C10–C22 = 177.0 (3)°. A weak intramolecular C1—H1A···N1 interaction generates an S(5) ring motif (Bernstein *et al.*, 1995). The bond distances agree with the literature values (Allen *et al.*, 1987) and are comparable with those for a related structure (Chantrapromma *et al.*, 2009).

In the crystal structure (Fig. 2), the molecules are linked by weak intermolecular C—H···N interactions (Table 1) into screw chains along the *b* axis. These chains stacked along the *a* axis. The crystal is further stabilized by π···π interactions with the Cg_1···Cg_2 distances of 3.8724 (16) Å (symmetry code: -1 + *x*, *y*, *z*) and 3.8727 (16) Å (symmetry code: 1 + *x*, *y*, *z*); Cg_1 and Cg_2 are the centroids of C7–C11/N1 and C1–C6 rings, respectively.

Experimental

(*E*-1-(4-Bromophenyl)-3-(4-ethoxyphenyl)prop-2-en-1-one (0.50 g, 0.0015 mole) were added with continuous stirring to a freshly prepared sodium alkoxide (0.0014 mole of sodium in 100 ml of ethanol). Malononitrile (1.30 g, 0.02 mol) was then added with continuous stirring at room temperature until the precipitate separated out. The resulting solid was filtered (yield 65%). Colorless needle-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystalized from acetone/ethanol (1:1 *v*/*v*) by the slow evaporation of the solvent at room temperature over several days, Mp. 418–419 K.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C—H) = 0.93 Å for aromatic, 0.97 for CH_2 and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 1.07 Å from Br1 and the deepest hole is located at 0.96 Å from Br1.

Figures

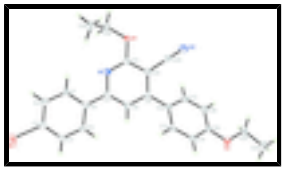


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

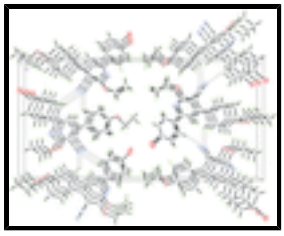


Fig. 2. The crystal packing of the title compound viewed along the *a* axis, showing chains stacked down the *a* axis. Hydrogen bonds are shown as dashed lines.

6-(4-Bromophenyl)-2-ethoxy-4-(4-ethoxyphenyl)nicotinonitrile

Crystal data

$C_{22}H_{19}BrN_2O_2$	D_x = 1.492 Mg m^{-3}
M_r = 423.29	Melting point = 418–419 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 5478 reflections
a = 4.3414 (2) Å	θ = 1.4–30.0°
b = 14.7392 (6) Å	μ = 2.20 mm^{-1}
c = 29.4409 (13) Å	*T* = 100 K
V = 1883.89 (14) Å^3	Needle, colourless
Z = 4	0.57 × 0.05 × 0.03 mm
F(000) = 864	

Data collection

Bruker APEXII CCD area detector diffractometer	5477 independent reflections
Radiation source: sealed tube	4081 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.076
φ and ω scans	θ_{max} = 30.0°, θ_{min} = 1.6°
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	*h* = −6→6

T_{min} = 0.368, T_{max} = 0.931	k = −20→20
18389 measured reflections	l = −41→41

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.084	$w = 1/[\sigma^2(F_o^2) + (0.0276P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.99	$(\Delta/\sigma)_{max}$ = 0.001
5477 reflections	$\Delta\rho_{max}$ = 0.58 e Å^{-3}
246 parameters	$\Delta\rho_{min}$ = −0.54 e Å^{-3}
0 restraints	Absolute structure: Flack (1983), 2269 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.008 (9)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	1.58148 (8)	0.282863 (19)	0.048491 (10)	0.02362 (8)
O1	0.4205 (6)	0.74788 (11)	0.14589 (6)	0.0216 (4)
O2	−0.0548 (6)	0.47728 (13)	0.40035 (6)	0.0252 (5)
N2	0.0125 (7)	0.76955 (17)	0.24500 (8)	0.0298 (7)
N1	0.7019 (6)	0.61534 (15)	0.14495 (8)	0.0196 (5)
C1	1.0999 (8)	0.50365 (18)	0.09800 (9)	0.0197 (6)
H1A	1.0551	0.5618	0.0877	0.024*
C2	1.2818 (8)	0.44749 (19)	0.07176 (10)	0.0214 (7)
H2A	1.3606	0.4679	0.0442	0.026*
C3	1.3459 (7)	0.36055 (18)	0.08677 (9)	0.0191 (7)
C4	1.2361 (8)	0.32993 (19)	0.12783 (10)	0.0220 (7)
H4A	1.2821	0.2716	0.1377	0.026*

C5	1.0564 (8)	0.38691 (17)	0.15427 (9)	0.0204 (6)
H5A	0.9838	0.3665	0.1822	0.025*
C6	0.9818 (7)	0.47489 (18)	0.13976 (9)	0.0185 (7)
C7	0.7817 (7)	0.53544 (18)	0.16580 (10)	0.0178 (6)
C8	0.6704 (7)	0.51430 (19)	0.20904 (10)	0.0195 (7)
H8A	0.7262	0.4597	0.2226	0.023*
C9	0.4766 (7)	0.57403 (18)	0.23214 (9)	0.0170 (6)
C10	0.3912 (8)	0.65388 (18)	0.20974 (9)	0.0189 (6)
C11	0.5132 (7)	0.67004 (18)	0.16607 (9)	0.0186 (7)
C12	0.3562 (8)	0.55145 (19)	0.27789 (9)	0.0189 (7)
C13	0.2478 (8)	0.46338 (18)	0.28616 (10)	0.0197 (7)
H13A	0.2680	0.4192	0.2638	0.024*
C14	0.1114 (9)	0.44126 (18)	0.32705 (9)	0.0200 (7)
H14A	0.0369	0.3829	0.3318	0.024*
C15	0.0855 (9)	0.50606 (18)	0.36103 (9)	0.0199 (6)
C16	0.1977 (8)	0.5937 (2)	0.35411 (10)	0.0230 (7)
H16A	0.1834	0.6371	0.3769	0.028*
C17	0.3314 (7)	0.61494 (19)	0.31246 (10)	0.0222 (7)
H17A	0.4061	0.6733	0.3077	0.027*
C18	0.5818 (8)	0.7761 (2)	0.10480 (8)	0.0230 (6)
H18A	0.7994	0.7626	0.1081	0.028*
H18B	0.5605	0.8412	0.1012	0.028*
C19	0.4616 (8)	0.7296 (2)	0.06258 (9)	0.0284 (7)
H19A	0.5543	0.7564	0.0362	0.043*
H19B	0.2420	0.7366	0.0610	0.043*
H19C	0.5123	0.6662	0.0637	0.043*
C20	−0.1371 (8)	0.5444 (2)	0.43323 (10)	0.0243 (8)
H20A	−0.2638	0.5911	0.4195	0.029*
H20B	0.0460	0.5726	0.4458	0.029*
C21	−0.3148 (8)	0.4952 (2)	0.46998 (11)	0.0306 (8)
H21A	−0.3648	0.5368	0.4939	0.046*
H21B	−0.1911	0.4467	0.4819	0.046*
H21C	−0.5011	0.4707	0.4574	0.046*
C22	0.1818 (7)	0.7186 (2)	0.22906 (9)	0.0210 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02481 (15)	0.02126 (12)	0.02480 (13)	0.00135 (15)	−0.00071 (16)	−0.00381 (13)
O1	0.0280 (11)	0.0181 (9)	0.0186 (9)	0.0039 (10)	0.0014 (12)	0.0043 (7)
O2	0.0376 (14)	0.0207 (9)	0.0173 (9)	0.0022 (11)	0.0044 (11)	−0.0004 (8)
N2	0.040 (2)	0.0236 (13)	0.0260 (12)	0.0087 (13)	−0.0018 (12)	0.0006 (10)
N1	0.0204 (14)	0.0189 (11)	0.0195 (12)	−0.0008 (11)	−0.0024 (11)	0.0008 (9)
C1	0.0211 (16)	0.0187 (12)	0.0192 (13)	0.0005 (15)	−0.0017 (15)	0.0004 (10)
C2	0.0186 (16)	0.0227 (15)	0.0229 (15)	−0.0008 (14)	−0.0008 (14)	0.0022 (12)
C3	0.0169 (18)	0.0196 (13)	0.0209 (14)	−0.0009 (12)	−0.0019 (13)	−0.0023 (11)
C4	0.0259 (18)	0.0154 (13)	0.0246 (16)	0.0012 (13)	−0.0041 (15)	0.0017 (11)
C5	0.0233 (17)	0.0196 (13)	0.0184 (13)	0.0003 (15)	−0.0004 (15)	0.0035 (10)

C6	0.0169 (18)	0.0186 (13)	0.0199 (14)	−0.0010 (12)	−0.0061 (13)	0.0020 (11)
C7	0.0172 (16)	0.0152 (13)	0.0210 (15)	−0.0022 (12)	−0.0055 (13)	0.0014 (11)
C8	0.0186 (19)	0.0190 (14)	0.0208 (15)	0.0006 (13)	−0.0027 (13)	0.0010 (12)
C9	0.0161 (17)	0.0181 (12)	0.0167 (13)	−0.0037 (12)	−0.0049 (12)	0.0021 (10)
C10	0.0219 (16)	0.0165 (12)	0.0182 (13)	0.0004 (14)	−0.0045 (14)	−0.0016 (10)
C11	0.0203 (19)	0.0141 (12)	0.0213 (13)	−0.0008 (12)	−0.0040 (12)	0.0016 (10)
C12	0.023 (2)	0.0186 (13)	0.0157 (14)	−0.0007 (13)	−0.0018 (13)	0.0008 (10)
C13	0.0243 (17)	0.0159 (13)	0.0191 (15)	0.0034 (13)	−0.0033 (14)	−0.0017 (11)
C14	0.0243 (18)	0.0157 (13)	0.0199 (14)	0.0017 (14)	−0.0019 (15)	0.0015 (10)
C15	0.0240 (16)	0.0215 (13)	0.0140 (13)	0.0030 (16)	0.0007 (15)	0.0007 (10)
C16	0.0288 (19)	0.0214 (14)	0.0188 (15)	0.0010 (14)	−0.0027 (15)	−0.0025 (12)
C17	0.0260 (19)	0.0169 (13)	0.0237 (15)	−0.0011 (13)	−0.0053 (14)	0.0001 (11)
C18	0.0266 (15)	0.0221 (13)	0.0204 (13)	−0.0010 (18)	0.0012 (15)	0.0036 (11)
C19	0.0302 (19)	0.0321 (16)	0.0230 (13)	0.0023 (17)	0.0012 (14)	0.0030 (12)
C20	0.029 (2)	0.0276 (15)	0.0165 (13)	0.0029 (15)	0.0022 (14)	−0.0077 (11)
C21	0.033 (2)	0.0360 (18)	0.0228 (16)	−0.0011 (17)	0.0033 (15)	−0.0058 (14)
C22	0.0264 (17)	0.0196 (12)	0.0171 (13)	−0.0023 (16)	−0.0042 (12)	0.0026 (13)

Geometric parameters (Å, °)

Br1—C3	1.905 (3)	C10—C11	1.411 (4)
O1—C11	1.353 (3)	C10—C22	1.435 (4)
O1—C18	1.458 (3)	C12—C17	1.387 (4)
O2—C15	1.375 (3)	C12—C13	1.402 (4)
O2—C20	1.429 (3)	C13—C14	1.380 (4)
N2—C22	1.151 (4)	C13—H13A	0.9300
N1—C11	1.307 (4)	C14—C15	1.388 (4)
N1—C7	1.372 (3)	C14—H14A	0.9300
C1—C2	1.380 (4)	C15—C16	1.395 (4)
C1—C6	1.398 (4)	C16—C17	1.392 (4)
C1—H1A	0.9300	C16—H16A	0.9300
C2—C3	1.384 (4)	C17—H17A	0.9300
C2—H2A	0.9300	C18—C19	1.512 (4)
C3—C4	1.376 (4)	C18—H18A	0.9700
C4—C5	1.386 (4)	C18—H18B	0.9700
C4—H4A	0.9300	C19—H19A	0.9600
C5—C6	1.403 (4)	C19—H19B	0.9600
C5—H5A	0.9300	C19—H19C	0.9600
C6—C7	1.462 (4)	C20—C21	1.514 (4)
C7—C8	1.397 (4)	C20—H20A	0.9700
C8—C9	1.395 (4)	C20—H20B	0.9700
C8—H8A	0.9300	C21—H21A	0.9600
C9—C10	1.399 (4)	C21—H21B	0.9600
C9—C12	1.483 (4)	C21—H21C	0.9600
C11—O1—C18	117.6 (2)	C14—C13—H13A	119.5
C15—O2—C20	117.9 (2)	C12—C13—H13A	119.5
C11—N1—C7	118.4 (3)	C13—C14—C15	120.1 (3)
C2—C1—C6	121.4 (3)	C13—C14—H14A	120.0
C2—C1—H1A	119.3	C15—C14—H14A	120.0

C6—C1—H1A	119.3	O2—C15—C14	115.5 (2)
C1—C2—C3	119.4 (3)	O2—C15—C16	124.3 (2)
C1—C2—H2A	120.3	C14—C15—C16	120.2 (3)
C3—C2—H2A	120.3	C17—C16—C15	118.8 (3)
C4—C3—C2	121.0 (3)	C17—C16—H16A	120.6
C4—C3—Br1	120.6 (2)	C15—C16—H16A	120.6
C2—C3—Br1	118.4 (2)	C12—C17—C16	121.8 (3)
C3—C4—C5	119.3 (3)	C12—C17—H17A	119.1
C3—C4—H4A	120.3	C16—C17—H17A	119.1
C5—C4—H4A	120.3	O1—C18—C19	112.8 (3)
C4—C5—C6	121.3 (3)	O1—C18—H18A	109.0
C4—C5—H5A	119.4	C19—C18—H18A	109.0
C6—C5—H5A	119.4	O1—C18—H18B	109.0
C1—C6—C5	117.6 (3)	C19—C18—H18B	109.0
C1—C6—C7	119.6 (2)	H18A—C18—H18B	107.8
C5—C6—C7	122.8 (3)	C18—C19—H19A	109.5
N1—C7—C8	120.8 (3)	C18—C19—H19B	109.5
N1—C7—C6	116.1 (3)	H19A—C19—H19B	109.5
C8—C7—C6	123.2 (2)	C18—C19—H19C	109.5
C9—C8—C7	120.8 (3)	H19A—C19—H19C	109.5
C9—C8—H8A	119.6	H19B—C19—H19C	109.5
C7—C8—H8A	119.6	O2—C20—C21	106.2 (2)
C8—C9—C10	117.5 (3)	O2—C20—H20A	110.5
C8—C9—C12	120.9 (3)	C21—C20—H20A	110.5
C10—C9—C12	121.6 (3)	O2—C20—H20B	110.5
C9—C10—C11	118.2 (3)	C21—C20—H20B	110.5
C9—C10—C22	122.7 (3)	H20A—C20—H20B	108.7
C11—C10—C22	119.1 (2)	C20—C21—H21A	109.5
N1—C11—O1	120.1 (2)	C20—C21—H21B	109.5
N1—C11—C10	124.4 (3)	H21A—C21—H21B	109.5
O1—C11—C10	115.6 (2)	C20—C21—H21C	109.5
C17—C12—C13	118.1 (3)	H21A—C21—H21C	109.5
C17—C12—C9	122.9 (2)	H21B—C21—H21C	109.5
C13—C12—C9	118.9 (2)	N2—C22—C10	179.0 (3)
C14—C13—C12	120.9 (3)		

C6—C1—C2—C3	0.8 (5)	C7—N1—C11—C10	1.9 (4)
C1—C2—C3—C4	−1.3 (5)	C18—O1—C11—N1	−11.6 (4)
C1—C2—C3—Br1	177.1 (2)	C18—O1—C11—C10	168.9 (3)
C2—C3—C4—C5	0.6 (5)	C9—C10—C11—N1	−0.1 (5)
Br1—C3—C4—C5	−177.8 (2)	C22—C10—C11—N1	−179.0 (3)
C3—C4—C5—C6	0.6 (5)	C9—C10—C11—O1	179.4 (3)
C2—C1—C6—C5	0.4 (5)	C22—C10—C11—O1	0.5 (4)
C2—C1—C6—C7	−178.0 (3)	C8—C9—C12—C17	140.0 (3)
C4—C5—C6—C1	−1.1 (5)	C10—C9—C12—C17	−42.6 (5)
C4—C5—C6—C7	177.2 (3)	C8—C9—C12—C13	−43.3 (4)
C11—N1—C7—C8	−1.7 (4)	C10—C9—C12—C13	134.0 (3)
C11—N1—C7—C6	177.2 (3)	C17—C12—C13—C14	2.0 (5)
C1—C6—C7—N1	5.4 (4)	C9—C12—C13—C14	−174.8 (3)
C5—C6—C7—N1	−173.0 (3)	C12—C13—C14—C15	−1.3 (5)

C1—C6—C7—C8	−175.7 (3)	C20—O2—C15—C14	−169.1 (3)
C5—C6—C7—C8	5.9 (5)	C20—O2—C15—C16	10.8 (5)
N1—C7—C8—C9	−0.3 (4)	C13—C14—C15—O2	179.8 (3)
C6—C7—C8—C9	−179.1 (3)	C13—C14—C15—C16	−0.2 (5)
C7—C8—C9—C10	2.0 (4)	O2—C15—C16—C17	−179.1 (3)
C7—C8—C9—C12	179.5 (3)	C14—C15—C16—C17	0.9 (5)
C8—C9—C10—C11	−1.9 (4)	C13—C12—C17—C16	−1.3 (5)
C12—C9—C10—C11	−179.3 (3)	C9—C12—C17—C16	175.4 (3)
C8—C9—C10—C22	177.0 (3)	C15—C16—C17—C12	−0.1 (5)
C12—C9—C10—C22	−0.4 (5)	C11—O1—C18—C19	83.0 (3)
C7—N1—C11—O1	−177.6 (3)	C15—O2—C20—C21	174.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1A···N1	0.93	2.41	2.758 (4)	102
C5—H5A···N2^{i}	0.93	2.58	3.446 (4)	156
C13—H13A···N2^{ii}	0.93	2.53	3.206 (4)	130

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) −*x*, *y*−1/2, −*z*+1/2.

Fig. 1

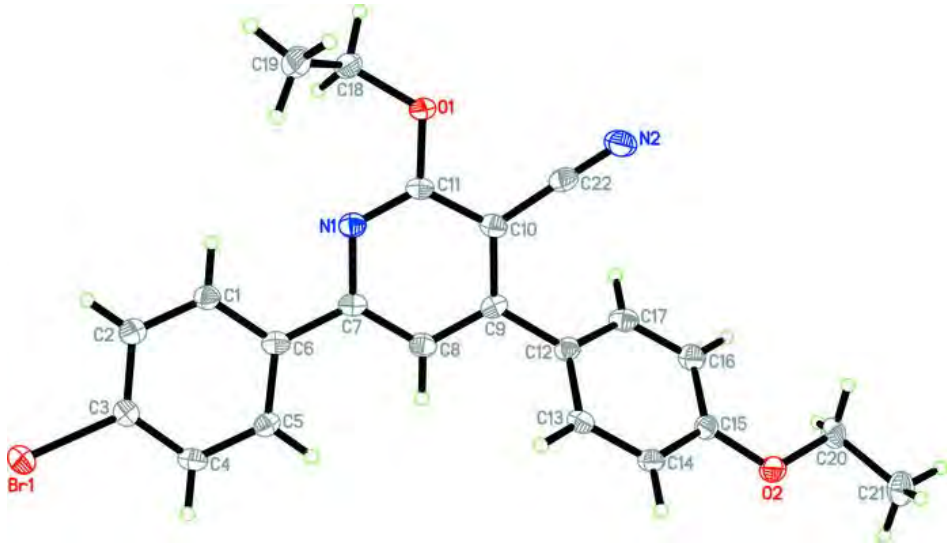
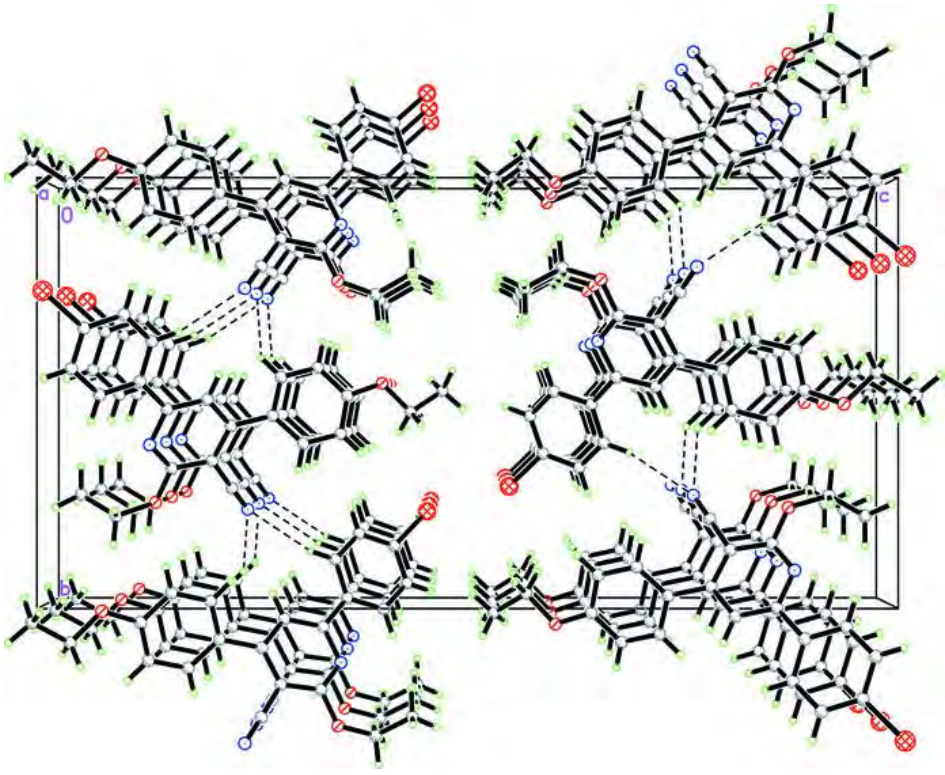


Fig. 2

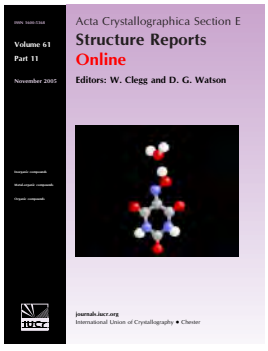


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

A second orthorhombic polymorph of (*Z*)-3-(9-anthryl)-1-(2-thienyl)prop-2-en-1-one

Suchada Chantrapromma, Thitipone Suwunwong, Nawong Boonnak and Hoong-Kun Fun

Acta Cryst. (2010). E**66**, o312–o313



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A second orthorhombic polymorph of (*Z*)-3-(9-anthryl)-1-(2-thienyl)prop-2-en-1-one[1]

Suchada Chantrapromma,[a]*§ Thitipone Suwunwong,[a] Nawong Boonnak[a] and Hoong-Kun Fun[b]¶

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

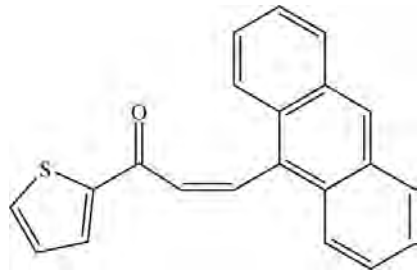
Received 19 December 2009; accepted 1 January 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.108; data-to-parameter ratio = 17.1.

The title heteroaryl chalcone, $C_{21}H_{14}OS$, is a second orthorhombic polymorph which crystallizes in the space group $P2_12_12_1$. The structure was previously reported [Fun *et al.* (2009). *Acta Cryst.* E**65**, o2168-o2169] in the space group $Pna2_1$. The bond distances and angles are similar in both structures. In contrast, the overall crystal packing is different from that in the first orthorhombic $Pna2_1$ polymorph in which molecules were stacked into columns along the *b* axis and the thiophene units of two adjacent columns were stacked in a head to tail fashion. In the present polymorph, molecules are found to dimerize through a weak S···S interaction [3.6513 (7) Å] and these dimers are arranged into sheets parallel to the *bc* plane. There are no classical hydrogen bonds in the packing which features short C···O [3.2832 (2)–3.6251 (9) Å], C···S [3.4879 (17)–3.6251 (19) Å] and S···O [2.9948 (16) Å] contacts, together with C—H···π interactions. Similar contacts were found in the other polymorph.

Related literature

For bond-length data, see: Allen *et al.* (1987). For the structure of the first polymorph, see: Fun *et al.* (2009). For background to and applications of chalcones, see: Chantrapromma *et al.* (2009); Patil & Dharmaprakash (2008); Saydam *et al.* (2003); Suwunwong *et al.* (2009); Svetlichny *et al.* (2007). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{21}H_{14}OS$
M_r = 314.38
Orthorhombic, $P2_12_12_1$
a = 5.5116 (1) Å
b = 14.8497 (2) Å
c = 18.3625 (3) Å
V = 1502.89 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 100 K
0.50 × 0.19 × 0.11 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.900, T_{max} = 0.977
14062 measured reflections
4354 independent reflections
4035 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.108
S = 1.05
4354 reflections
254 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}
Absolute structure: Flack (1983), 1830 Friedel pairs
Flack parameter: 0.04 (8)

Table 1
Hydrogen-bond geometry (Å, °).

Cg_1, Cg_2 and Cg_3 are the centroids of the S1/C18–C21, C1–C6 and C8–C13 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···$Cg1^{i}$	0.91 (3)	2.64 (3)	3.443 (2)	149 (2)
C15—H15A···$Cg2^{ii}$	0.95 (2)	2.74 (2)	3.565 (2)	146.4 (17)
C21—H21A···$Cg3^{iii}$	1.04 (3)	2.91 (3)	3.711 (2)	134.4 (19)

Symmetry codes: (i) $x+\frac{3}{2}, -y-\frac{1}{2}, -z+1$; (ii) $x+1, y, z$; (iii) $-x, y+\frac{3}{2}, -z+\frac{5}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for research grant (RSA 5280033) and the Prince of Songkla University for financial support through the Crystal Materials Research Unit. The authors also thank Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012.

[1] This paper is dedicated to His Majesty King Bhumibol Adulyadej of Thailand (King Rama IX) for his sustainable development of the country.
§ Thomson Reuters ResearcherID: A-5085-2009.
¶ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2716).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chantrapromma, S., Suwunwong, T., Karalai, C. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o893–o894.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Fun, H.-K., Suwunwong, T., Boonnak, N. & Chantrapromma, S. (2009). *Acta Cryst.* E**65**, o2168–o2169.

Patil, P. S. & Dharmaprakash, S. M. (2008). *Mater. Lett.* **62**, 451–453.

Saydam, G., Aydin, H. H., Sahin, F., Kucukoglu, O., Erciyas, E., Terzioglu, E., Buyukkececi, F. & Omay, S. B. (2003). *Leuk. Res*. **27**, 57–64.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Suwunwong, T., Chantrapromma, S., Karalai, C., Pakdeevanich, P. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o420–o421.

Svetlichny, V. Y., Merola, F., Dobretsov, G. E., Gularyan, S. K. & Syrejshchikova, T. I. (2007). *Chem. Phys. Lipids*, **145**, 13–26.

supplementary materials

Acta Cryst. (2010). E**66**, o312-o313 [**doi:10.1107/S1600536810000061**]

A second orthorhombic polymorph of (*Z*)-3-(9-anthryl)-1-(2-thienyl)prop-2-en-1-one

S. Chantrapromma, T. Suwunwong, N. Boonnak and H.-K. Fun

Comment

In continuation of our study of chalcone derivatives (Chantrapromma *et al.*, 2009; Fun *et al.*, 2009; Suwunwong *et al.*, 2009) which can be used for non-linear optical (NLO) materials (Patil & Dharmaprakash, 2008), fluorescent materials (Svetlichny *et al.*, 2007) and bioactive compounds (Saydam *et al.*, 2003), the title heteroaryl chalcone (I) was synthesized and its crystal structure was previously reported in the orthorhombic space group $Pna2_1$ (Fun *et al.*, 2009). In the present work, the compound crystallized in the orthorhombic space group $P2_12_12_1$ from an ethanol/acetone (1:1) solvent mixture, while the crystal of the $Pna2_1$ form crystallized from hot ethanol. (I) exhibits fluorescence with the maximum emission at 402 nm when the compound is excited at 335 nm.

The molecule of (I) (Fig. 1) exists in an *Z* configuration with respect to the C15═C16 double bond and the torsion angle C14–C15–C16–C17 = -2.9 (3)° [compared to -3.7 (7)° in one molecule and -4.0 (7)° in the other in the $Pna2_1$ polymorph which contains two molecules in an asymmetric unit]. The molecule of the present polymorph is less twisted as indicated by the interplanar angles between thiophene and anthracene rings being 56.36 (7)° and the least squares plane through the prop-2-en-1-one unit (C15–C17/O1) makes interplanar angles of 12.2 and 68.00 (11)° with the thiophene and anthracene rings, respectively [the corresponding values are 75.07 (17), 13.1 (3) and 71.2 (3)° in one molecule and 76.32 (17), 15.2 (3) and 72.3 (3)° in the other for the $Pna2_1$ polymorph]. Bond distances are within normal ranges (Allen *et al.*, 1987).

In the crystal packing (Fig. 2), molecules are found to dimerize through a non-bonding S···S interaction [S···S = 3.6513 (7) Å]. The dimers are arranged into sheets parallel to the *bc* plane. These sheets are stacked along the *a* axis. The intermolecular interactions and short contacts are almost similar in both polymorph. There is no classic hydrogen bond and the crystal is consolidated by short C···O [3.2832 (2)–3.6251 (9) Å], C···S [3.4879 (17)–3.6251 (19) Å] and S···O [2.9948 (16) Å] contacts, as well as C—H···π interactions (Table 1); Cg_1, Cg_2 and Cg_3 are the centroids of the S1/C18–C21, C1–C6 and C8–C13 rings, respectively.

Experimental

The title compound was synthesized as reported by Fun *et al.* (2009). Yellow block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystallized from ethanol/acetone (1:1 *v*/*v*) by slow evaporation of the solvent at room temperature over several days, Mp. 400–401 K.

Refinement

The H atom attached to C19 was placed in a calculated position, with d(C—H) = 0.93 Å, $U_{iso} = 1.2U_{eq}$(C). The remaining H atoms were located from the difference maps and refined isotropically. The highest residual electron density peak is located at 0.04 Å from C19 and the deepest hole is 0.07 Å from C20. A total of 1830 Friedel pairs were used to determine the absolute configuration.

Figures

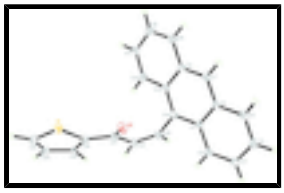


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

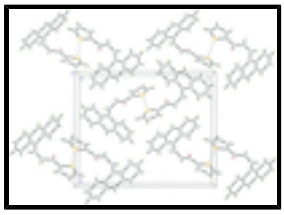


Fig. 2. The crystal packing of the title compound viewed along the *a* axis. S···S contacts are shown as dashed lines.

(*Z*)-3-(9-anthryl)-1-(2-thienyl)prop-2-en-1-one

Crystal data

$C_{21}H_{14}OS$	D_x = 1.390 Mg m^{-3}
M_r = 314.38	Melting point = 400–401 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 4354 reflections
a = 5.5116 (1) Å	θ = 1.8–30.0°
b = 14.8497 (2) Å	μ = 0.22 mm^{-1}
c = 18.3625 (3) Å	*T* = 100 K
V = 1502.89 (4) Å^3	Block, yellow
Z = 4	0.50 × 0.19 × 0.11 mm
F(000) = 656	

Data collection

Bruker APEXII CCD area-detector diffractometer	4354 independent reflections
Radiation source: sealed tube	4035 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.025
φ and ω scans	θ_{max} = 30.0°, θ_{min} = 1.8°
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	*h* = −7→5
T_{min} = 0.900, T_{max} = 0.977	*k* = −17→20
14062 measured reflections	*l* = −25→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.042$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.108$	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.8977P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
4354 reflections	$\Delta\rho_{max} = 0.79$ e Å^{-3}
254 parameters	$\Delta\rho_{min} = -0.62$ e Å^{-3}
0 restraints	Absolute structure: Flack (1983), 1830 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.04 (8)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.79023 (9)	0.82740 (3)	0.98167 (3)	0.01920 (11)
O1	0.6379 (3)	0.74101 (10)	0.84157 (8)	0.0208 (3)
C1	0.6215 (3)	0.62511 (12)	0.66251 (10)	0.0142 (3)
C2	0.6199 (4)	0.69201 (13)	0.60656 (10)	0.0176 (4)
H2A	0.747 (4)	0.7404 (15)	0.6071 (12)	0.019 (6)*
C3	0.4549 (4)	0.68828 (13)	0.55096 (10)	0.0193 (4)
H3A	0.459 (4)	0.7304 (16)	0.5132 (14)	0.022 (6)*
C4	0.2761 (4)	0.61930 (13)	0.54827 (11)	0.0198 (4)
H4A	0.150 (5)	0.6169 (17)	0.5090 (13)	0.026 (6)*
C5	0.2685 (4)	0.55527 (13)	0.60124 (10)	0.0184 (4)
H5A	0.155 (5)	0.5111 (18)	0.6021 (14)	0.033 (7)*
C6	0.4394 (3)	0.55565 (12)	0.65992 (10)	0.0146 (3)
C7	0.4332 (4)	0.49043 (12)	0.71450 (10)	0.0169 (4)
H7A	0.311 (4)	0.4456 (14)	0.7129 (11)	0.010 (5)*
C8	0.6064 (4)	0.48855 (12)	0.76949 (10)	0.0159 (4)
C9	0.6059 (4)	0.41897 (13)	0.82390 (11)	0.0213 (4)
H9A	0.479 (4)	0.3744 (16)	0.8218 (13)	0.019 (6)*
C10	0.7811 (4)	0.41528 (14)	0.87607 (11)	0.0240 (4)
H10A	0.788 (5)	0.3656 (16)	0.9099 (13)	0.022 (6)*
C11	0.9682 (4)	0.48106 (15)	0.87753 (11)	0.0222 (4)

H11A	1.103 (5)	0.4744 (16)	0.9123 (13)	0.023 (6)*
C12	0.9731 (4)	0.54951 (14)	0.82813 (10)	0.0189 (4)
H12A	1.091 (5)	0.5966 (17)	0.8299 (13)	0.027 (7)*
C13	0.7917 (4)	0.55659 (12)	0.77254 (9)	0.0156 (3)
C14	0.7917 (3)	0.62627 (12)	0.72009 (9)	0.0143 (3)
C15	0.9746 (4)	0.69969 (13)	0.72208 (10)	0.0172 (4)
H15A	1.077 (4)	0.7024 (15)	0.6807 (12)	0.016 (6)*
C16	1.0028 (4)	0.76158 (13)	0.77411 (10)	0.0178 (4)
H16A	1.138 (5)	0.8035 (17)	0.7711 (13)	0.026 (6)*
C17	0.8452 (3)	0.77098 (12)	0.83955 (10)	0.0153 (3)
C18	0.9491 (3)	0.82083 (12)	0.90132 (9)	0.0146 (3)
C19	1.1727 (3)	0.86783 (12)	0.90518 (10)	0.0146 (2)
H19A	1.2863	0.8714	0.8678	0.018*
C20	1.1971 (3)	0.90888 (11)	0.97520 (10)	0.0146 (2)
H20A	1.351 (5)	0.9445 (17)	0.9876 (14)	0.031 (7)*
C21	1.0083 (4)	0.89174 (13)	1.02086 (11)	0.0198 (4)
H21A	0.993 (5)	0.9095 (18)	1.0753 (15)	0.034 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0185 (2)	0.0204 (2)	0.0188 (2)	−0.00094 (17)	0.00214 (18)	−0.00059 (18)
O1	0.0163 (6)	0.0219 (7)	0.0243 (7)	−0.0038 (6)	0.0037 (6)	−0.0053 (6)
C1	0.0159 (8)	0.0132 (8)	0.0136 (8)	0.0006 (7)	0.0018 (6)	−0.0028 (6)
C2	0.0200 (9)	0.0167 (8)	0.0162 (8)	0.0000 (7)	0.0013 (7)	−0.0014 (7)
C3	0.0234 (9)	0.0181 (9)	0.0163 (8)	0.0033 (7)	−0.0002 (7)	0.0009 (7)
C4	0.0211 (9)	0.0210 (9)	0.0174 (8)	0.0038 (8)	−0.0038 (8)	−0.0049 (7)
C5	0.0182 (9)	0.0167 (8)	0.0204 (8)	0.0001 (7)	−0.0007 (8)	−0.0057 (7)
C6	0.0140 (8)	0.0143 (8)	0.0155 (8)	0.0007 (7)	−0.0001 (7)	−0.0056 (7)
C7	0.0187 (9)	0.0131 (8)	0.0188 (8)	−0.0010 (7)	0.0030 (7)	−0.0038 (7)
C8	0.0192 (9)	0.0142 (8)	0.0143 (8)	0.0023 (7)	0.0050 (7)	−0.0007 (7)
C9	0.0279 (11)	0.0152 (9)	0.0208 (9)	0.0012 (8)	0.0065 (8)	0.0002 (7)
C10	0.0325 (11)	0.0215 (9)	0.0179 (8)	0.0078 (9)	0.0061 (9)	0.0032 (7)
C11	0.0254 (10)	0.0262 (10)	0.0149 (8)	0.0088 (9)	0.0012 (8)	0.0007 (8)
C12	0.0182 (9)	0.0219 (9)	0.0166 (8)	0.0029 (8)	0.0016 (7)	−0.0013 (7)
C13	0.0164 (8)	0.0170 (8)	0.0133 (7)	0.0038 (7)	0.0036 (7)	−0.0019 (6)
C14	0.0146 (7)	0.0144 (8)	0.0141 (7)	0.0014 (7)	0.0019 (7)	−0.0028 (6)
C15	0.0163 (8)	0.0213 (9)	0.0141 (8)	−0.0014 (7)	0.0026 (7)	0.0002 (7)
C16	0.0170 (9)	0.0198 (9)	0.0165 (8)	−0.0033 (7)	0.0013 (7)	0.0000 (7)
C17	0.0173 (9)	0.0118 (8)	0.0167 (8)	−0.0001 (7)	0.0011 (7)	−0.0010 (6)
C18	0.0161 (8)	0.0151 (8)	0.0125 (7)	0.0021 (7)	0.0022 (6)	0.0003 (7)
C19	0.0125 (5)	0.0149 (5)	0.0164 (6)	0.0005 (5)	−0.0024 (5)	−0.0019 (5)
C20	0.0125 (5)	0.0149 (5)	0.0164 (6)	0.0005 (5)	−0.0024 (5)	−0.0019 (5)
C21	0.0243 (9)	0.0190 (8)	0.0161 (8)	0.0040 (7)	−0.0012 (8)	−0.0017 (7)

Geometric parameters (Å, °)

S1—C21	1.696 (2)	C9—H9A	0.96 (3)
S1—C18	1.7184 (18)	C10—C11	1.421 (3)

O1—C17	1.227 (2)	C10—H10A	0.97 (2)
C1—C14	1.414 (3)	C11—C12	1.363 (3)
C1—C2	1.429 (3)	C11—H11A	0.98 (3)
C1—C6	1.440 (3)	C12—C13	1.433 (3)
C2—C3	1.368 (3)	C12—H12A	0.96 (3)
C2—H2A	1.00 (2)	C13—C14	1.414 (2)
C3—C4	1.422 (3)	C14—C15	1.485 (3)
C3—H3A	0.93 (2)	C15—C16	1.335 (3)
C4—C5	1.361 (3)	C15—H15A	0.95 (2)
C4—H4A	1.00 (3)	C16—C17	1.489 (3)
C5—C6	1.431 (3)	C16—H16A	0.97 (3)
C5—H5A	0.91 (3)	C17—C18	1.470 (3)
C6—C7	1.394 (3)	C18—C19	1.418 (3)
C7—C8	1.390 (3)	C19—C20	1.429 (2)
C7—H7A	0.95 (2)	C19—H19A	0.9300
C8—C9	1.437 (3)	C20—C21	1.360 (3)
C8—C13	1.438 (3)	C20—H20A	1.02 (3)
C9—C10	1.361 (3)	C21—H21A	1.04 (3)
C21—S1—C18	92.02 (10)	C12—C11—H11A	119.5 (15)
C14—C1—C2	122.21 (17)	C10—C11—H11A	119.4 (14)
C14—C1—C6	119.73 (17)	C11—C12—C13	121.0 (2)
C2—C1—C6	118.05 (17)	C11—C12—H12A	122.4 (16)
C3—C2—C1	120.81 (18)	C13—C12—H12A	116.5 (16)
C3—C2—H2A	120.0 (13)	C14—C13—C12	122.62 (18)
C1—C2—H2A	119.1 (13)	C14—C13—C8	119.20 (17)
C2—C3—C4	121.04 (18)	C12—C13—C8	118.16 (17)
C2—C3—H3A	120.8 (15)	C13—C14—C1	120.04 (17)
C4—C3—H3A	118.2 (15)	C13—C14—C15	121.37 (16)
C5—C4—C3	119.99 (18)	C1—C14—C15	118.55 (16)
C5—C4—H4A	118.0 (15)	C16—C15—C14	127.01 (17)
C3—C4—H4A	122.0 (15)	C16—C15—H15A	118.3 (14)
C4—C5—C6	120.99 (19)	C14—C15—H15A	114.7 (14)
C4—C5—H5A	122.6 (17)	C15—C16—C17	125.05 (17)
C6—C5—H5A	116.4 (17)	C15—C16—H16A	119.3 (15)
C7—C6—C5	121.47 (17)	C17—C16—H16A	115.6 (15)
C7—C6—C1	119.43 (17)	O1—C17—C18	121.46 (17)
C5—C6—C1	119.10 (17)	O1—C17—C16	122.25 (17)
C8—C7—C6	121.27 (17)	C18—C17—C16	116.26 (16)
C8—C7—H7A	119.8 (13)	C19—C18—C17	128.65 (16)
C6—C7—H7A	118.9 (13)	C19—C18—S1	111.83 (13)
C7—C8—C9	121.21 (18)	C17—C18—S1	119.51 (14)
C7—C8—C13	120.14 (17)	C18—C19—C20	109.67 (16)
C9—C8—C13	118.65 (18)	C18—C19—H19A	125.2
C10—C9—C8	121.1 (2)	C20—C19—H19A	125.2
C10—C9—H9A	121.0 (14)	C21—C20—C19	113.75 (16)
C8—C9—H9A	117.8 (14)	C21—C20—H20A	126.4 (15)
C9—C10—C11	120.04 (18)	C19—C20—H20A	119.8 (15)
C9—C10—H10A	120.8 (16)	C20—C21—S1	112.71 (15)
C11—C10—H10A	119.0 (16)	C20—C21—H21A	127.5 (16)

C12—C11—C10	121.0 (2)	S1—C21—H21A	119.7 (16)
C14—C1—C2—C3	179.06 (17)	C9—C8—C13—C12	2.5 (3)
C6—C1—C2—C3	−2.1 (3)	C12—C13—C14—C1	174.11 (17)
C1—C2—C3—C4	1.7 (3)	C8—C13—C14—C1	−4.4 (3)
C2—C3—C4—C5	−0.5 (3)	C12—C13—C14—C15	−3.3 (3)
C3—C4—C5—C6	−0.4 (3)	C8—C13—C14—C15	178.18 (16)
C4—C5—C6—C7	179.95 (18)	C2—C1—C14—C13	−177.53 (17)
C4—C5—C6—C1	0.0 (3)	C6—C1—C14—C13	3.6 (3)
C14—C1—C6—C7	0.2 (3)	C2—C1—C14—C15	0.0 (3)
C2—C1—C6—C7	−178.75 (17)	C6—C1—C14—C15	−178.90 (16)
C14—C1—C6—C5	−179.92 (16)	C13—C14—C15—C16	−64.2 (3)
C2—C1—C6—C5	1.2 (3)	C1—C14—C15—C16	118.4 (2)
C5—C6—C7—C8	176.94 (17)	C14—C15—C16—C17	−2.9 (3)
C1—C6—C7—C8	−3.1 (3)	C15—C16—C17—O1	−21.8 (3)
C6—C7—C8—C9	−177.26 (17)	C15—C16—C17—C18	160.02 (19)
C6—C7—C8—C13	2.3 (3)	O1—C17—C18—C19	−172.54 (18)
C7—C8—C9—C10	177.47 (19)	C16—C17—C18—C19	5.7 (3)
C13—C8—C9—C10	−2.1 (3)	O1—C17—C18—S1	6.9 (2)
C8—C9—C10—C11	0.2 (3)	C16—C17—C18—S1	−174.86 (13)
C9—C10—C11—C12	1.3 (3)	C21—S1—C18—C19	0.62 (15)
C10—C11—C12—C13	−0.9 (3)	C21—S1—C18—C17	−178.93 (15)
C11—C12—C13—C14	−179.56 (18)	C17—C18—C19—C20	178.21 (17)
C11—C12—C13—C8	−1.0 (3)	S1—C18—C19—C20	−1.30 (19)
C7—C8—C13—C14	1.5 (3)	C18—C19—C20—C21	1.5 (2)
C9—C8—C13—C14	−178.92 (17)	C19—C20—C21—S1	−1.1 (2)
C7—C8—C13—C12	−177.10 (17)	C18—S1—C21—C20	0.27 (15)

Hydrogen-bond geometry (Å, °)

Cg_1, Cg_2 and Cg_3 are the centroids of the S1/C18–C21, C1–C6 and C8–C13 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5A···Cg1^{i}	0.91 (3)	2.64 (3)	3.443 (2)	149 (2)
C15—H15A···Cg2^{ii}	0.95 (2)	2.74 (2)	3.565 (2)	146.4 (17)
C21—H21A···Cg3^{iii}	1.04 (3)	2.91 (3)	3.711 (2)	134.4 (19)

Symmetry codes: (i) *x*+3/2, −*y*−1/2, −*z*+1; (ii) *x*+1, *y*, *z*; (iii) −*x*, *y*+3/2, −*z*+5/2.

Fig. 1

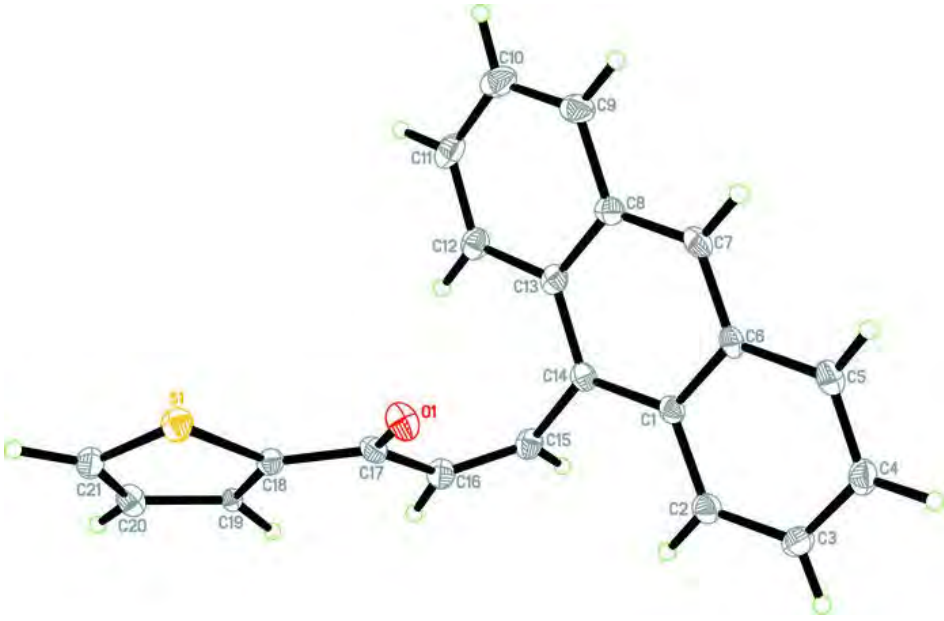
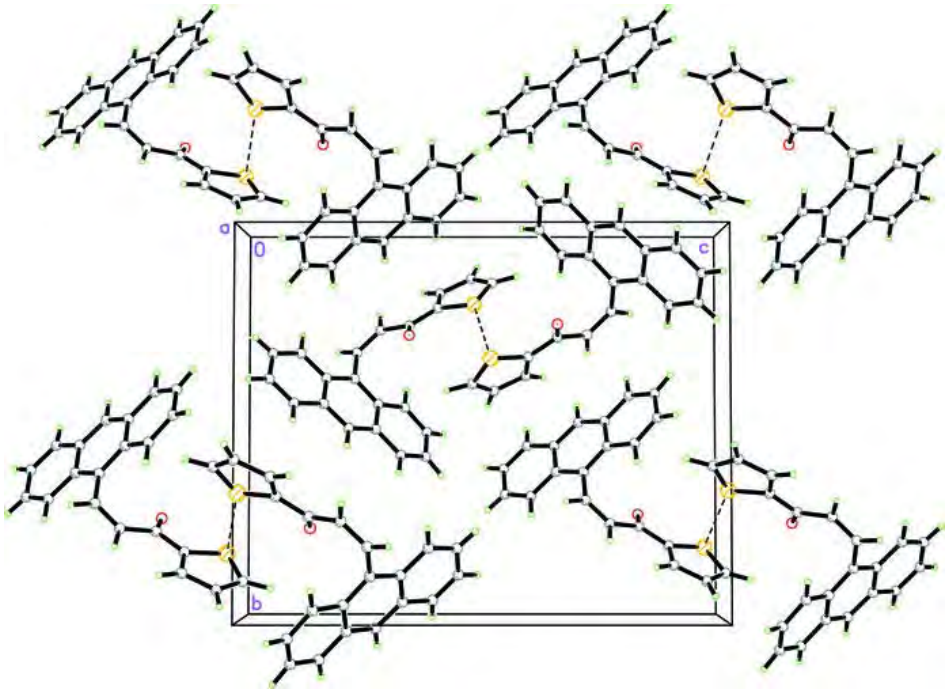


Fig. 2



Full Paper

CSIRO PUBLISHING

Aust. J. Chem. **2010**, *63*, 1550–1556

www.publish.csiro.au/journals/ajc

Nitric Oxide Inhibitory Activity of Xanthones from the Green Fruits of *Cratoxylum formosum* ssp. *pruniflorum*

Nawong Boonnak,[A,B] *Achjana Khamthip,*[A] *Chatchanok Karalai,*[A,B,H] *Suchada Chantrapromma,*[A] *Chanita Ponglimanont,*[B] *Akkharawit Kanjana-Opas,*[C] *Supinya Tewtrakul,*[D] *Kan Chantrapromma,*[E] *Hoong-Kun Fun,*[E] *and Shigeru Kato*[G]

[A]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand.

[B]Department of Chemistry and Center for Innovation in Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand.

[C]Department of Industrial Biotechnology, Faculty of Agro-Industry, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand.

[D]Department of Pharmacognosy and Pharmaceutical Botany, Faculty of Pharmaceutical Sciences, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand.

[E]Research Unit of Natural Products Utilization, Walailak University, Thasala, Nakhon Si Thammarat 80160, Thailand.

[F]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia.

[G]Department of Materials and Life Science, Seikei University, Tokyo 180 8633, Japan.

[H]Corresponding author. Email: chatchanok.k@psu.ac.th

Three new xanthones, pruniflorone M-O (**1**–**3**), and a new xanthonolignoid, 3-methoxy-5′-demethoxycadensin G (**4**), were isolated from the green fruits of *Cratoxylum formosum* ssp. *pruniflorum* along with three known xanthones (**5**–**7**) and a known flavonoid (**8**). Their structures were elucidated by spectroscopic methods and the structure of **1** was also determined by X-ray crystallography. Compounds **2** and **7** showed potent nitric oxide inhibitory activity with IC_{50} values of 4.4 and 4.3 μM, respectively. Moreover, **7** also showed strong antibacterial activity against both Gram-positive and Gram-negative bacteria with an MIC value of 4.67 μg mL^{-1}.

Manuscript received: 12 May 2010.
Manuscript accepted: 18 August 2010.

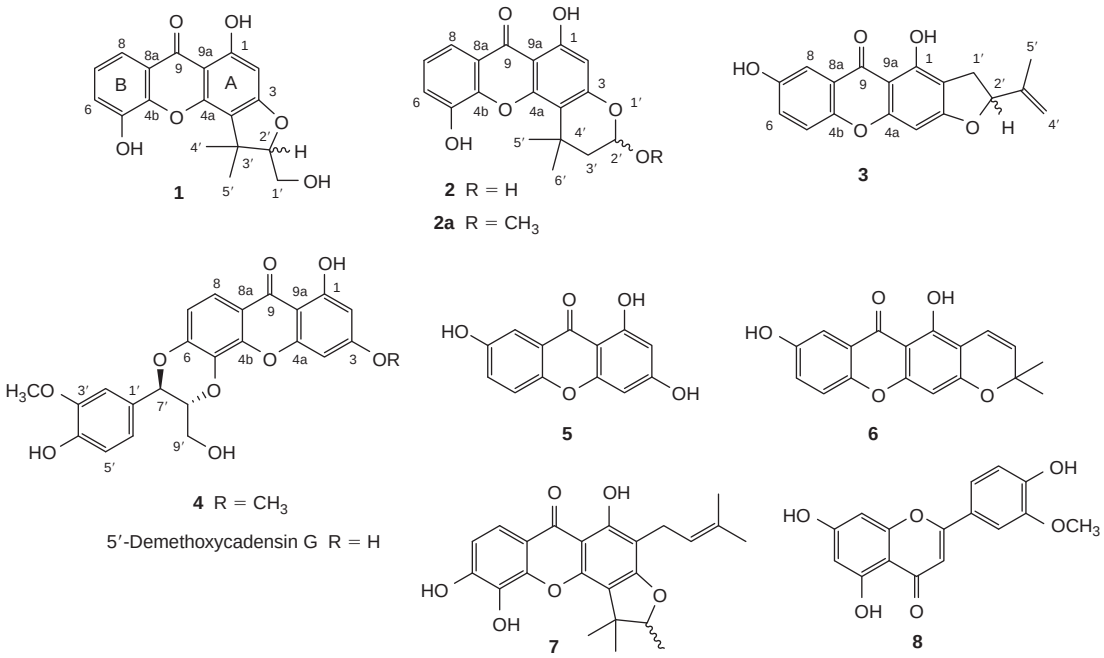
Introduction

The plants in the *Cratoxylum* genus were mainly found to produce the xanthones as major components.[1–5] Several isolated xanthones showed various interesting biological activities.[6–9] In our recent work, we have reported the isolation and characterization of constituents from the green fruits of *C. cochinchinense*,[5] whose results suggested that 1,3,7-trioxygenated xanthones were produced as major components in the green fruits of this plant and some of them exhibited potent antibacterial activity, specifically against *Pseudomonas aeruginosa*. These interesting results led us to investigate the chemical constituents from the green fruits of *C. formosum* ssp. *pruniflorum*. Herein, we report four new compounds: two 1,3,5-trioxygenated xanthones (**1**–**2**), a 1,3,7-trioxygenated xanthone (**3**), and a xanthonolignoid (**4**), along with three known xanthones (**5**–**7**) and a known flavonoid (**8**) from crude CH_2Cl_2 extract of the green fruits of *C. formosum* ssp. *pruniflorum*. In addition, the antimicrobial and nitric oxide (NO) inhibitory activities were also evaluated.

Results and Discussion

The green fruits of *C. formosum* ssp. *pruniflorum* were extracted with CH_2Cl_2 and CH_3OH successively and evaporated under vacuum to afford the brownish crude CH_2Cl_2 and CH_3OH extracts, respectively, which were further subjected to preliminary screening for antimicrobial and NO inhibitory activities. The crude CH_2Cl_2 and CH_3OH extracts were found to exhibit an inhibitory effect on NO production in murine macrophage-like RAW264.7 cells with IC_{50} values of 27.3 and 33.1 μg mL^{-1}, respectively, whereas they were found to be inactive for antimicrobial activity. Only the crude CH_2Cl_2 extract was subjected to silica gel chromatography, leading to the isolation of three new xanthones, pruniflorone M-O (**1**–**3**), and a new xanthonolignoid, 3-methoxy-5′-demethoxycadensin G (**4**), together with three known xanthones and a known flavonoid identified as 1,3,7-trihydroxyxanthone (**5**),[10] osajaxanthone (**6**),[11] formoxanthone C (**7**),[2] and chrysoeriol (**8**)[12] (Scheme 1). Their structures were elucidated by NMR analysis and by

10.1071/CH10193 0004-9425/10/111550



Scheme 1. Structures of compounds **1**–**8**.

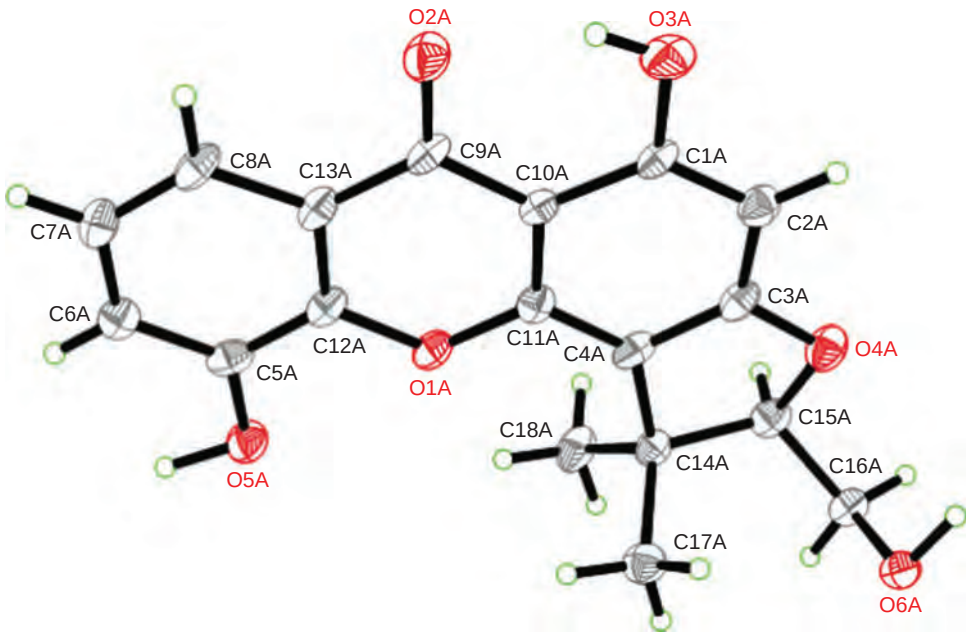


Fig. 1. *ORTEP* representation of pruniflorone M (**1**), with thermal ellipsoids at 50%.

comparison of their spectroscopic data with those reported in the literature.

Pruniflorone M (**1**) was obtained as yellow powder, which was further recrystallized from acetone to give yellow single crystals. The X-ray structure of **1** (Fig. 1) suggested a xanthone skeleton bearing a 2-hydroxymethyl-3,3-dimethyldihydrofuran ring. The molecular ion peak at *m/z* 328.0947 $[M]^+$ in the high resolution-electron ionization mass spectrometry (HR-EIMS) spectrum corresponded to $C_{18}H_{16}O_6$. The UV spectrum showed the absorption bands of xanthone at 246, 257, 315, and 357 nm, while the IR spectrum exhibited the hydroxyl and conjugated carbonyl functionalities at ν_{max} 3368 and 1648 cm^{-1}, respectively. The ^1H and ^{13}C NMR spectral data of **1** are summarized in Table 1. The ^1H NMR spectrum of **1** showed a chelated hydroxyl proton at δ 13.27 (s), an aromatic proton in ring A at δ 6.21 (s, H-2) and the characteristic peaks of the 1,2,3-trisubstituted benzene ring (ring B) at δ 7.68 (dd, *J* 7.8, 1.8, H-8), δ 7.39 (dd, *J* 7.8, 1.8, H-6), and δ 7.26 (t, *J* 7.8, H-7). Moreover, the ^1H NMR spectrum of **1** also showed the characteristic signal of a 2′-hydroxymethyl-3′,3′-dimethyldihydrofuran ring at δ 4.54 (t, *J* 5.7, H-2′), δ 3.93 (d, *J* 5.7, 2H-1′), δ 1.71 (s, 4′-CH_3), and δ 1.48 (s, 5′-CH_3). The connection of the furan ring to ring A at C-3 and C-4 in an angular orientation was indicated from the following HMBC correlations (Table 1). The methyl protons 4′-CH_3 (δ 1.71) and 5′-CH_3 (δ 1.48)

Table 1. NMR spectral data (d_6-acetone) of compounds 1–3

No.	1			2			3		
	δ_H (*J*, in Hz)	δ_C	HMBC	δ_H (*J*, in Hz)	δ_C	HMBC	δ_H (*J*, in Hz)	δ_C	HMBC
1-OH	13.27, s	164.4	1, 2, 3, 9a	12.82, s	161.1	1, 2, 9, 9a	13.42, s	160.9	1, 2, 9a
2	6.21, s	93.2	1, 3, 4, 4a, 9a	6.03, s	99.0	1, 4, 9a, 4a, 4′		108.3	
3		166.4			160.6			165.6	
4		113.1			109.6		6.44, s	94.0	2, 3, 9, 4a, 9a
5		146.3			146.8		7.42, d, 8.7	118.7	7, 4b, 8a
6	7.39, dd, 7.8, 1.8	120.4	5, 8, 4b	7.26, brd, 7.8	119.9	5, 8, 5a	7.33, dd, 8.7, 3.0	124.1	8, 4b
7	7.26, t, 7.8	123.9	5, 8, 4b, 8a	7.10, t, 7.8	123.9	5, 6, 8, 5a, 8a		154.0	
8	7.68, dd, 7.8, 1.8	115.4	6, 9, 4b	7.68, d, 7.8	114.8	5, 6, 7, 9, 5a	7.57, d, 3.0	108.4	6, 7, 9, 4b
9		180.7			181.2			180.2	
4a		152.6			155.7			156.4	
4b		145.2			145.3			149.7	
8a		121.5			121.2			121.0	
9a		103.4			104.1			103.0	
1′	3.93, d, 5.7	60.4	2′, 3′	–	–	–	3.05, dd, 14.1, 2.4	29.0	1, 2, 3, 3′
							2.92, dd, 14.1, 7.5		1, 2, 3, 3′
2′	4.54, t, 5.7	94.8	3, 4, 1′, 3′, 4′, 5′	5.39, dd, 7.8, 2.1	93.1	3, 3′, 4′	4.42, m	75.3	2, 4′
3′		43.1		1.87, dd, 13.8, 2.1	45.9	4, 2′, 4′, 5′, 6′		147.6	
				1.78, dd, 13.8, 7.8		4, 2′, 4′			
4′	1.71, s	26.2	4, 2′, 3′, 5′		31.9		4.89, brs	109.4	3′, 4′, 5′
							4.72, brs		3′, 5′
5′	1.48, s	20.2	4, 1′, 2′, 3′, 4′	1.59, s	28.1	4, 2′, 4′, 6′	1.83, brs	17.3	3′, 4′
6′	–	–	–	1.48, s	28.0	4, 4′	–	–	–

showed ^3J correlation with C-4 (δ 113.1), whereas H-2′ at δ 4.54 showed correlations with C-3 (δ 166.4), C-4 (δ 113.1), C-1′ (δ 60.4), C-3′ (δ 43.1), C-4′ (δ 26.2), and C-5′ (δ 20.2). Therefore, compound **1** was characterized as 2′-hydroxymethyl-3′,3′-dimethyldihydrofuran-1,5-dihydroxyxanthone and designated as pruniflorone M.

Pruniflorone N (**2**) was isolated as a yellow powder, mp 250–252°C. The molecular ion peak at *m/z* 328.0948 $[M]^+$ in the HR-EIMS spectrum corresponded to $C_{18}H_{16}O_6$. The UV spectrum showed the absorption bands of xanthone at 246, 259, 316, and 356 nm, while the IR spectrum exhibited the hydroxyl and conjugated carbonyl functionalities at ν_{max} 3411 and 1651 cm^{-1}, respectively. The ^1H NMR spectral data of **2** (Table 1) showed similarity with those of **1**, except for the appearance of a 2′-hydroxy-4′,4′-dimethylpyran ring at δ 5.39 (dd, *J* 7.8, 2.1, H-2′), δ 1.87 (dd, *J* 13.8, 2.1, H-3′), δ 1.78 (dd, *J* 13.8, 7.8, H-3′), 1.59 (s, 5′-CH_3), and 1.48 (s, 6′-CH_3) in **2** instead of the 2′-hydroxymethyl-3′,3′-dimethyldihydrofuran ring in **1**. The pyran ring was connected to C-3 and C-4 in an angular orientation as indicated from the following HMBC correlations (Table 1). The signal of an oxymethine H-2′ at δ 5.39 showed correlations with C-3 (δ 160.6), C-3′ (δ 45.9), and C-4′ (δ 31.9), whereas H_2-3′ at δ 1.87 and 1.78, CH_3-5′ (δ 1.59) and CH_3-6′ (δ 1.48) showed correlations with C-4 (δ 109.6). Therefore, compound **2** was characterized as 2′-hydroxy-4′,4′-dimethylpyran-1,5-dihydroxyxanthone and assigned as pruniflorone N.

Pruniflorone O (**3**) was obtained as a yellow viscous oil. The molecular ion peak at *m/z* 310.0845 $[M]^+$ in the HR-EIMS spectrum corresponded to $C_{18}H_{14}O_5$. The UV spectrum showed characteristic absorption bands of xanthone at 243, 269, 280, 315, and 352 nm, while the IR spectrum exhibited the hydroxyl and conjugated carbonyl functionalities at ν_{max} 3378 and 1630 cm^{-1}, respectively. The ^1H NMR spectrum of **3** (Table 1) showed a chelated hydroxyl proton at δ 13.42 (s), an aromatic proton in ring A at δ 6.44 (s, H-4) and the characteristic signals of ABX trisubstituted benzene in ring B at δ 7.57 (d, *J* 3.0, H-8), δ 7.42 (d, *J* 8.7, H-5), and δ 7.33 (dd, *J* 8.7, 3.0, H-6). Moreover, the ^1H NMR spectrum of **3** (Table 1) also showed the typical signal of a dihydrofuran ring with an isopropenyl side chain at δ 4.89 (br s, H-4′), δ 4.72 (br s, H-4′), δ 4.42 (m, H-2′), δ 3.05 (dd, *J* 14.1, 2.4, H-1′), δ 2.92 (dd, *J* 14.1, 7.5, H-1′), and δ 1.83 (br s, 5′-CH_3). A dihydrofuran ring was connected to C-2 and C-3 of ring A in a linear fashion as indicated by HMBC correlations (Table 1). The non-equivalent methylene protons H_2-1′ (δ 3.05 and 2.92) showed correlations with C-1 (δ 160.9), C-2 (δ 108.3), C-3 (δ 165.6), and C-3′ (δ 147.6), whereas an oxymethine H-2′ (δ 4.42) showed correlations with C-2 (δ 108.3) and C-4′ (δ 109.4). Therefore, compound **3** was characterized as 2′-isopropenyldihydrofuran-1,7-dihydroxyxanthone and assigned as pruniflorone O.

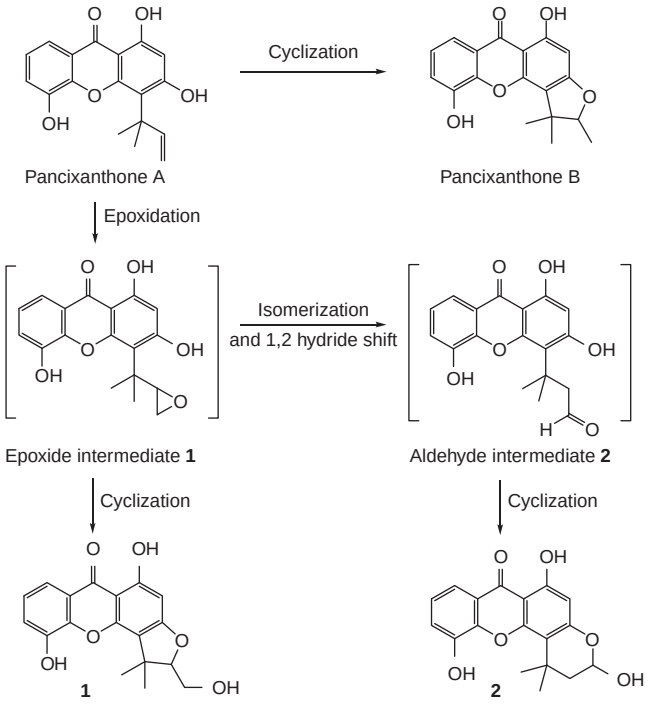
Compound **4** was obtained as a yellow powder with optical activity of $[\alpha]_D^{26} = +53.4$ (*c* 0.06 in acetone). The molecular ion peak at *m/z* 452.1119 $[M]^+$ in the HR-EIMS spectrum corresponded to $C_{24}H_{20}O_9$. The UV spectrum of **4** showed similar absorption bands (253, 281, and 318 nm) to those of 5′-demethoxycadensin G,[13] while the IR spectrum exhibited the hydroxyl and conjugated carbonyl functionalities at ν_{max} 3431 and 1646 cm^{-1}, respectively. The ^1H and ^{13}C NMR spectral data of **4** (Table 2) were closely related to those of 5′-demethoxycadensin G previously isolated from *C. cochinchinense*.[13] The difference was shown as an additional methoxyl group (δ_H 3.83, δ_C 56.0) in compound **4** which was assigned at C-3 due to its HMBC correlation (Table 2) to δ 166.7 (C-3). Therefore, compound **4** was characterized as 3-methoxy-5′-demethoxycadensin G.

The plausible biosynthetic pathway of pruniflorone M and N (**1** and **2**, respectively) is illustrated in Scheme 2. We propose that compounds **1** and **2** could be derived from pancixanthone A, which was previously isolated from the stem barks of *Calophyllum panciflorum*.[14] The cyclization of the phenol group

Table 2. NMR spectral data ($CDCl_3$-CD_3OD (19:1 v/v)) of compound 4

No.	4			5′-demethoxycadensin G[A]	
	δ_H (J, in Hz)	δ_C	HMBC	δ_H (J, in Hz)[B]	δ_C[B]
1-OH		163.3		13.07, s	164.8
2	6.28, d, 2.4	97.5	1, 3, 4, 9a	6.26, d, 2.1	99.2
3		166.7			166.3
4	6.46, d, 2.4	93.2	2, 3, 4a, 9a	6.47, d, 2.1	94.9
5		131.9			115.7
6		146.5			150.3
7	6.92, d, 8.8	114.2	5, 6, 8a	7.01, d, 8.9	114.5
8	7.67, d, 8.8	117.8	6, 9, 4b	7.70, d, 8.9	117.8
9		180.6			180.8
4a		157.8			158.8
4b		149.5			147.2
8a		115.3			132.9
9a		103.6			103.3
1′		127.1			128.4
2′	6.89, d, 1.6	110.5	4′, 6′, 7′	7.18, d, 1.9	112.2
3′		147.9			148.7
4′		147.2			148.4
5′	6.86, d, 8.8	115.5	1′, 3′	6.92, d, 8.0	115.9
6′	6.89, dd, 8.8, 1.6	120.9	2′, 4′, 7′	7.03, dd, 8.0, 1.9	121.8
7′	5.06, d, 8.0	77.2	1′, 2′, 6′, 8′	5.21, d, 7.9	77.9
8′	4.07, ddd, 8.0, 3.6, 2.8	79.0	–	4.32, ddd, 7.9, 3.7, 2.5	79.7
9′	3.89, m	61.0	8′	3.94, dd, 12.6, 3.7	61.6
	3.54, dd, 12.8, 3.6		–	3.63, dd, 12.6, 2.5	
3-OCH_3	3.83, s	56.0	3	–	–
3′-OCH_3	3.85, s	56.2	3′	3.89, s	56.5

[A]It was previously reported by Sia et al.[13]
[B] ^1H (500 MHz) and ^{13}C (125 MHz) NMR spectral data were recorded in d_6-acetone.



Scheme 2. Plausible biosynthetic pathway of compounds **1** and **2**.

with the dimethylallyl group of pancixanthone A furnished a 2,3,3-trimethyldihydrofuran ring, which was reported as pancixanthone B.[14] This transformation was a common pathway occurring in natural products. Compounds **1** and **2** could be formed from different pathway to the pancixanthone B. We proposed that the dimethylallyl group of pancixanthone A could be oxidized leading to an epoxide intermediate **1**.[15] Then, the cyclization of the free hydroxyl group at C-3 to the epoxide moiety led to the formation of the 2-hydroxymethyl-3,3-dimethyldihydrofuran ring of compound **1**. The 2-hydroxy-4,4-dimethylpyran ring of **2** could be formed from epoxide intermediate **1** by isomerization of epoxide via a 1,2-hydride shift to form an aldehyde intermediate **2**. The cyclization of the hydroxyl group of ring A to the aldehyde moiety occurred in the last step to form the pyran ring of compound **2**. The formations of 2-hydroxymethyl-3,3-dimethyldihydrofuran ring and 2-hydroxy-4,4-dimethylpyran ring are found for the first time in natural products.

Only isolated compounds with sufficient amount were further evaluated for their NO inhibitory activity using RAW264.7 cells.[16] As shown in Table 3, compounds **2** and **7** possessed potent NO inhibitory activity against lipopolysaccharide (LPS)-induced NO release with IC_{50} values of 4.4 and 4.3 μM, respectively, better than that of the positive control, indomethacin, which is a non-steroidal anti-inflammatory drug ($IC_{50} = 20.1$ μM). Compound **1** ($IC_{50} = 20.6$ μM) exhibited moderate NO inhibitory activity when compared with that of compound **2** ($IC_{50} = 4.4$ μM), whereas compound **4** was inactive ($IC_{50} > 100$ μM). From these results, it might be proposed that the pyran ring of 1,3,5-trioxygenated xanthone (**2**) was essential for NO inhibitory activity rather than the furan ring (**1**), whereas the catechol unit of compound **7** might play an important role for NO inhibition. Moreover, compound **2** has a part of a hemiacetal moiety in its pyran ring, which could possibly open to an aldehyde side chain under acidic conditions. The experiment

Table 3. Nitric oxide inhibitory activity of 1, 2, 2a, 4, and 7
Statistical significance, $*P < 0.05$, $**P < 0.01$

Compounds	% inhibition of various concentrations [μM]					IC_{50} [μM]
	0	3	10	30	100	
1	0.0 ± 4.5	15.5 ± 0.9**	29.6 ± 0.9**	44.0 ± 1.6**	95.3 ± 0.6[B]**	20.6
2	0.0 ± 4.5	34.8 ± 1.4*	72.7 ± 1.1**	93.1 ± 1.7**	99.5 ± 0.6[B]**	4.4
2a	0.0 ± 2.4	15.7 ± 5.3	44.4 ± 1.9**	91.0 ± 1.0**	101.3 ± 3.0[B]**	9.5
4	0.0 ± 5.4	–	21.7 ± 1.0*	23.5 ± 2.0**	47.4 ± 3.1**	>100
7	0.0 ± 5.4	30.1 ± 1.6**	94.3 ± 0.7[B]**	102.1 ± 2.4[B]**	102.9 ± 0.7[B]**	4.3
Indomethacin	0.0 ± 4.2	16.6 ± 2.9	32.7 ± 2.6**	53.4 ± 3.0**	85.6 ± 1.8**	20.1
L-NA	0.0 ± 5.6	15.3 ± 2.8	21.4 ± 2.5	35.6 ± 2.1**	73.2 ± 3.5**	59.0
CAPE	0.0 ± 5.6	35.2 ± 3.0*	70.3 ± 2.7**	97.6 ± 2.4[B]**	99.5 ± 2.7[B]**	5.0

[A]Each value represents mean ± s.e.m. of four determinations.
[B]Cytotoxic effect was observed.

Table 4. Antimicrobial activity (MIC, μg mL^{-1}) of 1, 2, 2a, 4, 7, and 8

Compounds	Antibacterial activity								Antifungal activity
	Gram-positive bacteria[A]					Gram-negative bacteria[B]			
	B. subtilis	*S. aureus*	*E. faecalis*	MRSA	VRE	*S. typhi*	*S. sonei*	*P. aeruginosa*	*C. albicans*[C]
1	>300	>300	>300	>300	>300	>300	>300	>300	>300
2	300	>300	>300	9.37	300	150	>300	>300	>300
2a	18.75	300	300	37.5	75	300	300	37.5	300
4	>300	>300	>300	>300	>300	>300	>300	>300	>300
7	4.67	9.37	4.67	4.67	4.67	4.67	>300	37.5	>300
8	>300	>300	>300	37.5	37.5	300	>300	>300	>300
Vancomycin	<2.34	<2.34	<2.34	<2.34	<2.34	<2.34	<2.34	<2.34	<2.34

[A]*Bacillus subtilis*, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR459, methicillin-resistant *Staphylococcus aureus* (MRSA) ATCC43300, vancomycin-resistant *Enterococcus faecalis* (VRE) ATCC51299.
[B]*Salmonella typhi*, *Shigella sonei*, and *Pseudomonas aeruginosa*.
[C]*Candida albicans*.

has been set up in 20% HCl-CH_3OH. It was found that an aldehyde product was not observed, instead the methylated product **2a** was formed. Compound **2a** was further tested for NO inhibition and showed less NO inhibitory activity than **2** with an IC_{50} value of 9.5 μM. From this result, it could be suggested that the methoxyl group might decrease the NO inhibitory activity.

Moreover, the antibacterial and antifungal activities[2,5] were also evaluated and are summarized in Table 4. The activity results in Table 4 revealed that compound **7** exhibited strong antibacterial activity against both Gram-positive (*Bacillus subtilis*, *Enterococcus faecalis*, methicillin-resistant *Staphylococcus aureus* (MRSA), and vancomycin-resistant *E. faecalis* (VRE)) and Gram-negative (*S. typhi*) bacteria with an MIC value of 4.67 μg mL^{-1}, whereas compound **2** showed moderate antibacterial activity specifically against MRSA with an MIC value of 9.37 μg mL^{-1}. The methylated product **2a** was not significant for antibacterial activity but showed better activity against *B. subtilis* when compared with compound **2**. All tested compounds were found to be inactive for anti-fungal activity.

Conclusions

Four new compounds, pruniflorone M-O (**1**–**3**), and 3-methoxy-5′-demethoxycadensin G (**4**), were isolated from the green fruits of *C. formosum* ssp. *pruniflorum*. Pruniflorone N (**2**) and compound **7** showed potent NO inhibitory activity with IC_{50} values of 4.4 and 4.3 μM, respectively. Moreover, compound **7** also showed strong antibacterial activity against both Gram-positive and Gram-negative bacteria with an MIC value of 4.67 μg mL^{-1}.

Experimental

General Procedures

Melting points were determined on the Fisher–John melting point apparatus. Optical rotations were measured on a JASCO P-1020 digital polarimeter. UV and IR spectra were recorded on SPECORD S 100 (Analytikjena) and Perkin–Elmer FTS FT-IR spectrophotometer, respectively. The ^1H and ^{13}C NMR spectra were recorded on 300 MHz and 400 MHz Bruker FTNMR Ultra Shield™ spectrometers in d_6-acetone, $CDCl_3$, and CD_3OD with TMS as the internal standard. Chemical shifts are reported in δ [ppm] and coupling constants (*J*) are expressed in hertz. EI and HREI mass spectra were measured on a Kratos MS 25 RFA spectrometer. Quick column chromatography (QCC) and column chromatography (CC) were carried out on silica gel 60 F_{254} (Merck) and silica gel 100 (Merck), respectively.

Plant Material

Green Fruits of *C. formosum* ssp. *pruniflorum* were collected in August 2008 from Pha Yao Province, in northern Thailand. Botanical identification was carried out by comparison with a voucher specimen number 0012677 in the herbarium collection of Department of Biology, Faculty of Science, Prince of Songkla University, Thailand.

Extraction and Isolation of Compounds 1–8

The green fruits of *C. formosum* ssp. *pruniflorum* (5.00 kg) were extracted with CH_2Cl_2 and CH_3OH (each 2 × 20 L, for a week) successively at room temperature and were further evaporated under reduced pressure to afford the crude extracts of CH_2Cl_2 (31.42 g) and CH_3OH (65.74 g), respectively. Only the crude extract of CH_2Cl_2 was further subjected to QCC on silica gel using hexane as a first eluent and then increasing the polarity with acetone to give 14 fractions (F1–F14). Fraction F7 was separated by CC eluting with a gradient of acetone-hexane to give 10 subfractions (F7A–F7J). Subfraction F7G was separated by CC and eluted with 20% acetone-hexane to give 7 subfractions (F7G1–F7G7). Subfraction F7G3 was further purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give **6** (1.2 mg). Fraction F10 was separated by QCC eluting with a gradient of acetone-hexane to give 17 subfractions (F10A–F10Q). Subfractions F10N and F10O were separated by CC and eluted with a gradient of EtOAc-hexane to give 8 subfractions (F10N1–F10N8). Subfractions F10N1 was further purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give **3** (1.2 mg). Subfraction F10N2 was separated by CC and eluted with $CHCl_3$ to give **1** (28.0 mg) and **5** (1.5 mg). Subfraction F10N6 was separated by CC and eluted with $CHCl_3$ to give **2** (5.3 mg) and **7** (15.2 mg). Fractions F12 and F13 were separated by QCC eluting with a gradient of acetone-hexane to give 13 subfractions (F12A–F12J). Subfraction F12H was separated by CC and eluted with a gradient of acetone-hexane to give 10 subfractions (F12H1–F12H10). Subfractions F12H4 and F12H5 were further purified by CC eluting with a gradient of EtOAc-hexane to give **4** (3.7 mg). Subfraction F12I was further separated by CC eluting with a gradient of acetone-hexane to give **8** (3.0 mg).

Pruniflorone M (1)

Yellow single crystal; mp 235–237°C; $[\alpha]_D^{25} = +64.6$ (*c* 0.04 in $CHCl_3$). λ_{max} ($CHCl_3$)/nm (log ε) 246 (4.45), 257 (4.34), 315 (4.14), 357 (3.58). ν_{max} (neat)/cm^{-1} 3368, 1648, 1587. *m/z* (HRMS) 328.0947 [M^+] for $C_{18}H_{16}O_6$ (calc. 328.0947). *m/z* (EI) 328 $[M]^+$ (38%), 313 (93), 283 (100), 255 (25), 141 (5). For ^1H and ^{13}C NMR spectroscopic data, see Table 1.

Pruniflorone N (2)

Yellow powder; mp 250–252°C; $[\alpha]_D^{25} = +5.2$ (*c* 0.42 in acetone). λ_{max} ($CHCl_3$)/nm (log ε) 246 (4.32), 259 (4.21), 316 (4.02), 356 (3.39). ν_{max} (neat)/cm^{-1} 3411, 1651, 1622, 1578. *m/z* (HRMS) 328.0948 $[M]^+$ for $C_{18}H_{16}O_6$ (calc. 328.0947). *m/z* (EI) 328 $[M]^+$ (40%), 313 (100), 285 (31), 257 (16), 243 (12), 149 (6). For ^1H and ^{13}C NMR spectroscopic data, see Table 1.

Pruniflorone O (3)

Yellow viscous oil; $[\alpha]_D^{26} = +15.1$ (*c* 0.04 in acetone). λ_{max} ($CHCl_3$)/nm (log ε) 243 (4.38), 269 (4.07), 280 (3.99), 315 (3.78), 352 (3.61). ν_{max} (neat)/cm^{-1} 3378, 1630. *m/z* (HRMS) 310.0845 $[M]^+$ for $C_{18}H_{14}O_5$ (calc. 310.0841). *m/z* (EI) 310 $[M]^+$ (3%), 295 (4), 257 (100), 229 (4). For ^1H and ^{13}C NMR spectroscopic data, see Table 1.

3-Methoxy-5′-demethoxycadensin G (4)

Yellow powder; $[\alpha]_D^{26} = +53.4$ (*c* 0.06 in acetone). λ_{max} ($CHCl_3$)/nm (log ε) 253 (4.37), 281 (3.83), 318 (3.97). ν_{max} (neat)/cm^{-1} 3431, 1646. *m/z* (HRMS) 452.1119 $[M]^+$ for $C_{24}H_{20}O_9$ (calc. 452.1107). *m/z* (EI) 452 $[M]^+$ (100%), 434 (2), 420 (13), 393 (12), 315 (18), 285 (17), 274 (35), 245 (23), 180 (73), 162 (15), 137 (66), 124 (40), 119 (13), 101 (13), 77 (5). For ^1H and ^{13}C NMR spectroscopic data, see Table 2.

Hydrolysis of 2

A solution of compound **2** (7.8 mg) in 20% HCl–CH_3OH (2.0 mL) was left to stand for 4 days at room temperature. The solution was evaporated under vacuum to give a residue, which was purified by CC on silica gel and eluted with 25% acetone-hexane to give compound **2** (3.5 mg) and compound **2a** (3.5 mg). Compound **2a** was a yellow powder; mp 208–210°C; $[\alpha]_D^{28} = +40.8$ (*c* 0.18 in acetone), δ_H (300 MHz, d_6-acetone) 12.84 (s, 1-OH), 7.55 (dd, *J* 7.8, 1.5, H-8), 7.28 (dd, *J* 7.8, 1.5, H-6), 7.14 (t, *J* 7.8, H-7), 6.12 (s, H-2), 5.09 (dd, *J* 6.0, 3.0, H-2′), 3.41 (s, 2′-OCH_3), 1.89 (dd, *J* 13.8, 6.3, 1H-3′), 1.82 (dd, *J* 13.8, 6.3, 1H-3′), 1.58 (s, 5′-CH_3), 1.50 (s, 6′-CH_3). δ_C (75 MHz, d_6-acetone) 181.3 (C-9), 161.2 (C-1), 159.6 (C-3), 155.7 (C-4a), 146.5 (C-5), 145.4 (C-4b), 124.0 (C-7), 121.3 (C-8a), 120.0 (C-6), 115.1 (C-8), 110.0 (C-4), 104.4 (C-9a), 99.2 (C-2), 99.6 (C-2′), 55.6 (2′-OCH_3), 43.8 (C-3′), 31.1 (C-4′), 28.0 (C-5′), 28.4 (C-6′). *m/z* (HRMS) 342.1091 $[M]^+$ for $C_{19}H_{18}O_6$ (calc. 342.1103). *m/z* (EI) 342 $[M]^+$ (42%), 327 (100), 295 (68), 259 (7), 83 (4).

Crystallography

Crystallographic data were collected at 100.0(1) K with the Oxford Cryosystem Cobra low-temperature attachment. The data were collected using a Bruker Apex2 CCD diffractometer with a graphite monochromated MoKα radiation at a detector distance of 5 cm and swing angle of −35°. A hemisphere of the reciprocal space was covered by a combination of four sets of exposures using *SMART* program.[17] The collected data were reduced using *SAINT* program,[17] and the empirical absorption corrections were performed using *SADABS* program.[17] The structure was solved by direct methods and refined by least-squares using the *SHELXTL* software package.[18] All non-hydrogen atoms were refined anisotropically, whereas all H atoms were placed in calculated positions with an O-H distance of 0.82 Å and C-H distances in the range 0.93–0.98 Å after checking their positions in the difference map. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atoms for methyl H atoms and $1.2U_{eq}$ for hydroxyl and the other H atoms. The final refinement converged well. Materials for publication were prepared using *SHELXT*[18] and *PLATON*.[19]

Crystal data of **1**: $C_{18}H_{16}O_6 \cdot H_2O$, $M = 346.32$, $0.54 \times 0.17 \times 0.10\ mm^3$, orthorhombic, $P2_12_12_1$, $a = 9.8204(2)$ Å, $b = 15.4749(4)$ Å, $c = 20.4482(6)$ Å, $\alpha = \beta = \gamma = 90.00°$, $V = 3107.51(14)$ $Å^3$, $Z = 4$, $D_x = 1.480\ Mg\ m^{-3}$, $\mu(MoK\alpha) = 0.115\ mm^{-1}$, 16842 reflection measured, 6088 unique reflections, $r = 0.0654$, $R_w = 0.1345$.

The crystallographic-information file of **1** has been deposited in the Cambridge Crystallographic Data Centre as CCDC766256. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

Bioassays

Nitric Oxide Inhibitory Activity Assay

The inhibitory effect on NO production by murine macrophage-like RAW264.7 cells was evaluated using a modified method from that previously reported.[16] Briefly,

the RAW264.7 cell line (purchased from Cell Lines Services) was cultured in RPMI medium supplemented with 0.1% sodium bicarbonate and 2 mM glutamine, penicillin G (100 units mL^{-1}), streptomycin (100 $\mu g\,mL^{-1}$), and 10% FCS. The cells were harvested with trypsin-EDTA and diluted to a suspension in a fresh medium. The cells were seeded in 96-well plates with 10^5 cells/well and allowed to adhere for 1 h at 37°C in a humidified atmosphere containing 5% CO_2. Then the medium was replaced with a fresh medium containing 50 $\mu g\,mL^{-1}$ of LPS together with the test samples at various concentrations (3–100 $\mu g\,mL^{-1}$ for crude extract and 3–100 μM for pure compounds) and was then incubated for 24 h. NO production was determined by measuring the accumulation of nitrite in the culture supernatant using the Griess reagent. Cytotoxicity was determined using the MTT colorimetric method. Briefly, after 24 h incubation with the test samples, MTT solution (10 μL, 5 $mg\,mL^{-1}$ in PBS) was added to the wells. After 4 h incubation, the medium was removed, and isopropanol containing 0.04 M HCl was then added to dissolve the formazan production in the cells. The optical density of the formazan solution was measured with a microplate reader at 570 nm. The test compounds were considered to be cytotoxic when the optical density of the sample-treated group was less than 80% of that in the control (vehicle-treated) group. L-NA, CAPE, and indomethacin were used as positive controls. The stock solution of each test sample was dissolved in DMSO, and the solution was added to the medium RPMI (final DMSO is 1%). Inhibition [%] was calculated using the following equation and IC_{50} values were determined graphically ($n = 4$):

$$\text{Inhitition } [\%] = \frac{\text{A} - \text{B}}{\text{A} - \text{C}} \times 100 \qquad (1)$$

A–*C*: NO_2^- concentration [μM] [*A*: LPS (+), sample (−); *B*: LPS (+), sample (+); *C*: LPS (−), sample (−)].

Antibacterial Assay

Only isolated compounds with sufficient amount from the green fruits of *C. formosum* ssp. *pruniflorum* were tested against both Gram-positive and Gram-negative bacteria (i.e. *B. subtilis*, *S. aureus* TISTR517, *E. faecalis* TISTR459, MRSA ATCC43300, VRE ATCC51299, *S. faecalis*, *S. typhi*, *Shigella sonei*, and *P. aeruginosa*). The tested microorganisms were obtained from the culture collections, Department of Industrial Biotechnology and Department of Pharmacognosy and Botany, PSU, except for the TISTR and ATCC strains, which were obtained from Microbial Research Center (MIRCEN), Bangkok, Thailand. The antibacterial assay employed was the same as described in Boonsri et al.[2] Vancomycin, which was used as a standard, showed antibacterial activity of 75.0 $\mu g\,mL^{-1}$.

Antifungal Assay

Candida albicans was obtained from Department of Pharmacognosy and Botany, PSU. The antifungal assay employed was the same as described in Boonsri et al.[2]

Accessory Publication

The spectra of compounds **1**–**4** are available on the Journal's website.

Acknowledgements

We are greatly indebted to the Thailand Research Fund (TRF) for research grant (RSA 5280033). Financial support from the Center for Innovation in Chemistry (PERCH-CIC), Commission on Higher Education, Ministry of Education is gratefully acknowledged. N.B. thanks the Development and Promotion of Science and Technology Talents Project for fellowship and the Graduate School, Prince of Songkla University for financial supports. The Koshinocorporation Group, Japan is also acknowledged.

References

[1] N. Boonnak, C. Karalai, S. Chantrapromma, C. Ponglimanont, H.-K. Fun, A. Kanjana-Opas, S. Laphookhieo, *Tetrahedron* **2006**, *62*, 8850. doi:10.1016/J.TET.2006.06.003

[2] S. Boonsri, C. Karalai, C. Ponglimanont, A. Kanjana-Opas, K. Chantrapromma, *Phytochemistry* **2006**, *67*, 723. doi:10.1016/J.PHYTOCHEM.2006.01.007

[3] S. Laphookhieo, J. K. Syers, R. Kiattansakul, K. Chantrapromma, *Chem. Pharm. Bull.* **2006**, *54*, 745. doi:10.1248/CPB.54.745

[4] W. Mahabusarakam, S. Rattanaburi, S. Phongpaichit, A. Kanjana-Opas, *Phytochem. Lett.* **2008**, *1*, 211. doi:10.1016/J.PHYTOL.2008.09.012

[5] N. Boonnak, C. Karalai, S. Chantrapromma, C. Ponglimanont, H.-K. Fun, A. Kanjana-Opas, K. Chantrapromma, S. Kato, *Tetrahedron* **2009**, *65*, 3003. doi:10.1016/J.TET.2009.01.083

[6] S. Suksamrarn, O. Komutiban, P. Ratananukul, N. Chimnoi, N. Lartpornmatulee, A. Suksamrarn, *Chem. Pharm. Bull.* **2006**, *54*, 301. doi:10.1248/CPB.54.301

[7] J. R. Nguemeving, A. G. B. Azebaze, V. Kuete, N. N. E. Carly, V. P. Beng, M. Meyer, A. Blond, B. Bodo, A. E. Nkengfack, *Phytochemistry* **2006**, *67*, 1341. doi:10.1016/J.PHYTOCHEM.2006.03.018

[8] L.-G. Chen, L.-L. Yang, C.-C. Wang, *Food Chem. Toxicol.* **2008**, *46*, 688. doi:10.1016/J.FCT.2007.09.096

[9] J. Ngoupayo, T. K. Tabopda, M. S. Ali, *Bioorg. Med. Chem.* **2009**, *17*, 5688. doi:10.1016/J.BMC.2009.06.009

[10] S. Laphookhieo, W. Maneerat, T. Buatip, J. K. Syers, *Can. J. Chem.* **2008**, *86*, 757. doi:10.1139/V08-076

[11] M. Mondal, V. G. Puranik, N. P. Argade, *J. Org. Chem.* **2006**, *71*, 4992. doi:10.1021/JO0606655

[12] T. Nakasugi, M. Nakashima, K. Komai, *J. Agric. Food Chem.* **2000**, *48*, 3256. doi:10.1021/JF9906679

[13] G.-L. Sia, G. J. Bennett, L. J. Harrison, K.-Y. Sim, *Phytochemistry* **1995**, *38*, 1521. doi:10.1016/0031-9422(94)00526-Y

[14] C. Ito, Y. Miyamoto, K. S. Rao, H. Furukawa, *Chem. Pharm. Bull.* **1996**, *44*, 441.

[15] P. M. Dewick, *Medicinal Natural Products; A Biosynthetic Approach* **2002**, pp. 145 (John Wiley & Sons Ltd: Chichester).

[16] S. Tewtrakul, S. Cheenpracha, C. Karalai, *Phytomedicine* **2009**, *16*, 568. doi:10.1016/J.PHYMED.2008.12.015

[17] Bruker, *APEX2, SAINT, and SADABS* **2005** (Bruker AXS Inc.: Madison, WI).

[18] G. M. Sheldric, *SHELXTL Version 5.1* **1998** (Bruker AXS Inc.: Madison, WI).

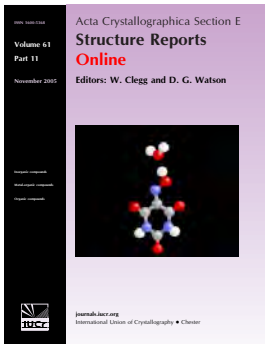
[19] A. L. Spek, *J. Appl. Cryst.* **2003**, *36*, 7. doi:10.1107/S0021889802022112

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

Vieillardiixanthone B

Nawong Boonnak, Suchada Chantrapromma, Hoong-Kun Fun and Chatchanok Karalai

Acta Cryst. (2010). E**66**, o817–o818



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Vieillardiixanthone B[1]

Nawong Boonnak,[a] Suchada Chantrapromma,[a]*§ Hoong-Kun Fun[b]¶ and Chatchanok Karalai[a]

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

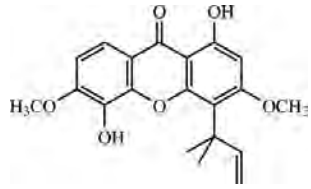
Received 14 February 2010; accepted 24 February 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.054; wR factor = 0.133; data-to-parameter ratio = 15.1.

The title compound [systematic name: 1,5-dihydroxy-3,6-dimethoxy-4-(2-methylbut-3-en-2-yl)-9*H*-xanthen-9-one], $C_{20}H_{20}O_6$, is a xanthone, which was isolated from the roots of *Cratoxylum formosum* ssp. *pruniflorum*. The three rings in the molecule are approximately coplanar, with an r.m.s. deviation of 0.0372 (2) Å for the plane through the 14 non-H atoms. The O atoms of the two hydroxy substituents also lie close to this plane with deviations of 0.0669 (2) and 0.1122 (2) Å, respectively. The 1,1-dimethyl-2-propenyl substituent is in a (−)-anticlinal conformation. Intramolecular O—H···O hydrogen bonds generate S(5) and S(6) ring motifs. In the crystal, molecules are linked into infinite chains along [010] by O—H···O hydrogen bonds and weak C—H···O interactions. π–π interactions with centroid–centroid distances of 3.6172 (10) and 3.6815 (10) Å are also observed.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987). For background to xanthones and their biological activity, see: Boonnak, Karalai *et al.* (2006, 2007, 2009); Hay *et al.* (2008). For a related structure, see: Boonnak, Chantrapromma & Fun (2006). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{20}H_{20}O_6$
M_r = 356.36
Monoclinic, $P2_1/c$
a = 12.1500 (4) Å
b = 14.7396 (4) Å
c = 9.5177 (3) Å
β = 90.208 (2)°
V = 1704.48 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.50 × 0.23 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.951, T_{max} = 0.978
37868 measured reflections
3906 independent reflections
2682 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.133
S = 1.03
3906 reflections
259 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1*O3*···O2	0.95 (3)	1.65 (3)	2.5573 (18)	160 (2)
O5—H1*O5*···O6	0.83 (2)	2.25 (2)	2.7019 (19)	115 (2)
O5—H1*O5*···O2^{i}	0.83 (2)	1.98 (2)	2.7520 (18)	155 (2)
C8—H8*A*···O5^{ii}	0.93	2.54	3.413 (2)	157

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for research grant (RSA 5280033) and the Prince of Songkla University for financial support. They also thank Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012. NB thanks the Development and Promotion of Science and Technology Talents Project for a fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2732).

[1] This paper is dedicated to His Majesty King Bhumibol Adulyadej of Thailand (King Rama IX) for his sustainable development of the country.
§ Thomson Reuters ResearcherID: A-5085-2009.
¶ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Boonnak, N., Chantrapromma, S. & Fun, H.-K. (2006). *Acta Cryst.* E**62**, o2034–o2036.

Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Fun, H.-K., Kanjana-Opas, A., Chantrapromma, K. & Kato, S. (2009). *Tetrahedron*, **65**, 3003–3013.

Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Fun, H.-K., Kanjana-Opas, A. & Laphookhieo, S. (2006). *Tetrahedron*, **62**, 8850–8859.

Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Kanjana-Opas, A., Chantrapromma, K. & Fun, H.-K. (2007). *Can. J. Chem.* **85**, 341–345.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Hay, A. E., Merza, J., Landreau, A., Litaudon, M., Pagniez, F., Pape, P. L. & Richomme, P. (2008). *Fitoterapia*, **79**, 42–46.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst*. D**65**, 148–155.

supplementary materials

Acta Cryst. (2010). E**66**, o817-o818 [**doi:10.1107/S1600536810007026**]

Vieillardiixanthone B

N. Boonnak, S. Chantrapromma, H.-K. Fun and C. Karalai

Comment

During the course of our studies into the chemical constituents and bioactive compounds from Thai medicinal plants, we previously reported the isolation of xanthones from the *Cratoxylum formosum* ssp. *pruniflorum*. We found that several isolated xanthones showed antibacterial, antifungal and cytotoxic activities (Boonnak, Karalai *et al.*, 2006; 2007; 2009). Further isolation of materials from the roots of this plant resulted in the title xanthone known as vieillardiixanthone B (Hay *et al.*, 2008). It was tested against both Gram-positive and Gram-negative bacteria i.e. *Bacillus subtilis*, *Staphylococcus aureus* TISTR517, *Enterococcus faecalis* TISTR459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC43300, Vancomycin-Risistant *Enterococcus faecalis* (VRE) ATCC 51299, *Streptococcus faecalis*, *Salmonella typhi*, *Shigella sonei* and *Pseudomonas aeruginosa*. Our results showed that the title compound has no antibacterial action against these pathogens. Herein we report the crystal structure of the title xanthone (I).

In compound (I) (Fig. 1), the three ring system [C1–C13/O1] is essentially planar with an r.m.s. deviation of 0.0372 (2) Å from the plane through all non-hydrogen atoms of the three rings and with a maximum deviation of -0.100 (2) Å for atom C4. The O3 and O5 hydroxyl O atoms lie close to this plane with deviations +0.0669 (2) for O3 and +0.1122 (2) Å for O5. The two methoxy groups lie close to the planes of their benzene rings with torsion angles C14–O4–C3–C2 = 3.8 (3)° and C20–O6–C6–C7 = -7.4 (3)°. The 1,1-dimethyl-2-propenyl [C15–C19] substituent is in a (-)-anticlinal conformation as indicated by the torsion angle C4–C15–C18–C19 = -132.1 (2)°. Intramolecular O3—H1O3···O2 and O5—H1O5..O6 hydrogen bonds (Table 1) generate S(6) and S(5) ring motifs, respectively (Bernstein *et al.*, 1995). The bond distances in (I) are within normal ranges (Allen *et al.*, 1987) and comparable to those in a related structure (Boonnak, Chantrapromma & Fun, 2006).

The crystal packing of (I) is stabilized by intermolecular O—H···O hydrogen bonds and weak C—H···O interactions (Table 1). The molecules are linked into infinite one dimensional chains along [010] by O—H···O and C—H···O hydrogen bonds (Fig. 2 and Table 1). π–π interactions with distances $Cg_1\cdots Cg_2$ = 3.6172 (10) Å (symmetry code: x, 1/2-y, z) and $Cg_1\cdots Cg_3$ = 3.6815 (10) Å (symmetry code: x, 1/2-y, -1/2+z) were also observed; Cg_1, Cg_2 and Cg_3 are the centroids of O1/C9–C13, C1–C4/C10–C11 and C5–C8/C12–C13 rings, respectively.

Experimental

The air-dried roots of *C. formosum* ssp. *pruniflorum* (5.00 kg) was extracted with CH_2Cl_2 (2 x 20 L, for a week) at room temperature and was further evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (58.87 g), which was subjected to QCC (Quick Column Chromatography) on silica gel using n-hexane as a first eluent and then increasing the polarity with acetone to give 12 fractions (F1-F12). Fractions F8-F11 were combined and separated by QCC eluting with 30% EtOAc-n-hexane to give 8 subfractions (F8A-F8H). Subfractions F8E and F8F were combined and separated by QCC and eluted with 30% EtOAc-n-hexane to obtain 20 subfractions (F8E1-F8E20). Subfractions F8E10-F8E12 were combined and separated by QCC and eluted with a gradient of CH_2Cl_2-n-hexane to give 12 subfractions (F8E10A-F8E10L). Subfrac-

tion F8E10D was separated by CC (Column Chromatography) eluting with 10% acetone-n-hexane to give 8 subfractions (F8E10D1-F8E10D8). Subfraction F8E10D5 was further purified by CC and eluted with a gradient of CH_2Cl_2-n-hexane to give the title compound as a yellow solid (3.5 mg). Yellow block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystallized from acetone/CH_3OH (9.5:0.5, v/v) after several days (M.p. 486-488 K).

Refinement

Hydroxy H atoms attached to O3 and O5 and H atoms attached to C18 and C19 were located from the difference map and refined isotropically. The remaining H atoms were placed in calculated positions with d(C—H) = 0.93 Å for aromatic and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.72 Å from C15 and the deepest hole is located at 0.72 Å from C5.

Figures

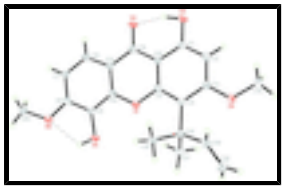


Fig. 1. The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Intramolecular hydrogen bonds are shown as dashed lines.

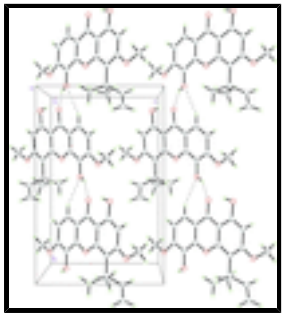


Fig. 2. The crystal packing of (I) viewed along the *a* axis, showing one dimensional chains along the [010] direction. Hydrogen bonds are shown as dashed lines.

1,5-dihydroxy-3,6-dimethoxy-4-(2-methylbut-3-en-2-yl)-9*H*-xanthen-9-one

Crystal data

$C_{20}H_{20}O_6$	$F(000) = 752$
$M_r = 356.36$	$D_x = 1.389$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point = 486–488 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 12.1500$ (4) Å	Cell parameters from 3906 reflections
$b = 14.7396$ (4) Å	$\theta = 2.2$–$27.5°$
$c = 9.5177$ (3) Å	$\mu = 0.10$ mm^{-1}
$\beta = 90.208$ (2)°	$T = 100$ K
$V = 1704.48$ (9) Å^3	Block, yellow
$Z = 4$	0.50 × 0.23 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	3906 independent reflections
Radiation source: sealed tube	2682 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.071$
φ and ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.2°$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -15 \rightarrow 15$
$T_{min} = 0.951$, $T_{max} = 0.978$	$k = -19 \rightarrow 19$
37868 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.054$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.133$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.5536P]$ where $P = (F_o^2 + 2F_c^2)/3$
3906 reflections	$(\Delta/\sigma)_{max} < 0.001$
259 parameters	$\Delta\rho_{max} = 0.27$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.35090 (10)	0.13014 (8)	0.63535 (13)	0.0187 (3)
O2	0.37354 (10)	0.40673 (8)	0.61270 (13)	0.0207 (3)
O3	0.21931 (11)	0.40714 (8)	0.42914 (14)	0.0230 (3)

H1O3	0.278 (2)	0.4208 (18)	0.491 (3)	0.055 (8)*
O4	0.07417 (11)	0.12110 (9)	0.29203 (14)	0.0257 (3)
O5	0.46803 (11)	0.02998 (8)	0.80738 (14)	0.0202 (3)
H1O5	0.522 (2)	0.0062 (16)	0.847 (2)	0.038 (7)*
O6	0.62914 (11)	0.10303 (8)	0.96779 (14)	0.0222 (3)
C1	0.21969 (15)	0.31542 (12)	0.43964 (19)	0.0186 (4)
C2	0.14722 (15)	0.26604 (12)	0.35834 (19)	0.0195 (4)
H2A	0.0986	0.2956	0.2981	0.023*
C3	0.14717 (15)	0.17086 (12)	0.36683 (19)	0.0187 (4)
C4	0.22012 (14)	0.12173 (12)	0.45343 (19)	0.0176 (4)
C5	0.48867 (15)	0.12058 (11)	0.80499 (19)	0.0174 (4)
C6	0.56984 (15)	0.16161 (12)	0.88664 (19)	0.0188 (4)
C7	0.58547 (16)	0.25558 (12)	0.8834 (2)	0.0201 (4)
H7A	0.6400	0.2822	0.9385	0.024*
C8	0.51960 (15)	0.30887 (12)	0.79793 (19)	0.0202 (4)
H8A	0.5291	0.3715	0.7972	0.024*
C9	0.36821 (15)	0.32157 (11)	0.62009 (18)	0.0171 (4)
C10	0.29140 (14)	0.27097 (11)	0.53441 (19)	0.0168 (4)
C11	0.28714 (14)	0.17552 (12)	0.53963 (19)	0.0166 (4)
C12	0.42547 (14)	0.17531 (12)	0.71676 (18)	0.0166 (4)
C13	0.43899 (15)	0.26940 (11)	0.71284 (19)	0.0174 (4)
C14	0.00024 (16)	0.16425 (14)	0.1967 (2)	0.0265 (5)
H14A	−0.0397	0.1190	0.1449	0.040*
H14B	0.0411	0.2015	0.1328	0.040*
H14C	−0.0505	0.2013	0.2483	0.040*
C15	0.22616 (15)	0.01618 (12)	0.45818 (19)	0.0184 (4)
C16	0.34762 (16)	−0.01610 (12)	0.4569 (2)	0.0221 (4)
H16A	0.3505	−0.0789	0.4305	0.033*
H16B	0.3791	−0.0087	0.5488	0.033*
H16C	0.3885	0.0193	0.3904	0.033*
C17	0.16997 (17)	−0.01792 (12)	0.5923 (2)	0.0250 (4)
H17A	0.0927	−0.0044	0.5879	0.038*
H17B	0.2020	0.0116	0.6726	0.038*
H17C	0.1801	−0.0823	0.6004	0.038*
C18	0.17864 (16)	−0.02757 (12)	0.3267 (2)	0.0223 (4)
H18	0.2133 (17)	−0.0069 (14)	0.240 (2)	0.028 (6)*
C19	0.10821 (18)	−0.09584 (14)	0.3216 (3)	0.0316 (5)
H19B	0.0844 (17)	−0.1203 (14)	0.231 (2)	0.030 (6)*
H19A	0.0703 (18)	−0.1196 (15)	0.407 (2)	0.038 (6)*
C20	0.72165 (16)	0.13735 (13)	1.0432 (2)	0.0256 (5)
H20A	0.7565	0.0888	1.0937	0.038*
H20B	0.6977	0.1830	1.1082	0.038*
H20C	0.7731	0.1634	0.9784	0.038*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0254 (7)	0.0110 (6)	0.0195 (7)	−0.0006 (5)	−0.0059 (6)	0.0008 (5)

O2	0.0294 (7)	0.0091 (6)	0.0236 (7)	−0.0007 (5)	−0.0048 (6)	0.0011 (5)
O3	0.0304 (8)	0.0116 (6)	0.0270 (8)	0.0018 (5)	−0.0076 (6)	0.0025 (6)
O4	0.0277 (8)	0.0176 (7)	0.0317 (8)	−0.0013 (5)	−0.0153 (6)	0.0021 (6)
O5	0.0266 (8)	0.0089 (6)	0.0251 (7)	−0.0005 (5)	−0.0072 (6)	0.0011 (5)
O6	0.0281 (7)	0.0123 (6)	0.0259 (7)	−0.0004 (5)	−0.0115 (6)	0.0017 (5)
C1	0.0246 (10)	0.0113 (8)	0.0199 (10)	0.0003 (7)	0.0024 (8)	0.0016 (7)
C2	0.0219 (10)	0.0171 (9)	0.0195 (10)	0.0023 (7)	−0.0041 (8)	0.0029 (7)
C3	0.0220 (10)	0.0165 (9)	0.0177 (9)	−0.0014 (7)	−0.0019 (8)	0.0002 (7)
C4	0.0214 (10)	0.0131 (8)	0.0183 (9)	0.0001 (7)	0.0005 (8)	0.0013 (7)
C5	0.0233 (10)	0.0100 (8)	0.0188 (9)	0.0000 (7)	0.0004 (8)	−0.0003 (7)
C6	0.0229 (10)	0.0150 (9)	0.0185 (10)	0.0016 (7)	−0.0042 (8)	0.0008 (7)
C7	0.0254 (10)	0.0130 (9)	0.0219 (10)	−0.0024 (7)	−0.0048 (8)	−0.0023 (8)
C8	0.0280 (10)	0.0100 (8)	0.0225 (10)	0.0003 (7)	−0.0009 (8)	−0.0002 (7)
C9	0.0225 (10)	0.0125 (8)	0.0164 (9)	0.0002 (7)	0.0017 (8)	−0.0001 (7)
C10	0.0210 (9)	0.0117 (8)	0.0177 (9)	0.0007 (7)	−0.0011 (8)	−0.0007 (7)
C11	0.0199 (9)	0.0132 (8)	0.0167 (9)	0.0020 (7)	−0.0011 (8)	0.0024 (7)
C12	0.0195 (9)	0.0141 (9)	0.0162 (9)	−0.0010 (7)	−0.0022 (7)	−0.0023 (7)
C13	0.0228 (10)	0.0122 (8)	0.0172 (9)	−0.0003 (7)	−0.0008 (8)	−0.0010 (7)
C14	0.0279 (11)	0.0258 (10)	0.0256 (11)	−0.0033 (8)	−0.0101 (9)	0.0040 (9)
C15	0.0234 (10)	0.0118 (8)	0.0201 (10)	−0.0012 (7)	−0.0023 (8)	0.0008 (7)
C16	0.0289 (11)	0.0115 (8)	0.0258 (10)	−0.0002 (7)	−0.0029 (8)	−0.0021 (8)
C17	0.0343 (11)	0.0147 (9)	0.0262 (10)	−0.0024 (8)	0.0029 (9)	0.0020 (8)
C18	0.0259 (10)	0.0155 (9)	0.0255 (10)	0.0021 (8)	−0.0032 (9)	−0.0012 (8)
C19	0.0346 (12)	0.0218 (10)	0.0384 (13)	−0.0040 (9)	−0.0087 (11)	−0.0066 (10)
C20	0.0266 (11)	0.0205 (10)	0.0295 (11)	0.0007 (8)	−0.0121 (9)	−0.0006 (8)

Geometric parameters (Å, °)

O1—C12	1.364 (2)	C8—H8A	0.9300
O1—C11	1.368 (2)	C9—C10	1.445 (2)
O2—C9	1.259 (2)	C9—C13	1.451 (2)
O3—C1	1.356 (2)	C10—C11	1.409 (2)
O3—H1O3	0.94 (3)	C12—C13	1.397 (2)
O4—C3	1.352 (2)	C14—H14A	0.9600
O4—C14	1.424 (2)	C14—H14B	0.9600
O5—C5	1.359 (2)	C14—H14C	0.9600
O5—H1O5	0.83 (3)	C15—C18	1.520 (3)
O6—C6	1.363 (2)	C15—C17	1.535 (3)
O6—C20	1.424 (2)	C15—C16	1.551 (3)
C1—C2	1.378 (3)	C16—H16A	0.9600
C1—C10	1.413 (2)	C16—H16B	0.9600
C2—C3	1.405 (2)	C16—H16C	0.9600
C2—H2A	0.9300	C17—H17A	0.9600
C3—C4	1.409 (2)	C17—H17B	0.9600
C4—C11	1.400 (2)	C17—H17C	0.9600
C4—C15	1.558 (2)	C18—C19	1.322 (3)
C5—C6	1.392 (3)	C18—H18	0.97 (2)
C5—C12	1.393 (2)	C19—H19B	0.98 (2)
C6—C7	1.398 (2)	C19—H19A	1.00 (2)

C7—C8	1.383 (3)	C20—H20A	0.9600
C7—H7A	0.9300	C20—H20B	0.9600
C8—C13	1.396 (3)	C20—H20C	0.9600
C12—O1—C11	120.87 (13)	C5—C12—C13	121.75 (16)
C1—O3—H1O3	99.5 (16)	C8—C13—C12	118.73 (16)
C3—O4—C14	120.28 (15)	C8—C13—C9	123.04 (16)
C5—O5—H1O5	106.1 (16)	C12—C13—C9	118.23 (16)
C6—O6—C20	118.38 (13)	O4—C14—H14A	109.5
O3—C1—C2	118.90 (16)	O4—C14—H14B	109.5
O3—C1—C10	120.77 (16)	H14A—C14—H14B	109.5
C2—C1—C10	120.32 (16)	O4—C14—H14C	109.5
C1—C2—C3	119.70 (17)	H14A—C14—H14C	109.5
C1—C2—H2A	120.2	H14B—C14—H14C	109.5
C3—C2—H2A	120.2	C18—C15—C17	112.15 (15)
O4—C3—C2	120.79 (16)	C18—C15—C16	102.82 (14)
O4—C3—C4	116.07 (15)	C17—C15—C16	109.42 (15)
C2—C3—C4	123.13 (17)	C18—C15—C4	112.47 (15)
C11—C4—C3	114.54 (16)	C17—C15—C4	109.27 (14)
C11—C4—C15	121.41 (15)	C16—C15—C4	110.54 (14)
C3—C4—C15	124.04 (16)	C15—C16—H16A	109.5
O5—C5—C6	123.24 (16)	C15—C16—H16B	109.5
O5—C5—C12	118.53 (16)	H16A—C16—H16B	109.5
C6—C5—C12	118.23 (16)	C15—C16—H16C	109.5
O6—C6—C5	114.41 (15)	H16A—C16—H16C	109.5
O6—C6—C7	124.65 (16)	H16B—C16—H16C	109.5
C5—C6—C7	120.93 (17)	C15—C17—H17A	109.5
C8—C7—C6	119.81 (17)	C15—C17—H17B	109.5
C8—C7—H7A	120.1	H17A—C17—H17B	109.5
C6—C7—H7A	120.1	C15—C17—H17C	109.5
C7—C8—C13	120.51 (16)	H17A—C17—H17C	109.5
C7—C8—H8A	119.7	H17B—C17—H17C	109.5
C13—C8—H8A	119.7	C19—C18—C15	126.7 (2)
O2—C9—C10	121.10 (16)	C19—C18—H18	119.3 (12)
O2—C9—C13	122.15 (16)	C15—C18—H18	113.4 (12)
C10—C9—C13	116.75 (15)	C18—C19—H19B	120.2 (13)
C11—C10—C1	117.58 (16)	C18—C19—H19A	122.6 (13)
C11—C10—C9	121.29 (16)	H19B—C19—H19A	116.8 (18)
C1—C10—C9	121.10 (15)	O6—C20—H20A	109.5
O1—C11—C4	116.11 (15)	O6—C20—H20B	109.5
O1—C11—C10	119.40 (15)	H20A—C20—H20B	109.5
C4—C11—C10	124.49 (16)	O6—C20—H20C	109.5
O1—C12—C5	115.05 (15)	H20A—C20—H20C	109.5
O1—C12—C13	123.19 (16)	H20B—C20—H20C	109.5
O3—C1—C2—C3	179.39 (16)	C3—C4—C11—C10	−5.7 (3)
C10—C1—C2—C3	−2.0 (3)	C15—C4—C11—C10	175.57 (17)
C14—O4—C3—C2	3.8 (3)	C1—C10—C11—O1	−176.47 (15)
C14—O4—C3—C4	−177.21 (16)	C9—C10—C11—O1	5.1 (3)
C1—C2—C3—O4	177.55 (16)	C1—C10—C11—C4	2.8 (3)

C1—C2—C3—C4	−1.4 (3)	C9—C10—C11—C4	−175.67 (17)
O4—C3—C4—C11	−174.00 (15)	C11—O1—C12—C5	−177.07 (15)
C2—C3—C4—C11	5.0 (3)	C11—O1—C12—C13	2.6 (2)
O4—C3—C4—C15	4.7 (3)	O5—C5—C12—O1	−2.9 (2)
C2—C3—C4—C15	−176.34 (17)	C6—C5—C12—O1	177.22 (15)
C20—O6—C6—C5	173.27 (16)	O5—C5—C12—C13	177.41 (16)
C20—O6—C6—C7	−7.4 (3)	C6—C5—C12—C13	−2.5 (3)
O5—C5—C6—O6	1.3 (3)	C7—C8—C13—C12	0.6 (3)
C12—C5—C6—O6	−178.80 (15)	C7—C8—C13—C9	−179.37 (17)
O5—C5—C6—C7	−178.04 (17)	O1—C12—C13—C8	−178.41 (16)
C12—C5—C6—C7	1.8 (3)	C5—C12—C13—C8	1.3 (3)
O6—C6—C7—C8	−179.31 (17)	O1—C12—C13—C9	1.6 (3)
C5—C6—C7—C8	0.0 (3)	C5—C12—C13—C9	−178.73 (16)
C6—C7—C8—C13	−1.2 (3)	O2—C9—C13—C8	−1.6 (3)
O3—C1—C10—C11	179.96 (16)	C10—C9—C13—C8	177.72 (16)
C2—C1—C10—C11	1.3 (3)	O2—C9—C13—C12	178.43 (17)
O3—C1—C10—C9	−1.6 (3)	C10—C9—C13—C12	−2.3 (2)
C2—C1—C10—C9	179.76 (17)	C11—C4—C15—C18	−160.22 (17)
O2—C9—C10—C11	178.32 (17)	C3—C4—C15—C18	21.2 (2)
C13—C9—C10—C11	−1.0 (3)	C11—C4—C15—C17	74.6 (2)
O2—C9—C10—C1	−0.1 (3)	C3—C4—C15—C17	−104.0 (2)
C13—C9—C10—C1	−179.36 (16)	C11—C4—C15—C16	−45.9 (2)
C12—O1—C11—C4	174.78 (15)	C3—C4—C15—C16	135.50 (18)
C12—O1—C11—C10	−5.9 (2)	C17—C15—C18—C19	−8.8 (3)
C3—C4—C11—O1	173.53 (15)	C16—C15—C18—C19	108.6 (2)
C15—C4—C11—O1	−5.2 (2)	C4—C15—C18—C19	−132.5 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1O3···O2	0.95 (3)	1.65 (3)	2.5573 (18)	160 (2)
O5—H1O5···O6	0.83 (2)	2.25 (2)	2.7019 (19)	115 (2)
O5—H1O5···O2^{i}	0.83 (2)	1.98 (2)	2.7520 (18)	155 (2)
C8—H8A···O5^{ii}	0.93	2.54	3.413 (2)	157

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+3/2; (ii) −*x*+1, *y*+1/2, −*z*+3/2.

Fig. 1

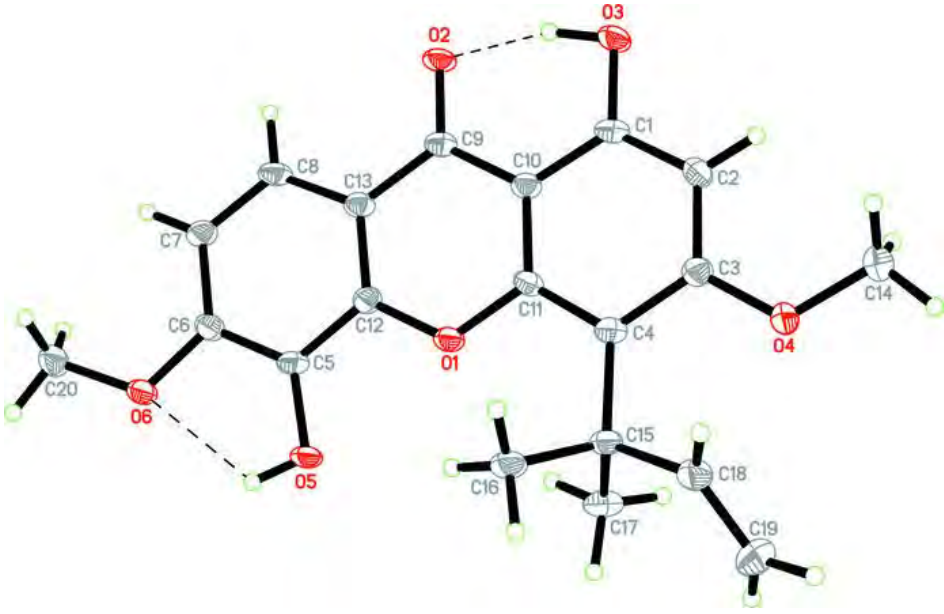
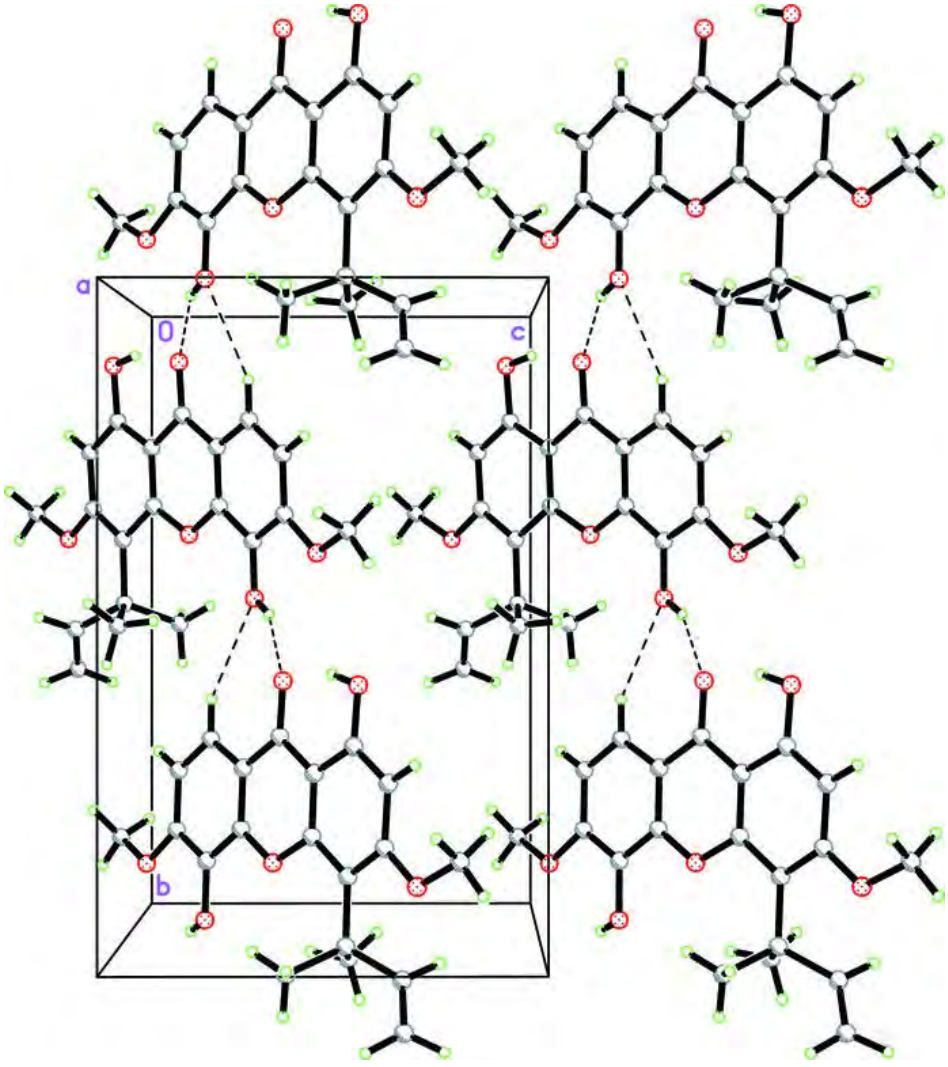


Fig. 2

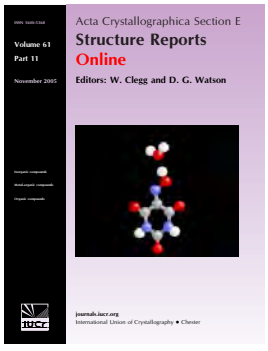


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

Brasilixanthone

Suchada Chantrapromma, Nawong Boonnak, Hoong-Kun Fun, Chatchanok Karalai and Kan Chantrapromma

Acta Cryst. (2010). E**66**, o2066–o2067



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Brasilixanthone[1]

Suchada Chantrapromma,[a]*§ Nawong Boonnak,[a] Hoong-Kun Fun,[b]¶ Chatchanok Karalai[a] and Kan Chantrapromma[c]

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [c]Utilization of Natural Products Research Unit, Walailak University, Thasala, Nakhon Si Thammarat 80160, Thailand
Correspondence e-mail: suchada.c@psu.ac.th

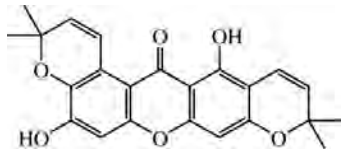
Received 4 July 2010; accepted 9 July 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.012 Å; R factor = 0.103; wR factor = 0.270; data-to-parameter ratio = 7.8.

The title xanthone [systematic name: 5,13-dihydroxy-3,3,-10,10-tetramethyl-3*H*-dipyrano[3,2-*a*:2′,3′-*i*]xanthen-14(10*H*)-one], $C_{23}H_{20}O_6$, was isolated from the roots of *Cratoxylum formosum* ssp. *pruniflorum*. There are two molecules (*A* and *B*) in the asymmetric unit, which show chemical but not crystallographic inversion symmetry. The xanthone skeleton in both molecules is approximately planar, with an r.m.s. deviation of 0.0326 (9) Å for molecule *A* and 0.0355 (9) Å for molecule *B* from the plane through the 14 non-H atoms. The pyran rings in both molecules adopt sofa conformations. Intramolecular O—H···O hydrogen bonds generate *S*(5) and *S*(6) ring motifs. Viewed onto the *bc* plane, the crystal structure resembles a herringbone pattern. Stacks of molecules are stabilized by π–π interactions with centroid–centroid distances of 3.600 (5) Å. The crystal structure is further stabilized by weak C—H···O and C—H···π interactions.

Related literature

For details of hydrogen-bond motifs, see: Bernstein *et al.* (1995) and for ring conformations, see: Cremer & Pople (1975). For background to xanthones and their biological activities, see: Boonnak *et al.* (2006, 2007, 2009); Hay *et al.* (2008); Mahabusarakum *et al.* (1983); Marques *et al.* (2000); Molinar-Toribio *et al.* (2006); Phongpaichit *et al.* (1994); Yu *et al.* (2007). For a related structure, see: Fun *et al.* (2006). For the stability of the temperature controller used in the data collection, see Cosier & Glazer (1986).



Experimental

Crystal data

$C_{23}H_{20}O_6$
M_r = 392.39
Monoclinic, *Pc*
a = 7.5842 (3) Å
b = 12.2937 (4) Å
c = 19.6023 (6) Å
β = 96.827 (2)°
V = 1814.72 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.37 × 0.13 × 0.07 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.963, T_{max} = 0.993
33718 measured reflections
3554 independent reflections
2954 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.103
$wR(F^2)$ = 0.270
S = 1.04
3554 reflections
453 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*5 and *Cg*20 are the centroids of C5*A*–C8*A*/C12*A*/C13*A* and C5*B*–C8*B*/C12*B*/C13*B* rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
O3*A*—H3*AA*···O2*A*	0.82	1.84	2.571 (9)	147
O5*A*—H5*AA*···O4*A*	0.82	2.19	2.656 (9)	116
O3*B*—H3*BA*···O2*B*	0.82	1.84	2.559 (9)	146
O5*B*—H5*BA*···O4*B*	0.82	2.15	2.628 (8)	117
C15*B*—H15*B*···O2*A*^{i}	0.93	2.60	3.514 (11)	168
C16*A*—H16*A*···O2*A*	0.93	2.29	2.879 (11)	121
C16*B*—H16*B*···O2*B*	0.93	2.31	2.890 (10)	120
C17*B*—H17*E*···O5*B*^{i}	0.96	2.58	3.441 (10)	149
C23*B*—H23*D*···O1*A*^{ii}	0.96	2.59	3.370 (11)	139
C18*A*—H18*B*···*Cg*20	0.96	2.75	3.611 (10)	150
C18*B*—H18*D*···*Cg*5	0.96	2.79	3.662 (9)	152

Symmetry codes: (i) $x-1, y, z$; (ii) $x+1, y-1, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for the research grant (RSA 5280033). Prince of Songkla University and the Koshinocorporation Group, Japan, are also acknowledged. NB thanks the Development and Promotion of Science and Technology Talents Project for a study grant. Financial support from Universiti Sains Malaysia for the

[1] This paper is dedicated to Her Royal Highness Princess Chulabhorn Walailak of Thailand on the occasion of her 53rd Birthday Anniversary which fell on July 4th, 2010.

§ Thomson Reuters ResearcherID: A-5085-2009.
¶ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Research University Golden Goose grant No. 1001/PFIZIK/811012 is greatly appreciated.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5295).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Fun, H.-K., Kanjana-Opas, A., Chantrapromma, K. & Kato, S. (2009). *Tetrahedron*, **65**, 3003–3013.

Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Fun, H.-K., Kanjana-Opas, A. & Laphookhieo, S. (2006). *Tetrahedron*, **62**, 8850–8859.

Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Kanjana-Opas, A., Chantrapromma, K. & Fun, H.-K. (2007). *Can. J. Chem.* **85**, 341–345.

Bruker (2005). *APEX2* , *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Fun, H.-K., Ng, S.-L., Razak, I. A., Boonnak, N. & Chantrapromma, S. (2006). *Acta Cryst.* E**62**, o130–o132.

Hay, A. E., Merza, J., Landreau, A., Litaudon, M., Pagniez, F., Pape, P. L. & Richomme, P. (2008). *Fitoterapia*, **79**, 42–46.

Mahabusarakum, W., Phongpaichit, S., Jansakul, C. & Wiriyachitra, P. (1983). *Songklanakarin J. Sci. Technol.* **5**, 337–339.

Marques, V. L. L., Oliveira, F. M. D., Conserva, L. M., Brito, R. G. L. & Guilhon, G. M. S. P. (2000). *Phytochemistry*, **55**, 815–818.

Molinar-Toribio, E., González, J., Ortega-Barría, E., Capson, T. L., Coley, P. D., Kursar, T. A., McPhail, K. & Cubilla-Rios, L. (2006). *Pharm. Biol.* **44**, 550–553.

Phongpaichit, S., Nilrat, L., Tharavichitkul, P., Bunchoo, S., Chuaprapaisilp, T. & Wiriyachitra, P. (1994). *Songklanakarin J. Sci. Technol.* **16**, 399–405.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Yu, L., Zhao, M., Yang, B., Zhao, Q. & Jiang, Y. (2007). *Food Chem.* **104**, 176–181.

supplementary materials

Acta Cryst. (2010). E**66**, o2066-o2067 [**doi:10.1107/S1600536810027285**]

Brasilixanthone

S. Chantrapromma, N. Boonnak, H.-K. Fun, C. Karalai and K. Chantrapromma

Comment

Xanthones are secondary metabolites extracted from several plants and have demonstrated to possess considerable biological properties such as antibacterial, antioxidant, antiprotozoal and cytotoxic activities (Boonnak *et al.*, 2006; 2007; 2009; Mahabusarakum *et al.*, 1983; Molinar-Toribio *et al.*, 2006; Phongpaichit *et al.*, 1994; Yu *et al.*, 2007). During the course of our investigation of the chemical constituents and bioactive compounds from the *Cratoxylum formosum* ssp. *pruniflorum*, a Thai medicinal plant, the title xanthone (I) namely brasilixanthone (Marques *et al.*, 2000) was isolated from the roots of this plant. It was tested against fungi (*Candida albicans*) and both ordinary and antibiotic-resistant bacterial strains such as *Bacillus subtilis*, *Staphylococcus aureus* TISTR517, *Enterococcus faecalis* TISTR459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC43300, Vancomycin-Risistant *Enterococcus faecalis* (VRE) ATCC 51299, *Salmonella typhi*, *Shigella sonei* and *Pseudomonas aeruginosa*. Our results showed that (I) does not possess antifungal and antibacterial activities against the tested pathogens with the MIC (Minimum Inhibition Concentration) > 300 µg/mol. Herein we report the crystal structure of (I).

Compound (I) crystallizes with two independent molecules (*A* and *B*) per asymmetric (Fig. 1). The conformations of molecule *A* differ from those observed in molecule *B* in which the two chromene rings in *A* pucker in the opposite direction from those in *B* (Fig. 1). In both molecules, the three ring system [C1–C13/O1] are essentially planar with the *r.m.s*. deviation of 0.0326 (9) and 0.0355 (9) Å, respectively for *A* and *B* from the plane through all 14 non-hydrogen atoms of the three rings and with a maximum deviation of -0.085 (9) Å (for *A*) and +0.081 (9) Å (for *B*) for atom C3. The O3 and O5 hydroxyl O atoms lie close to this plane with deviations +0.003 (6) for O3 and +0.023 (6) Å for O5 (in *A*) [the corresponding values are -0.018 (8) and -0.030 (1) Å in *B*]. The two chromene rings in *A*, (C1A-C2A/C14A–C16A/O4A; angular fashion chromene) and (C6A-C7A/C19A–C21A/O6A; linear fashion chromene), adopt screw-boat conformations (Cremer & Pople, 1975) with the puckering atoms C14A [-0.270 (10) Å] and O4A [+0.231 (7) Å] from the mean plane of C1A/C2A/C15A/C16A; and puckering atoms C19A [-0.273 (9) Å] and O6A [+0.225 (7) Å] from the C6A/C7A/C21A/C22A plane, with the puckering parameters Q=0.415 (9)Å, θ=69.5 (12)° and φ=320.1 (14)° for C1A-C2A/C14A–C16A/O4A and Q=0.402 (9)Å, θ=62.6 (13)° and φ=316.4 (16)° for C6A-C7A/C19A–C21A/O6A rings in *A*. In molecule *B* the two chromene rings are in twisted boat conformations with the corresponding parameters of 0.421 (8)Å: 116.2 (11)°:142.7 (13)° and 0.368 (8)Å: 111.4 (14)°:140.3 (15)° respectively for the angular fashion C1B-C2B/C14B–C16B/O4B and the linear fashion C6B-C7B/C19B–C21B/O6B chromene rings with puckering atoms, C14B [+0.277 (8) Å], O4B [-0.250 (7) Å], C19B [+0.240 (9) Å] and O6B [-0.207 (7) Å]. Interestingly, the two chromene rings (angular and linear fashion chromenes) in *A* pucker in opposite directions and these puckering parameters are also opposite compared with those in *B* (Fig. 2) which is the cause that the two molecules *A* and *B* differ in their conformations.

Intramolecular O—H···O hydrogen bonds (Table 1) involving O3A and O3B hydroxy O atoms generate S(6) whereas the one involving O5A and O5B atoms generate S(5) ring motifs (Fig. 1) (Bernstein *et al.*, 1995). There are weak intramolecular C—H···O interactions in the crystal structure, [C16A—H16A···O2A and C16B—H16B···O2B], which generate two S(6) ring motifs. The bond distances in (I) are comparable to those in a related structure (Fun *et al.*, 2006).

The crystal packing of (I) is stabilized by weak C—H···O interactions (Table 1). The molecules are arranged into zig-zag chains along the *c* axis (Fig. 2) These chains are stacked along the *b* axis by π–π interactions with distance $Cg_5\cdots Cg_{20}$ = 3.600 (5) Å (symmetry code: x, -1+y, z); Cg_5 and Cg_{20} are the centroids of C5A–C8A/C12A-C13A and C5B–C8B/C12B–C13B rings, respectively. C—H···π interactions were also observed (Table 1).

Experimental

The air-dried roots of *C. formosum* ssp. *pruniflorum* (5.00 kg) was extracted with CH_2Cl_2 (2 x 20 L, for a week) at room temperature and was further evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (58.87 g), which was subjected to QCC (Quick Column Chromatography) on silica gel using n-hexane as a first eluent and then increasing the polarity with acetone to give 12 fractions (F1-F12). Fractions F8-F11 were combined and separated by QCC eluting with 30% EtOAc-n-hexane to give 8 subfractions (F8A-F8H). Subfractions F8E and F8F were combined and then separated by QCC and eluted with 30% EtOAc-n-hexane to obtain 20 subfractions (F8E1-F8E20). Subfraction F8E10-F8E12 were combined and then separated by QCC and eluted with a gradient of CH_2Cl_2-n-hexane to give 12 subfractions (F8E10A-F8E10L). Subfraction FR8E10B was further purified by CC (Column Chromatography) and eluted with 5% acetone-n-hexane to give the title compound as yellow solid (4.5 mg). Yellow needle-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystallized from acetone/CH_3OH (9.5:0.5, v/v) after several days (M.p. 478-480 K).

Refinement

All H atoms were placed in calculated positions with d(O—H) = 0.82 Å and d(C—H) = 0.93 Å for aromatic and CH, and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.85 Å from O2A and the deepest hole is located at 0.76 Å from O1A. A total of 3445 Friedel pairs were merged before final refinement as there is no large anomalous dispersion for the determination of the absolute structure.

Figures

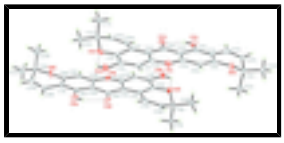


Fig. 1. The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Intramolecular O—H···O hydrogen bonds are shown as dashed lines.

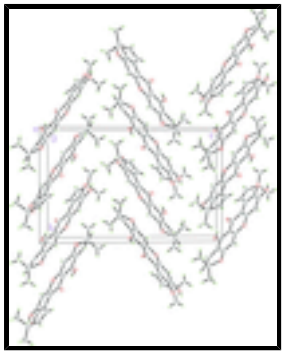


Fig. 2. The crystal packing of (I) viewed along the *a* axis, showing zig-zag chains stacked along the *b* axis.

5,13-dihydroxy-3,3,10,10-tetramethyl-3*H*- dipyrano[3,2-*a*:2',3'-*i*]xanthen-14(10*H*)-one

Crystal data

$C_{23}H_{20}O_6$	$F(000) = 824$
$M_r = 392.39$	$D_x = 1.436$ Mg m^{-3}
Monoclinic, *Pc*	Melting point = 478–480 K
Hall symbol: P -2yc	Mo *K*α radiation, λ = 0.71073 Å
$a = 7.5842$ (3) Å	Cell parameters from 3554 reflections
$b = 12.2937$ (4) Å	θ = 2.0–26.0°
$c = 19.6023$ (6) Å	μ = 0.10 mm^{-1}
β = 96.827 (2)°	$T = 100$ K
$V = 1814.72$ (11) Å^3	Needle, yellow
$Z = 4$	0.37 × 0.13 × 0.07 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	3554 independent reflections
Radiation source: sealed tube	2954 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.077$
φ and ω scans	$\theta_{max} = 26.0°$, $\theta_{min} = 2.0°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -9 \rightarrow 9$
$T_{min} = 0.963$, $T_{max} = 0.993$	$k = -15 \rightarrow 15$
33718 measured reflections	$l = -24 \rightarrow 23$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.103$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.270$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.1648P)^2 + 5.5181P]$ where $P = (F_o^2 + 2F_c^2)/3$
3554 reflections	$(\Delta/\sigma)_{max} = 0.001$
453 parameters	$\Delta\rho_{max} = 1.21$ e Å^{-3}
2 restraints	$\Delta\rho_{min} = -0.39$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.2594 (8)	0.6225 (5)	0.5423 (3)	0.0284 (14)
O2A	0.7864 (8)	0.5793 (5)	0.6095 (4)	0.0300 (15)
O3A	0.7794 (8)	0.7513 (5)	0.6837 (3)	0.0299 (15)
H3AA	0.8227	0.6975	0.6674	0.045*
O4A	0.5559 (8)	0.2608 (5)	0.4390 (3)	0.0254 (13)
O5A	0.2102 (8)	0.3007 (5)	0.4131 (3)	0.0289 (14)
H5AA	0.2715	0.2500	0.4024	0.043*
O6A	0.2246 (8)	0.9394 (5)	0.6739 (3)	0.0256 (14)
C1A	0.6044 (13)	0.4207 (7)	0.5107 (5)	0.0251 (19)
C2A	0.4941 (11)	0.3517 (7)	0.4674 (4)	0.0219 (6)
C3A	0.3142 (12)	0.3725 (7)	0.4539 (4)	0.0252 (18)
C4A	0.2407 (11)	0.4679 (7)	0.4795 (4)	0.0219 (6)
H4AA	0.1211	0.4849	0.4685	0.026*
C5A	0.2460 (11)	0.7814 (7)	0.6065 (5)	0.0254 (18)
H5AB	0.1273	0.7895	0.5889	0.031*
C6A	0.3293 (13)	0.8539 (7)	0.6537 (4)	0.0275 (14)
C7A	0.5028 (12)	0.8473 (7)	0.6790 (5)	0.029 (2)
C8A	0.6086 (12)	0.7602 (8)	0.6578 (5)	0.030 (2)
C9A	0.6311 (12)	0.5885 (8)	0.5873 (4)	0.028 (2)
C10A	0.5222 (12)	0.5135 (7)	0.5380 (5)	0.028 (2)
C11A	0.3485 (13)	0.5325 (7)	0.5197 (5)	0.0271 (19)
C12A	0.3513 (13)	0.6930 (8)	0.5859 (4)	0.0275 (14)
C13A	0.5217 (13)	0.6810 (8)	0.6099 (5)	0.029 (2)
C14A	0.7423 (11)	0.2633 (7)	0.4260 (5)	0.0219 (6)
C15A	0.8475 (12)	0.3104 (7)	0.4909 (5)	0.0244 (13)
H15A	0.9617	0.2847	0.5042	0.029*
C16A	0.7838 (12)	0.3844 (7)	0.5280 (4)	0.0244 (13)
H16A	0.8539	0.4139	0.5656	0.029*
C17A	0.7541 (11)	0.3372 (7)	0.3641 (4)	0.0235 (17)
H17A	0.6762	0.3105	0.3256	0.035*
H17B	0.8739	0.3378	0.3529	0.035*
H17C	0.7197	0.4097	0.3750	0.035*
C18A	0.7952 (13)	0.1488 (7)	0.4115 (5)	0.031 (2)
H18A	0.7115	0.1184	0.3760	0.047*
H18B	0.7965	0.1059	0.4525	0.047*

H18C	0.9115	0.1487	0.3968	0.047*
C19A	0.2789 (11)	0.9818 (7)	0.7425 (4)	0.0219 (6)
C20A	0.4757 (11)	0.9990 (8)	0.7499 (4)	0.0219 (6)
H20A	0.5231	1.0607	0.7725	0.026*
C21A	0.5832 (12)	0.9296 (7)	0.7253 (5)	0.0269 (19)
H21A	0.7055	0.9332	0.7372	0.032*
C22A	0.2221 (11)	0.9032 (7)	0.7950 (4)	0.0219 (6)
H22A	0.2928	0.8383	0.7954	0.033*
H22B	0.0992	0.8849	0.7832	0.033*
H22C	0.2384	0.9363	0.8396	0.033*
C23A	0.1815 (13)	1.0908 (8)	0.7424 (5)	0.030 (2)
H23A	0.2025	1.1318	0.7025	0.046*
H23B	0.2245	1.1310	0.7830	0.046*
H23C	0.0564	1.0781	0.7417	0.046*
O1B	0.7714 (6)	0.1279 (5)	0.6348 (3)	0.0170 (11)
O2B	0.2460 (7)	0.1655 (5)	0.5601 (3)	0.0224 (13)
O3B	0.2679 (7)	−0.0013 (5)	0.4835 (3)	0.0243 (14)
H3BA	0.2190	0.0505	0.4997	0.036*
O4B	0.4578 (7)	0.4885 (5)	0.7314 (3)	0.0234 (13)
O5B	0.7974 (7)	0.4482 (5)	0.7649 (3)	0.0265 (14)
H5BA	0.7316	0.4976	0.7742	0.032*
O6B	0.8254 (8)	−0.1813 (5)	0.5014 (3)	0.0230 (13)
C1B	0.4180 (11)	0.3280 (7)	0.6589 (4)	0.0219 (6)
C2B	0.5194 (11)	0.3932 (7)	0.7039 (4)	0.0219 (6)
C3B	0.7035 (11)	0.3754 (7)	0.7233 (4)	0.0195 (16)
C4B	0.7810 (10)	0.2841 (7)	0.6989 (4)	0.0186 (16)
H4BA	0.9004	0.2689	0.7120	0.022*
C5B	0.7971 (10)	−0.0268 (6)	0.5681 (4)	0.0168 (9)
H5BB	0.9150	−0.0335	0.5871	0.020*
C6B	0.7222 (10)	−0.1000 (7)	0.5196 (4)	0.0168 (9)
C7B	0.5435 (10)	−0.0911 (6)	0.4902 (4)	0.0177 (11)
C8B	0.4416 (10)	−0.0059 (6)	0.5117 (4)	0.0139 (15)
C9B	0.4054 (10)	0.1592 (6)	0.5834 (4)	0.0155 (15)
C10B	0.4968 (9)	0.2335 (7)	0.6322 (4)	0.0158 (16)
C11B	0.6794 (10)	0.2163 (7)	0.6551 (4)	0.0168 (9)
C12B	0.6909 (11)	0.0571 (6)	0.5876 (4)	0.0177 (11)
C13B	0.5122 (10)	0.0696 (7)	0.5609 (4)	0.0161 (16)
C14B	0.2689 (10)	0.4816 (6)	0.7418 (4)	0.0145 (15)
C15B	0.1709 (11)	0.4396 (7)	0.6785 (4)	0.0188 (16)
H15B	0.0594	0.4685	0.6640	0.023*
C16B	0.2338 (10)	0.3616 (7)	0.6407 (4)	0.0201 (17)
H16B	0.1629	0.3299	0.6041	0.024*
C17B	0.2476 (11)	0.4105 (7)	0.8039 (4)	0.0213 (13)
H17D	0.3047	0.3417	0.7990	0.032*
H17E	0.1236	0.3989	0.8069	0.032*
H17F	0.3009	0.4459	0.8448	0.032*
C18B	0.2195 (11)	0.6005 (7)	0.7543 (4)	0.0213 (13)
H18D	0.2315	0.6426	0.7139	0.032*
H18E	0.2972	0.6291	0.7923	0.032*

H18F	0.0990	0.6040	0.7645	0.032*
C19B	0.7833 (11)	−0.2314 (7)	0.4314 (4)	0.0216 (18)
C20B	0.5868 (11)	−0.2446 (7)	0.4176 (5)	0.0244 (18)
H20B	0.5415	−0.3022	0.3901	0.029*
C21B	0.4738 (10)	−0.1771 (7)	0.4431 (5)	0.0240 (18)
H21B	0.3520	−0.1846	0.4311	0.029*
C22B	0.8534 (13)	−0.1520 (8)	0.3810 (5)	0.034 (2)
H22D	0.9794	−0.1439	0.3921	0.050*
H22E	0.7969	−0.0826	0.3842	0.050*
H22F	0.8279	−0.1797	0.3351	0.050*
C23B	0.8837 (12)	−0.3365 (7)	0.4352 (5)	0.028 (2)
H23D	1.0078	−0.3223	0.4477	0.042*
H23E	0.8670	−0.3718	0.3912	0.042*
H23F	0.8408	−0.3828	0.4690	0.042*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.024 (3)	0.022 (3)	0.040 (4)	0.005 (2)	0.004 (3)	0.004 (3)
O2A	0.017 (3)	0.029 (3)	0.042 (4)	0.002 (3)	−0.002 (3)	0.007 (3)
O3A	0.018 (3)	0.037 (4)	0.034 (4)	0.013 (3)	0.001 (3)	−0.001 (3)
O4A	0.021 (3)	0.018 (3)	0.038 (4)	−0.006 (2)	0.005 (3)	0.002 (3)
O5A	0.023 (3)	0.025 (3)	0.037 (4)	−0.006 (2)	−0.002 (3)	−0.007 (3)
O6A	0.029 (3)	0.021 (3)	0.026 (3)	0.004 (3)	0.002 (3)	−0.002 (3)
C1A	0.043 (5)	0.012 (4)	0.023 (4)	−0.009 (4)	0.017 (4)	−0.003 (3)
C2A	0.0241 (14)	0.0209 (14)	0.0229 (14)	0.0040 (11)	0.0117 (11)	0.0043 (11)
C3A	0.035 (5)	0.020 (4)	0.021 (4)	−0.005 (3)	0.006 (4)	0.003 (3)
C4A	0.0241 (14)	0.0209 (14)	0.0229 (14)	0.0040 (11)	0.0117 (11)	0.0043 (11)
C5A	0.015 (4)	0.027 (4)	0.035 (5)	0.004 (3)	0.006 (3)	−0.002 (4)
C6A	0.046 (4)	0.025 (3)	0.013 (3)	−0.013 (3)	0.008 (3)	0.001 (2)
C7A	0.028 (5)	0.024 (4)	0.039 (5)	0.010 (4)	0.023 (4)	0.017 (4)
C8A	0.026 (4)	0.034 (5)	0.029 (5)	−0.018 (4)	0.004 (4)	0.002 (4)
C9A	0.028 (5)	0.035 (5)	0.019 (4)	−0.022 (4)	0.003 (4)	−0.001 (4)
C10A	0.035 (5)	0.017 (4)	0.036 (5)	0.008 (4)	0.027 (4)	0.015 (4)
C11A	0.037 (5)	0.023 (4)	0.023 (4)	−0.007 (4)	0.013 (4)	0.013 (3)
C12A	0.046 (4)	0.025 (3)	0.013 (3)	−0.013 (3)	0.008 (3)	0.001 (2)
C13A	0.033 (5)	0.023 (5)	0.032 (5)	0.003 (4)	0.011 (4)	0.005 (4)
C14A	0.0241 (14)	0.0209 (14)	0.0229 (14)	0.0040 (11)	0.0117 (11)	0.0043 (11)
C15A	0.034 (3)	0.019 (3)	0.021 (3)	−0.012 (2)	0.005 (2)	−0.001 (2)
C16A	0.034 (3)	0.019 (3)	0.021 (3)	−0.012 (2)	0.005 (2)	−0.001 (2)
C17A	0.021 (4)	0.029 (4)	0.021 (4)	−0.003 (3)	0.004 (3)	−0.002 (3)
C18A	0.043 (5)	0.022 (4)	0.030 (5)	0.010 (4)	0.011 (4)	−0.003 (4)
C19A	0.0241 (14)	0.0209 (14)	0.0229 (14)	0.0040 (11)	0.0117 (11)	0.0043 (11)
C20A	0.0241 (14)	0.0209 (14)	0.0229 (14)	0.0040 (11)	0.0117 (11)	0.0043 (11)
C21A	0.031 (5)	0.027 (5)	0.023 (4)	0.013 (4)	0.006 (3)	0.012 (4)
C22A	0.0241 (14)	0.0209 (14)	0.0229 (14)	0.0040 (11)	0.0117 (11)	0.0043 (11)
C23A	0.035 (5)	0.030 (5)	0.030 (5)	0.010 (4)	0.021 (4)	0.001 (4)
O1B	0.011 (2)	0.028 (3)	0.011 (3)	−0.001 (2)	0.001 (2)	−0.004 (2)

O2B	0.017 (3)	0.035 (3)	0.016 (3)	0.004 (2)	0.003 (2)	−0.003 (2)
O3B	0.015 (3)	0.033 (3)	0.023 (3)	0.005 (2)	−0.005 (2)	−0.007 (3)
O4B	0.017 (3)	0.018 (3)	0.036 (3)	0.007 (2)	0.010 (2)	−0.009 (3)
O5B	0.018 (3)	0.024 (3)	0.039 (4)	0.001 (2)	0.006 (3)	−0.009 (3)
O6B	0.028 (3)	0.017 (3)	0.023 (3)	0.004 (2)	−0.005 (2)	−0.010 (2)
C1B	0.0241 (14)	0.0209 (14)	0.0229 (14)	0.0040 (11)	0.0117 (11)	0.0043 (11)
C2B	0.0241 (14)	0.0209 (14)	0.0229 (14)	0.0040 (11)	0.0117 (11)	0.0043 (11)
C3B	0.023 (4)	0.022 (4)	0.015 (4)	0.000 (3)	0.007 (3)	−0.007 (3)
C4B	0.010 (3)	0.030 (4)	0.016 (4)	0.001 (3)	0.004 (3)	0.007 (3)
C5B	0.016 (2)	0.018 (2)	0.017 (2)	0.0005 (17)	0.0055 (17)	−0.0026 (18)
C6B	0.016 (2)	0.018 (2)	0.017 (2)	0.0005 (17)	0.0055 (17)	−0.0026 (18)
C7B	0.025 (3)	0.012 (2)	0.017 (3)	0.003 (2)	0.006 (2)	−0.006 (2)
C8B	0.018 (4)	0.009 (3)	0.016 (3)	0.000 (3)	0.008 (3)	0.004 (3)
C9B	0.018 (4)	0.013 (4)	0.016 (4)	0.005 (3)	0.004 (3)	0.005 (3)
C10B	0.012 (3)	0.026 (4)	0.009 (3)	0.000 (3)	0.000 (3)	−0.004 (3)
C11B	0.016 (2)	0.018 (2)	0.017 (2)	0.0005 (17)	0.0055 (17)	−0.0026 (18)
C12B	0.025 (3)	0.012 (2)	0.017 (3)	0.003 (2)	0.006 (2)	−0.006 (2)
C13B	0.016 (4)	0.026 (4)	0.006 (3)	0.001 (3)	−0.001 (3)	0.003 (3)
C14B	0.013 (3)	0.013 (4)	0.019 (4)	0.006 (3)	0.007 (3)	−0.001 (3)
C15B	0.024 (4)	0.015 (4)	0.016 (4)	0.008 (3)	0.000 (3)	−0.001 (3)
C16B	0.020 (4)	0.021 (4)	0.020 (4)	0.001 (3)	0.010 (3)	0.003 (3)
C17B	0.024 (3)	0.019 (3)	0.023 (3)	0.012 (2)	0.012 (2)	0.004 (2)
C18B	0.024 (3)	0.019 (3)	0.023 (3)	0.012 (2)	0.012 (2)	0.004 (2)
C19B	0.028 (4)	0.028 (4)	0.009 (3)	0.007 (3)	0.000 (3)	−0.008 (3)
C20B	0.023 (4)	0.016 (4)	0.031 (5)	0.002 (3)	−0.007 (3)	−0.006 (3)
C21B	0.009 (3)	0.031 (5)	0.029 (4)	−0.008 (3)	−0.007 (3)	−0.007 (4)
C22B	0.033 (5)	0.040 (5)	0.030 (5)	0.015 (4)	0.016 (4)	0.003 (4)
C23B	0.029 (4)	0.024 (4)	0.031 (5)	0.016 (4)	0.003 (4)	−0.006 (4)

Geometric parameters (Å, °)

O1A—C12A	1.351 (11)	O1B—C12B	1.361 (9)
O1A—C11A	1.395 (11)	O1B—C11B	1.375 (9)
O2A—C9A	1.211 (11)	O2B—C9B	1.242 (9)
O3A—C8A	1.338 (11)	O3B—C8B	1.368 (9)
O3A—H3AA	0.8200	O3B—H3BA	0.8200
O4A—C2A	1.356 (10)	O4B—C2B	1.393 (10)
O4A—C14A	1.467 (10)	O4B—C14B	1.473 (9)
O5A—C3A	1.376 (10)	O5B—C3B	1.355 (10)
O5A—H5AA	0.8200	O5B—H5BA	0.8200
O6A—C6A	1.402 (11)	O6B—C6B	1.343 (9)
O6A—C19A	1.455 (11)	O6B—C19B	1.503 (9)
C1A—C2A	1.405 (13)	C1B—C2B	1.361 (13)
C1A—C16A	1.433 (14)	C1B—C10B	1.434 (11)
C1A—C10A	1.434 (12)	C1B—C16B	1.460 (11)
C2A—C3A	1.382 (12)	C2B—C3B	1.420 (12)
C3A—C4A	1.416 (12)	C3B—C4B	1.379 (11)
C4A—C11A	1.330 (13)	C4B—C11B	1.367 (12)
C4A—H4AA	0.9300	C4B—H4BA	0.9300

C5A—C6A	1.382 (12)	C5B—C6B	1.382 (11)
C5A—C12A	1.434 (12)	C5B—C12B	1.390 (10)
C5A—H5AB	0.9300	C5B—H5BB	0.9300
C6A—C7A	1.352 (14)	C6B—C7B	1.412 (11)
C7A—C8A	1.429 (13)	C7B—C8B	1.397 (10)
C7A—C21A	1.446 (14)	C7B—C21B	1.461 (11)
C8A—C13A	1.454 (13)	C8B—C13B	1.398 (11)
C9A—C13A	1.505 (13)	C9B—C10B	1.439 (11)
C9A—C10A	1.508 (13)	C9B—C13B	1.466 (10)
C10A—C11A	1.344 (14)	C10B—C11B	1.420 (10)
C12A—C13A	1.331 (14)	C12B—C13B	1.402 (11)
C14A—C18A	1.501 (12)	C14B—C15B	1.463 (11)
C14A—C17A	1.527 (12)	C14B—C17B	1.522 (11)
C14A—C15A	1.532 (12)	C14B—C18B	1.535 (10)
C15A—C16A	1.293 (13)	C15B—C16B	1.334 (11)
C15A—H15A	0.9300	C15B—H15B	0.9300
C16A—H16A	0.9300	C16B—H16B	0.9300
C17A—H17A	0.9600	C17B—H17D	0.9600
C17A—H17B	0.9600	C17B—H17E	0.9600
C17A—H17C	0.9600	C17B—H17F	0.9600
C18A—H18A	0.9600	C18B—H18D	0.9600
C18A—H18B	0.9600	C18B—H18E	0.9600
C18A—H18C	0.9600	C18B—H18F	0.9600
C19A—C20A	1.498 (11)	C19B—C20B	1.491 (11)
C19A—C22A	1.511 (11)	C19B—C23B	1.497 (11)
C19A—C23A	1.530 (12)	C19B—C22B	1.528 (13)
C20A—C21A	1.309 (12)	C20B—C21B	1.333 (12)
C20A—H20A	0.9300	C20B—H20B	0.9300
C21A—H21A	0.9300	C21B—H21B	0.9300
C22A—H22A	0.9600	C22B—H22D	0.9600
C22A—H22B	0.9600	C22B—H22E	0.9600
C22A—H22C	0.9600	C22B—H22F	0.9600
C23A—H23A	0.9600	C23B—H23D	0.9600
C23A—H23B	0.9600	C23B—H23E	0.9600
C23A—H23C	0.9600	C23B—H23F	0.9600
C12A—O1A—C11A	118.5 (7)	C12B—O1B—C11B	120.0 (6)
C8A—O3A—H3AA	109.5	C8B—O3B—H3BA	109.5
C2A—O4A—C14A	116.2 (6)	C2B—O4B—C14B	112.4 (6)
C3A—O5A—H5AA	109.5	C3B—O5B—H5BA	109.5
C6A—O6A—C19A	115.0 (7)	C6B—O6B—C19B	118.6 (6)
C2A—C1A—C16A	115.8 (8)	C2B—C1B—C10B	119.1 (8)
C2A—C1A—C10A	116.9 (9)	C2B—C1B—C16B	116.3 (8)
C16A—C1A—C10A	127.0 (9)	C10B—C1B—C16B	124.6 (8)
O4A—C2A—C3A	117.1 (8)	C1B—C2B—O4B	123.8 (8)
O4A—C2A—C1A	122.4 (8)	C1B—C2B—C3B	123.0 (8)
C3A—C2A—C1A	120.5 (8)	O4B—C2B—C3B	113.0 (7)
O5A—C3A—C2A	118.5 (8)	O5B—C3B—C4B	122.1 (7)
O5A—C3A—C4A	121.0 (8)	O5B—C3B—C2B	119.2 (7)
C2A—C3A—C4A	120.5 (8)	C4B—C3B—C2B	118.6 (8)

C11A—C4A—C3A	117.7 (8)	C11B—C4B—C3B	118.9 (7)
C11A—C4A—H4AA	121.2	C11B—C4B—H4BA	120.6
C3A—C4A—H4AA	121.2	C3B—C4B—H4BA	120.6
C6A—C5A—C12A	116.8 (8)	C6B—C5B—C12B	117.9 (7)
C6A—C5A—H5AB	121.6	C6B—C5B—H5BB	121.1
C12A—C5A—H5AB	121.6	C12B—C5B—H5BB	121.1
C7A—C6A—C5A	123.6 (9)	O6B—C6B—C5B	117.5 (7)
C7A—C6A—O6A	120.0 (8)	O6B—C6B—C7B	120.7 (7)
C5A—C6A—O6A	116.3 (8)	C5B—C6B—C7B	121.8 (7)
C6A—C7A—C8A	119.6 (9)	C8B—C7B—C6B	118.3 (7)
C6A—C7A—C21A	120.5 (8)	C8B—C7B—C21B	124.2 (7)
C8A—C7A—C21A	119.9 (8)	C6B—C7B—C21B	117.3 (7)
O3A—C8A—C7A	120.2 (8)	O3B—C8B—C7B	116.7 (7)
O3A—C8A—C13A	122.2 (9)	O3B—C8B—C13B	121.5 (6)
C7A—C8A—C13A	117.5 (9)	C7B—C8B—C13B	121.7 (7)
O2A—C9A—C13A	120.7 (8)	O2B—C9B—C10B	125.2 (7)
O2A—C9A—C10A	127.5 (9)	O2B—C9B—C13B	118.9 (7)
C13A—C9A—C10A	111.8 (8)	C10B—C9B—C13B	115.9 (7)
C11A—C10A—C1A	119.7 (10)	C11B—C10B—C1B	116.0 (7)
C11A—C10A—C9A	120.6 (8)	C11B—C10B—C9B	119.4 (7)
C1A—C10A—C9A	119.7 (8)	C1B—C10B—C9B	124.6 (7)
C4A—C11A—C10A	124.6 (9)	C4B—C11B—O1B	113.3 (7)
C4A—C11A—O1A	112.0 (8)	C4B—C11B—C10B	124.3 (7)
C10A—C11A—O1A	123.3 (9)	O1B—C11B—C10B	122.4 (7)
C13A—C12A—O1A	124.0 (9)	O1B—C12B—C5B	115.7 (7)
C13A—C12A—C5A	122.3 (8)	O1B—C12B—C13B	121.2 (7)
O1A—C12A—C5A	113.7 (8)	C5B—C12B—C13B	123.0 (7)
C12A—C13A—C8A	120.0 (9)	C8B—C13B—C12B	117.3 (7)
C12A—C13A—C9A	121.7 (9)	C8B—C13B—C9B	121.7 (7)
C8A—C13A—C9A	118.2 (8)	C12B—C13B—C9B	121.0 (7)
O4A—C14A—C18A	107.3 (7)	C15B—C14B—O4B	107.9 (6)
O4A—C14A—C17A	107.4 (7)	C15B—C14B—C17B	112.5 (7)
C18A—C14A—C17A	111.4 (7)	O4B—C14B—C17B	110.0 (6)
O4A—C14A—C15A	106.3 (7)	C15B—C14B—C18B	111.2 (7)
C18A—C14A—C15A	112.8 (8)	O4B—C14B—C18B	103.1 (6)
C17A—C14A—C15A	111.2 (7)	C17B—C14B—C18B	111.6 (6)
C16A—C15A—C14A	122.7 (9)	C16B—C15B—C14B	123.1 (7)
C16A—C15A—H15A	118.7	C16B—C15B—H15B	118.5
C14A—C15A—H15A	118.7	C14B—C15B—H15B	118.5
C15A—C16A—C1A	119.6 (8)	C15B—C16B—C1B	117.5 (8)
C15A—C16A—H16A	120.2	C15B—C16B—H16B	121.2
C1A—C16A—H16A	120.2	C1B—C16B—H16B	121.2
C14A—C17A—H17A	109.5	C14B—C17B—H17D	109.5
C14A—C17A—H17B	109.5	C14B—C17B—H17E	109.5
H17A—C17A—H17B	109.5	H17D—C17B—H17E	109.5
C14A—C17A—H17C	109.5	C14B—C17B—H17F	109.5
H17A—C17A—H17C	109.5	H17D—C17B—H17F	109.5
H17B—C17A—H17C	109.5	H17E—C17B—H17F	109.5
C14A—C18A—H18A	109.5	C14B—C18B—H18D	109.5

C14A—C18A—H18B	109.5	C14B—C18B—H18E	109.5
H18A—C18A—H18B	109.5	H18D—C18B—H18E	109.5
C14A—C18A—H18C	109.5	C14B—C18B—H18F	109.5
H18A—C18A—H18C	109.5	H18D—C18B—H18F	109.5
H18B—C18A—H18C	109.5	H18E—C18B—H18F	109.5
O6A—C19A—C20A	108.0 (6)	C20B—C19B—C23B	114.0 (8)
O6A—C19A—C22A	109.1 (7)	C20B—C19B—O6B	108.1 (6)
C20A—C19A—C22A	112.7 (7)	C23B—C19B—O6B	104.9 (7)
O6A—C19A—C23A	103.3 (7)	C20B—C19B—C22B	111.7 (7)
C20A—C19A—C23A	110.6 (8)	C23B—C19B—C22B	111.6 (7)
C22A—C19A—C23A	112.6 (7)	O6B—C19B—C22B	106.0 (7)
C21A—C20A—C19A	122.0 (9)	C21B—C20B—C19B	122.5 (7)
C21A—C20A—H20A	119.0	C21B—C20B—H20B	118.8
C19A—C20A—H20A	119.0	C19B—C20B—H20B	118.8
C20A—C21A—C7A	116.9 (9)	C20B—C21B—C7B	119.2 (7)
C20A—C21A—H21A	121.6	C20B—C21B—H21B	120.4
C7A—C21A—H21A	121.6	C7B—C21B—H21B	120.4
C19A—C22A—H22A	109.5	C19B—C22B—H22D	109.5
C19A—C22A—H22B	109.5	C19B—C22B—H22E	109.5
H22A—C22A—H22B	109.5	H22D—C22B—H22E	109.5
C19A—C22A—H22C	109.5	C19B—C22B—H22F	109.5
H22A—C22A—H22C	109.5	H22D—C22B—H22F	109.5
H22B—C22A—H22C	109.5	H22E—C22B—H22F	109.5
C19A—C23A—H23A	109.5	C19B—C23B—H23D	109.5
C19A—C23A—H23B	109.5	C19B—C23B—H23E	109.5
H23A—C23A—H23B	109.5	H23D—C23B—H23E	109.5
C19A—C23A—H23C	109.5	C19B—C23B—H23F	109.5
H23A—C23A—H23C	109.5	H23D—C23B—H23F	109.5
H23B—C23A—H23C	109.5	H23E—C23B—H23F	109.5
C14A—O4A—C2A—C3A	153.0 (7)	C10B—C1B—C2B—O4B	178.1 (7)
C14A—O4A—C2A—C1A	−29.3 (11)	C16B—C1B—C2B—O4B	−1.1 (12)
C16A—C1A—C2A—O4A	−5.1 (12)	C10B—C1B—C2B—C3B	3.3 (12)
C10A—C1A—C2A—O4A	−179.3 (7)	C16B—C1B—C2B—C3B	−175.9 (7)
C16A—C1A—C2A—C3A	172.5 (7)	C14B—O4B—C2B—C1B	33.4 (11)
C10A—C1A—C2A—C3A	−1.7 (12)	C14B—O4B—C2B—C3B	−151.3 (7)
O4A—C2A—C3A—O5A	0.0 (11)	C1B—C2B—C3B—O5B	175.7 (7)
C1A—C2A—C3A—O5A	−177.7 (7)	O4B—C2B—C3B—O5B	0.4 (11)
O4A—C2A—C3A—C4A	−177.6 (7)	C1B—C2B—C3B—C4B	−4.2 (12)
C1A—C2A—C3A—C4A	4.7 (12)	O4B—C2B—C3B—C4B	−179.5 (7)
O5A—C3A—C4A—C11A	178.7 (7)	O5B—C3B—C4B—C11B	−177.6 (7)
C2A—C3A—C4A—C11A	−3.8 (12)	C2B—C3B—C4B—C11B	2.3 (12)
C12A—C5A—C6A—C7A	2.1 (13)	C19B—O6B—C6B—C5B	−153.9 (7)
C12A—C5A—C6A—O6A	−179.4 (7)	C19B—O6B—C6B—C7B	26.5 (11)
C19A—O6A—C6A—C7A	−29.2 (11)	C12B—C5B—C6B—O6B	−179.6 (7)
C19A—O6A—C6A—C5A	152.2 (7)	C12B—C5B—C6B—C7B	0.0 (11)
C5A—C6A—C7A—C8A	−1.2 (13)	O6B—C6B—C7B—C8B	179.1 (7)
O6A—C6A—C7A—C8A	−179.6 (7)	C5B—C6B—C7B—C8B	−0.5 (12)
C5A—C6A—C7A—C21A	176.5 (8)	O6B—C6B—C7B—C21B	3.9 (11)
O6A—C6A—C7A—C21A	−1.9 (12)	C5B—C6B—C7B—C21B	−175.7 (7)

C6A—C7A—C8A—O3A	−178.4 (8)	C6B—C7B—C8B—O3B	−178.4 (7)
C21A—C7A—C8A—O3A	3.8 (12)	C21B—C7B—C8B—O3B	−3.5 (11)
C6A—C7A—C8A—C13A	−0.9 (12)	C6B—C7B—C8B—C13B	0.3 (11)
C21A—C7A—C8A—C13A	−178.7 (7)	C21B—C7B—C8B—C13B	175.1 (7)
C2A—C1A—C10A—C11A	−2.1 (12)	C2B—C1B—C10B—C11B	−0.7 (11)
C16A—C1A—C10A—C11A	−175.6 (8)	C16B—C1B—C10B—C11B	178.5 (7)
C2A—C1A—C10A—C9A	177.4 (7)	C2B—C1B—C10B—C9B	−178.7 (7)
C16A—C1A—C10A—C9A	4.0 (13)	C16B—C1B—C10B—C9B	0.4 (13)
O2A—C9A—C10A—C11A	177.6 (9)	O2B—C9B—C10B—C11B	179.5 (7)
C13A—C9A—C10A—C11A	−0.6 (11)	C13B—C9B—C10B—C11B	0.0 (10)
O2A—C9A—C10A—C1A	−1.9 (13)	O2B—C9B—C10B—C1B	−2.5 (13)
C13A—C9A—C10A—C1A	179.8 (7)	C13B—C9B—C10B—C1B	178.0 (7)
C3A—C4A—C11A—C10A	−0.2 (13)	C3B—C4B—C11B—O1B	−179.6 (7)
C3A—C4A—C11A—O1A	−179.0 (7)	C3B—C4B—C11B—C10B	0.3 (12)
C1A—C10A—C11A—C4A	3.1 (13)	C12B—O1B—C11B—C4B	−175.6 (7)
C9A—C10A—C11A—C4A	−176.4 (8)	C12B—O1B—C11B—C10B	4.6 (11)
C1A—C10A—C11A—O1A	−178.1 (7)	C1B—C10B—C11B—C4B	−1.1 (12)
C9A—C10A—C11A—O1A	2.3 (12)	C9B—C10B—C11B—C4B	177.0 (7)
C12A—O1A—C11A—C4A	177.5 (7)	C1B—C10B—C11B—O1B	178.7 (7)
C12A—O1A—C11A—C10A	−1.4 (12)	C9B—C10B—C11B—O1B	−3.2 (11)
C11A—O1A—C12A—C13A	−1.4 (12)	C11B—O1B—C12B—C5B	177.2 (6)
C11A—O1A—C12A—C5A	−179.1 (7)	C11B—O1B—C12B—C13B	−2.7 (11)
C6A—C5A—C12A—C13A	−0.8 (13)	C6B—C5B—C12B—O1B	−179.2 (7)
C6A—C5A—C12A—O1A	177.0 (7)	C6B—C5B—C12B—C13B	0.8 (12)
O1A—C12A—C13A—C8A	−178.8 (8)	O3B—C8B—C13B—C12B	179.0 (7)
C5A—C12A—C13A—C8A	−1.3 (13)	C7B—C8B—C13B—C12B	0.5 (11)
O1A—C12A—C13A—C9A	3.1 (14)	O3B—C8B—C13B—C9B	−1.6 (11)
C5A—C12A—C13A—C9A	−179.3 (7)	C7B—C8B—C13B—C9B	179.8 (7)
O3A—C8A—C13A—C12A	179.6 (8)	O1B—C12B—C13B—C8B	178.9 (7)
C7A—C8A—C13A—C12A	2.2 (13)	C5B—C12B—C13B—C8B	−1.0 (11)
O3A—C8A—C13A—C9A	−2.3 (13)	O1B—C12B—C13B—C9B	−0.5 (11)
C7A—C8A—C13A—C9A	−179.7 (7)	C5B—C12B—C13B—C9B	179.6 (7)
O2A—C9A—C13A—C12A	179.6 (8)	O2B—C9B—C13B—C8B	2.9 (11)
C10A—C9A—C13A—C12A	−2.0 (12)	C10B—C9B—C13B—C8B	−177.6 (7)
O2A—C9A—C13A—C8A	1.5 (12)	O2B—C9B—C13B—C12B	−177.7 (7)
C10A—C9A—C13A—C8A	179.9 (7)	C10B—C9B—C13B—C12B	1.8 (11)
C2A—O4A—C14A—C18A	166.4 (7)	C2B—O4B—C14B—C15B	−48.5 (9)
C2A—O4A—C14A—C17A	−73.7 (9)	C2B—O4B—C14B—C17B	74.5 (8)
C2A—O4A—C14A—C15A	45.5 (9)	C2B—O4B—C14B—C18B	−166.3 (7)
O4A—C14A—C15A—C16A	−34.4 (11)	O4B—C14B—C15B—C16B	38.8 (11)
C18A—C14A—C15A—C16A	−151.7 (8)	C17B—C14B—C15B—C16B	−82.7 (10)
C17A—C14A—C15A—C16A	82.2 (10)	C18B—C14B—C15B—C16B	151.2 (8)
C14A—C15A—C16A—C1A	3.6 (13)	C14B—C15B—C16B—C1B	−8.3 (12)
C2A—C1A—C16A—C15A	18.1 (12)	C2B—C1B—C16B—C15B	−12.5 (11)
C10A—C1A—C16A—C15A	−168.4 (8)	C10B—C1B—C16B—C15B	168.3 (8)
C6A—O6A—C19A—C20A	46.9 (9)	C6B—O6B—C19B—C20B	−41.9 (10)
C6A—O6A—C19A—C22A	−75.9 (8)	C6B—O6B—C19B—C23B	−163.9 (7)
C6A—O6A—C19A—C23A	164.1 (7)	C6B—O6B—C19B—C22B	77.9 (8)
O6A—C19A—C20A—C21A	−40.2 (11)	C23B—C19B—C20B—C21B	147.1 (9)

C22A—C19A—C20A—C21A	80.4 (10)	O6B—C19B—C20B—C21B	30.9 (12)
C23A—C19A—C20A—C21A	−152.6 (8)	C22B—C19B—C20B—C21B	−85.3 (11)
C19A—C20A—C21A—C7A	12.1 (12)	C19B—C20B—C21B—C7B	−4.3 (14)
C6A—C7A—C21A—C20A	10.9 (12)	C8B—C7B—C21B—C20B	169.7 (8)
C8A—C7A—C21A—C20A	−171.4 (8)	C6B—C7B—C21B—C20B	−15.4 (12)

Hydrogen-bond geometry (Å, °)

Cg5 and Cg20 are the centroids of C5A–C8A/C12A/C13A and C5B–C8B/C12B/C13B rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3A—H3AA···O2A	0.82	1.84	2.571 (9)	147
O5A—H5AA···O4A	0.82	2.19	2.656 (9)	116
O3B—H3BA···O2B	0.82	1.84	2.559 (9)	146
O5B—H5BA···O4B	0.82	2.15	2.628 (8)	117
C15B—H15B···O2A^{i}	0.93	2.60	3.514 (11)	168
C16A—H16A···O2A	0.93	2.29	2.879 (11)	121
C16B—H16B···O2B	0.93	2.31	2.890 (10)	120
C17B—H17E···O5B^{i}	0.96	2.58	3.441 (10)	149
C23B—H23D···O1A^{ii}	0.96	2.59	3.370 (11)	139
C18A—H18B···Cg20	0.96	2.75	3.611 (10)	150
C18B—H18D···Cg5	0.96	2.79	3.662 (9)	152

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*+1, *y*−1, *z*.

Fig. 1

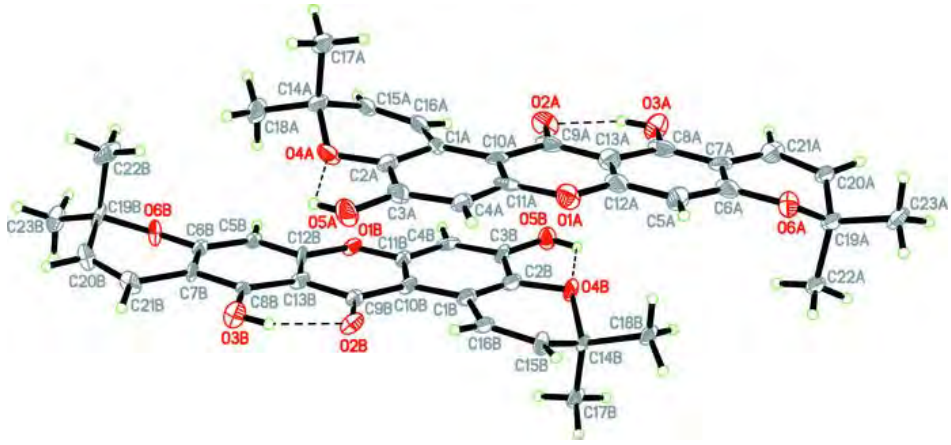
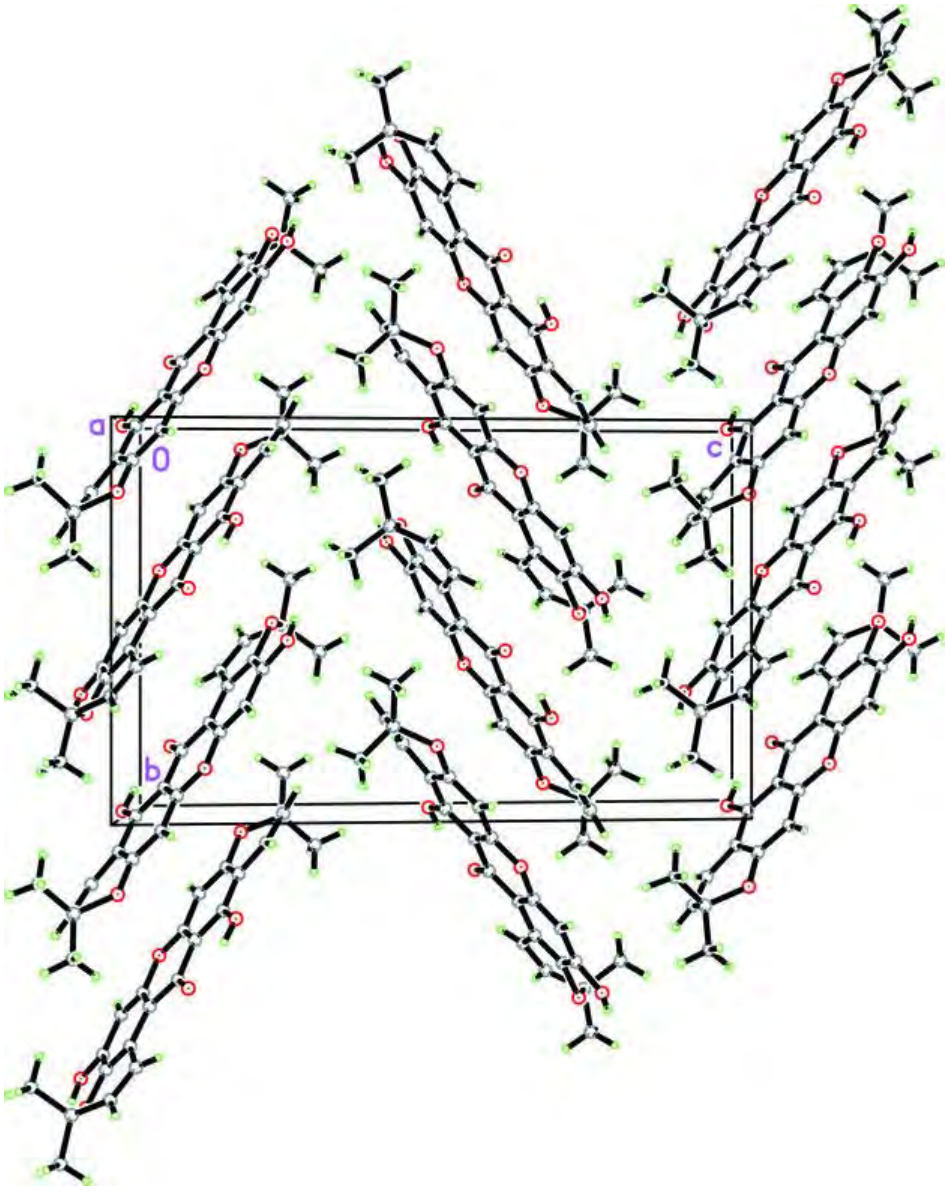


Fig. 2

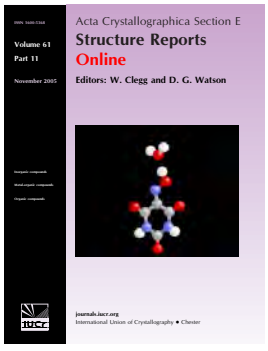


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

6-(4-Bromophenyl)-2-ethoxy-4-(2,4,5-trimethoxyphenyl)nicotinonitrile

Suchada Chantrapromma, Hoong-Kun Fun, Mahesh Padaki, Thitipone Suwunwong and Arun M. Isloor

Acta Cryst. (2010). E**66**, o641–o642



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-(4-Bromophenyl)-2-ethoxy-4-(2,4,5-trimethoxyphenyl)nicotinonitrile[1]

Suchada Chantrapromma,[a]*§ Hoong-Kun Fun,[b]¶ Mahesh Padaki,[c] Thitipone Suwunwong[a] and Arun M. Isloor[c]

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [c]Department of Chemistry, National Institute of Technology-Karnataka, Surathkal, Mangalore 575 025, India
Correspondence e-mail: suchada.c@psu.ac.th

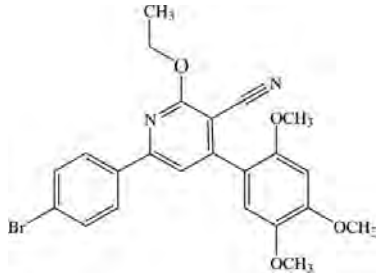
Received 26 January 2010; accepted 9 February 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.098; data-to-parameter ratio = 17.3.

There are two molecules in the asymmetric unit of the title compound, $C_{23}H_{21}BrN_2O_4$, which differ in the conformation of their ethoxy residues, *i.e.* almost co-planar with the pyridine ring in one molecule [C—O—C—C = −174.0 (2)°] but almost perpendicular in the other [C—O—C—C = 92.8 (3)°]. The dihedral angles between the central pyridine ring and the 4-bromophenyl and 2,4,5-trimethoxyphenyl rings are 11.05 (12) and 63.78 (12)°, respectively, in one molecule; the corresponding angles in the other molecule are 30.38 (13) and 65.38 (13)°, respectively. In the crystal structure, pairs of molecules are arranged in a face-to-face sandwich structure which further stacks along the b axis. The crystal packing features C—H···π interactions and Br···O [3.5417 (17) Å], Br···C [3.748 (3) Å], C···N [3.376 (4) Å] and C···O [3.351 (3)–3.409 (3) Å] contacts. Finally, π···π interactions [centroid···centroid distances = 3.6346 (19) and 3.6882 (19) Å] are observed.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the synthesis and applications of nicotinonitrile derivatives, see: Borgna *et al.* (1993); Goda *et al.* (2004). For related structures, see Chantrapromma *et al.* (2009, 2010). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{23}H_{21}BrN_2O_4$
M_r = 469.32
Triclinic, $P\overline{1}$
a = 7.9631 (2) Å
b = 11.0499 (3) Å
c = 23.9690 (6) Å
α = 92.201 (1)°
β = 91.968 (1)°
γ = 99.586 (1)°
V = 2076.31 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.01 mm^{-1}
T = 100 K
0.59 × 0.10 × 0.05 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.384, T_{max} = 0.899
31800 measured reflections
9488 independent reflections
7074 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
S = 1.07
9488 reflections
549 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 and *Cg*2 are the centroids of C7*A*–C11*A*/N1*A* and C12*A*–C17*A* rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C22*A*—H22*C*···*Cg*2^{i}	0.96	2.74	3.585 (3)	147
C22*B*—H22*E*···*Cg*1^{ii}	0.96	2.68	3.511 (3)	145

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for a research grant (RSA 5280033) and the Koshinocorporation Group (Japan), the Prince of Songkla University and Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012. AMI is grateful to the Director, NITK-Surathkal, India for providing research facilities and the Head of the Department of Chemistry and Dean R&D, NITK Surathkal, for their encouragement.

[1] This paper is dedicated to His Majesty King Bhumibol Adulyadej of Thailand (King Rama IX) for his sustainable development of the country.
§ Thomson Reuters ResearcherID: A-5085-2009.
¶ Additional correspondence author, email: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2622).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Borgna, P., Pregnolato, M., Gamba, I. A. & Mellerio, G. (1993). *J. Heterocyclic Chem*, **30**, 1079–1084.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chantrapromma, S., Fun, H.-K., Suwunwong, T., Padaki, M. & Isloor, A. M. (2009). *Acta Cryst.* E**65**, o2914–o2915.

Chantrapromma, S., Fun, H.-K., Suwunwong, T., Padaki, M. & Isloor, A. M. (2010). *Acta Cryst.* E**66**, o79–o80.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Goda, F. E., Abdel-Aziz, A. A.-M. & Attef, O. A. (2004). *Bioorg. Med. Chem.* **12**, 1845–1852.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst*. D**65**, 148–155.

supplementary materials

Acta Cryst. (2010). E**66**, o641-o642 [**doi:10.1107/S1600536810005210**]

6-(4-Bromophenyl)-2-ethoxy-4-(2,4,5-trimethoxyphenyl)nicotinonitrile

S. Chantrapromma, H.-K. Fun, M. Padaki, T. Suwunwong and A. M. Isloor

Comment

Substituted pyridine derivatives have been claimed to have several biological activities (Borgna *et al.*, 1993; Goda *et al.*, 2004). We have previously reported the syntheses and crystal structures of the nicotinonitrile derivatives (Chantrapromma *et al.*, 2009; 2010). In continuation of our research into the synthesis of antimicrobial agents, the title malononitrile derivative was synthesised and studied for its anti-bacterial activities. However, our results showed that the title compound does not possess anti-bacterial activities. Herein, we report the crystal structure of the title compound (I).

There are two crystallographic independent molecules *A* and *B* in the asymmetric unit of (I) (Fig. 1) with differences in conformation of the ethoxy group distinguishing them. In molecule *A* the ethoxy residue is almost co-planar with the pyridine ring [C11A–O1A–C18A–C19A = -174.0 (2) °] whereas it is almost perpendicular in molecule *B* [C11B–O1B–C18B–C19B = 92.8 (3) °]. The dihedral angles between the central pyridine ring and the 4-bromophenyl and 2,4,5-trimethoxyphenyl rings are 11.05 (12) and 63.78 (12) ° respectively in molecule *A* whereas the corresponding pair of angles in molecule *B* are 30.38 (13) and 65.38 (13) °. All three methoxy groups are nearly co-planar with the attached benzene ring [torsion angles C20–O2–C13–C14 = -11.6 (4) °, C21–O3–C15–C16 = 180.0 (2) ° and C22–O4–C16–C17 = 8.1 (4) ° in molecule *A*; and the corresponding values are -5.5 (4), -175.5 (2) and -7.7 (4) ° in molecule *B*. Weak intramolecular C1A—H1AA···N1A (in molecule *A*) and C18B–H18C—N1B (in molecule *B*) interactions generate S(5) ring motifs (Bernstein *et al.*, 1995). The bond distances are comparable with those in closely related structures (Chantrapromma *et al.*, 2009; 2010).

In the crystal structure (Fig. 2), the molecules are arranged into a face-to-face sandwich-like structure which further stack along the *b* axis. The crystal is consolidated by C—H···π interactions (Table 1) and Br···O [3.5417 (17) Å], Br···C [3.748 (3) Å], C···N [3.376 (4) Å] and C···O [3.351 (3) - 3.409 (3) Å] contacts. Finally, π···π interactions with the distances of $Cg_1\cdots Cg_4$ = 3.6346 (15) Å (symmetry code: 1-x, 1-y, 1-z) and $Cg_2\cdots Cg_3$ = 3.6882 (15) Å (symmetry code: -x, 1-y, 1-z) are observed; Cg_1, Cg_2, Cg_3 and Cg_4 are the centroids of C7A–C11A/N1A, C12A–C17A, C7B–C11B/N1B, and C12B–C17B rings, respectively.

Experimental

(*E*)-1-(4-Bromophenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one (0.57 g, 0.0015 mol) was added with continuous stirring to a freshly prepared sodium alkoxide solution (0.0014 mol of sodium in 100 ml of ethanol). Malononitrile (1.3 g, 0.02 mol) was then added with continuous stirring at room temperature until the precipitate separated out. The resulting solid was filtered (yield 68 %). Colorless needle-shaped single crystals of the title compound were obtained by recrystallization from ethanol by the slow evaporation of the solvent at room temperature after several days, Mp. 460-461 K.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C—H) = 0.93 Å for aromatic-H, 0.97 for CH_2 and 0.96 Å for methyl-H atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl-H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

Figures

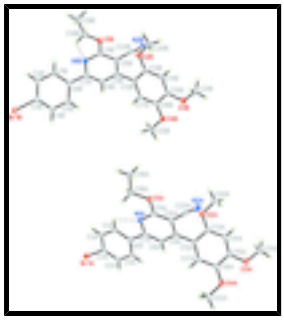


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. Intramolecular C—H···N interactions are shown as dashed lines.

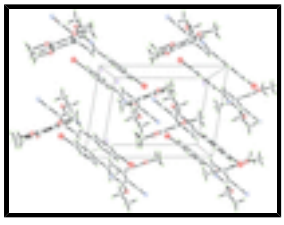


Fig. 2. The crystal packing of the title compound viewed along the *c* axis.

6-(4-Bromophenyl)-2-ethoxy-4-(2,4,5-trimethoxyphenyl)nicotinonitrile

Crystal data

$C_{23}H_{21}BrN_2O_4$	$Z = 4$
$M_r = 469.32$	$F(000) = 960$
Triclinic, $P\overline{1}$	$D_x = 1.501$ Mg m^{-3}
Hall symbol: -P 1	Melting point = 460–461 K
$a = 7.9631$ (2) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 11.0499$ (3) Å	Cell parameters from 9488 reflections
$c = 23.9690$ (6) Å	$\theta = 0.9$–$27.5°$
$\alpha = 92.201$ (1)°	$\mu = 2.01$ mm^{-1}
$\beta = 91.968$ (1)°	$T = 100$ K
$\gamma = 99.586$ (1)°	Needle, colorless
$V = 2076.31$ (9) Å^3	0.59 × 0.10 × 0.05 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	9488 independent reflections
Radiation source: sealed tube	7074 reflections with $I > 2\sigma(I)$

graphite

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

T_{min} = 0.384, T_{max} = 0.899

31800 measured reflections

R_{int} = 0.043

θ_{max} = 27.5°, θ_{min} = 0.9°

h = −10→10

k = −14→14

l = −31→31

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.036

$wR(F^2)$ = 0.098

S = 1.07

9488 reflections

549 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 0.7288P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.79 e Å^{-3}

$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor wR and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) etc. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1A	−0.17700 (4)	−0.35388 (2)	0.439858 (11)	0.02094 (8)
O1A	0.5666 (2)	0.35133 (16)	0.42626 (7)	0.0190 (4)
O2A	0.2036 (3)	0.48545 (17)	0.28313 (7)	0.0229 (4)
O3A	0.2540 (3)	0.46349 (17)	0.08142 (7)	0.0227 (4)
O4A	0.3749 (3)	0.26013 (17)	0.08836 (7)	0.0211 (4)
N1A	0.3634 (3)	0.18217 (19)	0.40057 (8)	0.0161 (5)
N2A	0.6447 (4)	0.5362 (2)	0.30967 (10)	0.0304 (6)
C1A	0.1472 (3)	−0.0213 (2)	0.43550 (10)	0.0171 (6)
H1AA	0.2118	0.0341	0.4613	0.021*
C2A	0.0518 (4)	−0.1272 (2)	0.45413 (11)	0.0183 (6)

H2AA	0.0519	−0.1434	0.4919	0.022*
C3A	−0.0433 (3)	−0.2081 (2)	0.41548 (11)	0.0160 (5)
C4A	−0.0472 (3)	−0.1856 (2)	0.35901 (11)	0.0177 (6)
H4AA	−0.1134	−0.2411	0.3337	0.021*
C5A	0.0488 (4)	−0.0796 (2)	0.34091 (10)	0.0179 (6)
H5AA	0.0470	−0.0639	0.3031	0.022*
C6A	0.1488 (3)	0.0047 (2)	0.37885 (10)	0.0145 (5)
C7A	0.2528 (3)	0.1189 (2)	0.36092 (10)	0.0153 (5)
C8A	0.2379 (3)	0.1613 (2)	0.30754 (10)	0.0157 (5)
H8AA	0.1637	0.1154	0.2809	0.019*
C9A	0.3339 (4)	0.2727 (2)	0.29383 (10)	0.0163 (5)
C10A	0.4480 (3)	0.3366 (2)	0.33414 (10)	0.0160 (5)
C11A	0.4564 (3)	0.2863 (2)	0.38748 (10)	0.0158 (5)
C12A	0.3084 (3)	0.3240 (2)	0.23801 (10)	0.0166 (6)
C13A	0.2479 (3)	0.4339 (2)	0.23373 (11)	0.0183 (6)
C14A	0.2272 (3)	0.4832 (2)	0.18187 (11)	0.0186 (6)
H14A	0.1854	0.5566	0.1793	0.022*
C15A	0.2695 (3)	0.4223 (2)	0.13403 (10)	0.0180 (6)
C16A	0.3318 (3)	0.3113 (2)	0.13770 (10)	0.0180 (6)
C17A	0.3479 (3)	0.2623 (2)	0.18950 (10)	0.0166 (5)
H17A	0.3856	0.1873	0.1920	0.020*
C18A	0.5755 (4)	0.2961 (2)	0.48023 (10)	0.0206 (6)
H18A	0.6219	0.2205	0.4764	0.025*
H18B	0.4625	0.2770	0.4949	0.025*
C19A	0.6888 (4)	0.3869 (3)	0.51911 (11)	0.0244 (6)
H19A	0.6960	0.3531	0.5552	0.037*
H19B	0.6423	0.4615	0.5224	0.037*
H19C	0.8007	0.4042	0.5045	0.037*
C20A	0.1635 (5)	0.6051 (3)	0.28312 (13)	0.0425 (9)
H20A	0.1407	0.6307	0.3204	0.064*
H20B	0.0645	0.6053	0.2591	0.064*
H20C	0.2579	0.6607	0.2698	0.064*
C21A	0.1906 (4)	0.5766 (3)	0.07668 (12)	0.0281 (7)
H21A	0.1801	0.5945	0.0380	0.042*
H21B	0.2683	0.6418	0.0960	0.042*
H21C	0.0810	0.5695	0.0929	0.042*
C22A	0.4189 (4)	0.1406 (2)	0.09113 (11)	0.0243 (6)
H22A	0.4428	0.1117	0.0544	0.036*
H22B	0.3256	0.0854	0.1053	0.036*
H22C	0.5179	0.1440	0.1155	0.036*
C23A	0.5564 (4)	0.4488 (3)	0.32183 (10)	0.0215 (6)
Br1B	0.38606 (4)	0.11796 (3)	0.926074 (11)	0.02560 (9)
O1B	0.0191 (3)	0.84937 (17)	0.92736 (7)	0.0231 (4)
O2B	0.4260 (3)	0.98663 (17)	0.78188 (7)	0.0240 (4)
O3B	0.2913 (3)	0.96201 (16)	0.58180 (7)	0.0210 (4)
O4B	0.0815 (3)	0.75855 (16)	0.59168 (7)	0.0209 (4)
N1B	0.1491 (3)	0.68209 (19)	0.90306 (8)	0.0164 (5)
N2B	−0.0051 (3)	1.0323 (2)	0.81419 (10)	0.0279 (6)
C1B	0.3117 (4)	0.4800 (2)	0.93221 (11)	0.0199 (6)

H1BA	0.3184	0.5436	0.9592	0.024*
C2B	0.3487 (4)	0.3679 (2)	0.94771 (11)	0.0202 (6)
H2BA	0.3800	0.3556	0.9845	0.024*
C3B	0.3378 (4)	0.2743 (2)	0.90675 (11)	0.0199 (6)
C4B	0.2924 (4)	0.2911 (3)	0.85171 (11)	0.0230 (6)
H4BA	0.2850	0.2270	0.8250	0.028*
C5B	0.2584 (4)	0.4043 (2)	0.83709 (11)	0.0226 (6)
H5BA	0.2310	0.4170	0.8000	0.027*
C6B	0.2645 (3)	0.5004 (2)	0.87740 (10)	0.0173 (6)
C7B	0.2157 (3)	0.6191 (2)	0.86213 (11)	0.0178 (6)
C8B	0.2394 (4)	0.6627 (2)	0.80881 (10)	0.0187 (6)
H8BA	0.2854	0.6167	0.7818	0.022*
C9B	0.1946 (3)	0.7745 (2)	0.79583 (10)	0.0165 (5)
C10B	0.1183 (3)	0.8366 (2)	0.83705 (10)	0.0179 (6)
C11B	0.0976 (4)	0.7856 (2)	0.88999 (10)	0.0179 (6)
C12B	0.2257 (3)	0.8253 (2)	0.74001 (10)	0.0164 (5)
C13B	0.3413 (4)	0.9333 (2)	0.73404 (11)	0.0185 (6)
C14B	0.3669 (4)	0.9808 (2)	0.68134 (10)	0.0178 (6)
H14B	0.4443	1.0526	0.6774	0.021*
C15B	0.2768 (4)	0.9209 (2)	0.63469 (10)	0.0170 (6)
C16B	0.1637 (3)	0.8103 (2)	0.64004 (10)	0.0160 (5)
C17B	0.1417 (3)	0.7636 (2)	0.69231 (10)	0.0172 (6)
H17B	0.0693	0.6893	0.6959	0.021*
C18B	−0.0121 (4)	0.8016 (3)	0.98201 (11)	0.0262 (7)
H18C	−0.0237	0.7127	0.9796	0.031*
H18D	−0.1179	0.8227	0.9951	0.031*
C19B	0.1320 (5)	0.8537 (3)	1.02274 (13)	0.0367 (8)
H19D	0.1053	0.8262	1.0594	0.055*
H19E	0.1481	0.9418	1.0233	0.055*
H19F	0.2346	0.8265	1.0115	0.055*
C20B	0.5570 (4)	1.0900 (3)	0.77626 (12)	0.0278 (7)
H20D	0.6111	1.1153	0.8122	0.042*
H20E	0.5081	1.1564	0.7614	0.042*
H20F	0.6400	1.0679	0.7514	0.042*
C21B	0.4143 (4)	1.0690 (3)	0.57435 (11)	0.0233 (6)
H21D	0.4166	1.0868	0.5355	0.035*
H21E	0.5248	1.0550	0.5871	0.035*
H21F	0.3845	1.1373	0.5955	0.035*
C22B	−0.0187 (4)	0.6395 (2)	0.59614 (11)	0.0227 (6)
H22D	−0.0687	0.6101	0.5601	0.034*
H22E	−0.1074	0.6447	0.6218	0.034*
H22F	0.0526	0.5839	0.6095	0.034*
C23B	0.0514 (4)	0.9469 (3)	0.82524 (11)	0.0212 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1A	0.02292 (16)	0.01573 (13)	0.02271 (14)	−0.00184 (12)	0.00184 (12)	0.00381 (10)

O1A	0.0229 (11)	0.0180 (9)	0.0136 (8)	−0.0040 (8)	−0.0027 (8)	0.0030 (7)
O2A	0.0305 (12)	0.0210 (10)	0.0184 (9)	0.0076 (9)	0.0038 (9)	0.0017 (8)
O3A	0.0238 (11)	0.0271 (10)	0.0185 (9)	0.0066 (9)	0.0006 (8)	0.0092 (8)
O4A	0.0245 (11)	0.0247 (10)	0.0153 (9)	0.0070 (9)	0.0012 (8)	0.0012 (7)
N1A	0.0153 (12)	0.0162 (11)	0.0161 (10)	0.0003 (10)	0.0009 (9)	0.0014 (8)
N2A	0.0366 (16)	0.0297 (14)	0.0200 (12)	−0.0087 (13)	−0.0015 (12)	0.0031 (10)
C1A	0.0157 (14)	0.0176 (13)	0.0175 (12)	0.0016 (11)	−0.0003 (11)	−0.0007 (10)
C2A	0.0217 (15)	0.0186 (13)	0.0148 (12)	0.0041 (12)	0.0008 (11)	0.0024 (10)
C3A	0.0123 (13)	0.0121 (12)	0.0238 (13)	0.0014 (11)	0.0036 (11)	0.0034 (10)
C4A	0.0135 (14)	0.0179 (13)	0.0209 (13)	0.0015 (11)	−0.0005 (11)	−0.0030 (10)
C5A	0.0205 (15)	0.0185 (13)	0.0144 (12)	0.0022 (12)	0.0001 (11)	0.0003 (10)
C6A	0.0080 (13)	0.0162 (12)	0.0194 (12)	0.0023 (11)	0.0006 (11)	0.0022 (10)
C7A	0.0127 (13)	0.0165 (12)	0.0175 (12)	0.0046 (11)	0.0026 (11)	−0.0007 (10)
C8A	0.0135 (14)	0.0171 (12)	0.0159 (12)	0.0018 (11)	−0.0023 (11)	−0.0012 (10)
C9A	0.0173 (14)	0.0178 (13)	0.0148 (12)	0.0050 (11)	0.0024 (11)	0.0010 (10)
C10A	0.0112 (13)	0.0176 (13)	0.0187 (12)	0.0006 (11)	0.0009 (11)	0.0012 (10)
C11A	0.0114 (13)	0.0189 (13)	0.0170 (12)	0.0030 (11)	−0.0006 (11)	−0.0006 (10)
C12A	0.0163 (14)	0.0165 (12)	0.0156 (12)	−0.0017 (11)	−0.0009 (11)	0.0023 (10)
C13A	0.0146 (14)	0.0190 (13)	0.0200 (13)	0.0000 (12)	0.0011 (11)	−0.0011 (10)
C14A	0.0165 (14)	0.0165 (13)	0.0232 (13)	0.0031 (12)	0.0014 (12)	0.0040 (10)
C15A	0.0151 (14)	0.0206 (13)	0.0169 (12)	−0.0026 (12)	0.0003 (11)	0.0054 (10)
C16A	0.0143 (14)	0.0199 (13)	0.0184 (13)	−0.0009 (12)	0.0012 (11)	0.0001 (10)
C17A	0.0131 (14)	0.0170 (12)	0.0183 (12)	−0.0012 (11)	−0.0010 (11)	0.0011 (10)
C18A	0.0254 (16)	0.0215 (13)	0.0142 (12)	0.0011 (13)	−0.0005 (12)	0.0065 (10)
C19A	0.0235 (16)	0.0273 (15)	0.0206 (14)	−0.0011 (13)	−0.0028 (12)	0.0032 (11)
C20A	0.071 (3)	0.0301 (17)	0.0321 (17)	0.0255 (19)	0.0053 (18)	0.0008 (14)
C21A	0.0297 (18)	0.0338 (16)	0.0237 (14)	0.0107 (15)	0.0022 (13)	0.0138 (12)
C22A	0.0315 (18)	0.0239 (14)	0.0173 (13)	0.0054 (14)	−0.0009 (13)	−0.0013 (11)
C23A	0.0262 (16)	0.0238 (14)	0.0126 (12)	−0.0010 (13)	−0.0007 (12)	0.0013 (11)
Br1B	0.03322 (19)	0.02264 (15)	0.02261 (14)	0.00896 (13)	0.00077 (13)	0.00399 (11)
O1B	0.0277 (12)	0.0230 (10)	0.0192 (9)	0.0039 (9)	0.0058 (9)	0.0030 (8)
O2B	0.0216 (11)	0.0272 (10)	0.0189 (9)	−0.0078 (9)	−0.0012 (8)	0.0018 (8)
O3B	0.0241 (11)	0.0224 (10)	0.0155 (9)	−0.0003 (9)	0.0016 (8)	0.0065 (7)
O4B	0.0233 (11)	0.0212 (9)	0.0160 (9)	−0.0030 (9)	−0.0007 (8)	0.0027 (7)
N1B	0.0122 (11)	0.0194 (11)	0.0160 (10)	−0.0021 (10)	−0.0012 (9)	0.0011 (9)
N2B	0.0287 (15)	0.0261 (13)	0.0282 (13)	0.0037 (12)	−0.0045 (11)	0.0023 (10)
C1B	0.0195 (15)	0.0201 (13)	0.0186 (13)	−0.0010 (12)	0.0011 (12)	−0.0008 (10)
C2B	0.0182 (15)	0.0256 (14)	0.0164 (12)	0.0018 (12)	−0.0012 (12)	0.0048 (11)
C3B	0.0167 (15)	0.0202 (13)	0.0234 (14)	0.0036 (12)	0.0023 (12)	0.0050 (11)
C4B	0.0270 (17)	0.0222 (14)	0.0199 (13)	0.0047 (13)	0.0020 (12)	−0.0021 (11)
C5B	0.0273 (16)	0.0260 (14)	0.0144 (12)	0.0039 (13)	−0.0003 (12)	0.0038 (11)
C6B	0.0147 (14)	0.0196 (13)	0.0163 (12)	−0.0016 (11)	0.0016 (11)	0.0018 (10)
C7B	0.0121 (14)	0.0210 (13)	0.0187 (13)	−0.0020 (11)	−0.0019 (11)	0.0018 (10)
C8B	0.0164 (14)	0.0224 (14)	0.0166 (12)	0.0014 (12)	0.0019 (11)	0.0003 (10)
C9B	0.0108 (13)	0.0213 (13)	0.0149 (12)	−0.0041 (11)	−0.0025 (10)	0.0033 (10)
C10B	0.0138 (14)	0.0205 (13)	0.0179 (12)	−0.0015 (12)	−0.0023 (11)	0.0032 (10)
C11B	0.0170 (14)	0.0181 (13)	0.0160 (12)	−0.0037 (12)	−0.0008 (11)	−0.0013 (10)
C12B	0.0141 (14)	0.0181 (13)	0.0172 (12)	0.0027 (11)	0.0007 (11)	0.0038 (10)
C13B	0.0163 (14)	0.0203 (13)	0.0186 (13)	0.0025 (12)	−0.0009 (11)	0.0015 (10)

C14B	0.0159 (14)	0.0165 (13)	0.0210 (13)	0.0019 (11)	0.0024 (11)	0.0040 (10)
C15B	0.0186 (15)	0.0186 (13)	0.0156 (12)	0.0063 (12)	0.0038 (11)	0.0051 (10)
C16B	0.0099 (13)	0.0196 (13)	0.0191 (13)	0.0041 (11)	0.0012 (11)	−0.0001 (10)
C17B	0.0148 (14)	0.0164 (12)	0.0205 (13)	0.0012 (11)	0.0035 (11)	0.0049 (10)
C18B	0.0331 (18)	0.0266 (15)	0.0195 (13)	0.0043 (14)	0.0077 (13)	0.0036 (11)
C19B	0.049 (2)	0.0237 (15)	0.0343 (17)	−0.0022 (16)	−0.0071 (16)	0.0035 (13)
C20B	0.0288 (18)	0.0264 (15)	0.0237 (14)	−0.0074 (14)	0.0019 (13)	−0.0040 (12)
C21B	0.0173 (15)	0.0282 (15)	0.0238 (14)	−0.0005 (13)	0.0019 (12)	0.0110 (12)
C22B	0.0263 (16)	0.0195 (13)	0.0205 (13)	−0.0015 (13)	0.0006 (12)	0.0001 (11)
C23B	0.0206 (15)	0.0254 (15)	0.0153 (13)	−0.0024 (13)	−0.0013 (12)	0.0012 (11)

Geometric parameters (Å, °)

Br1A—C3A	1.901 (2)	Br1B—C3B	1.902 (3)
O1A—C11A	1.354 (3)	O1B—C11B	1.352 (3)
O1A—C18A	1.456 (3)	O1B—C18B	1.445 (3)
O2A—C13A	1.377 (3)	O2B—C13B	1.371 (3)
O2A—C20A	1.411 (3)	O2B—C20B	1.427 (3)
O3A—C15A	1.366 (3)	O3B—C15B	1.365 (3)
O3A—C21A	1.431 (3)	O3B—C21B	1.425 (3)
O4A—C16A	1.370 (3)	O4B—C16B	1.368 (3)
O4A—C22A	1.426 (3)	O4B—C22B	1.430 (3)
N1A—C11A	1.316 (3)	N1B—C11B	1.322 (3)
N1A—C7A	1.362 (3)	N1B—C7B	1.354 (3)
N2A—C23A	1.150 (3)	N2B—C23B	1.147 (4)
C1A—C2A	1.384 (4)	C1B—C2B	1.381 (4)
C1A—C6A	1.398 (3)	C1B—C6B	1.391 (3)
C1A—H1AA	0.9300	C1B—H1BA	0.9300
C2A—C3A	1.376 (4)	C2B—C3B	1.388 (4)
C2A—H2AA	0.9300	C2B—H2BA	0.9300
C3A—C4A	1.386 (4)	C3B—C4B	1.383 (4)
C4A—C5A	1.381 (4)	C4B—C5B	1.379 (4)
C4A—H4AA	0.9300	C4B—H4BA	0.9300
C5A—C6A	1.402 (3)	C5B—C6B	1.402 (4)
C5A—H5AA	0.9300	C5B—H5BA	0.9300
C6A—C7A	1.477 (4)	C6B—C7B	1.485 (4)
C7A—C8A	1.387 (3)	C7B—C8B	1.391 (3)
C8A—C9A	1.393 (4)	C8B—C9B	1.385 (4)
C8A—H8AA	0.9300	C8B—H8BA	0.9300
C9A—C10A	1.390 (4)	C9B—C10B	1.394 (4)
C9A—C12A	1.494 (3)	C9B—C12B	1.484 (3)
C10A—C11A	1.417 (3)	C10B—C11B	1.412 (3)
C10A—C23A	1.435 (4)	C10B—C23B	1.443 (4)
C12A—C13A	1.384 (4)	C12B—C13B	1.396 (4)
C12A—C17A	1.398 (4)	C12B—C17B	1.399 (4)
C13A—C14A	1.392 (4)	C13B—C14B	1.395 (3)
C14A—C15A	1.387 (4)	C14B—C15B	1.390 (4)
C14A—H14A	0.9300	C14B—H14B	0.9300
C15A—C16A	1.402 (4)	C15B—C16B	1.406 (4)

C16A—C17A	1.384 (3)	C16B—C17B	1.379 (3)
C17A—H17A	0.9300	C17B—H17B	0.9300
C18A—C19A	1.503 (4)	C18B—C19B	1.504 (4)
C18A—H18A	0.9700	C18B—H18C	0.9700
C18A—H18B	0.9700	C18B—H18D	0.9700
C19A—H19A	0.9600	C19B—H19D	0.9600
C19A—H19B	0.9600	C19B—H19E	0.9600
C19A—H19C	0.9600	C19B—H19F	0.9600
C20A—H20A	0.9600	C20B—H20D	0.9600
C20A—H20B	0.9600	C20B—H20E	0.9600
C20A—H20C	0.9600	C20B—H20F	0.9600
C21A—H21A	0.9600	C21B—H21D	0.9600
C21A—H21B	0.9600	C21B—H21E	0.9600
C21A—H21C	0.9600	C21B—H21F	0.9600
C22A—H22A	0.9600	C22B—H22D	0.9600
C22A—H22B	0.9600	C22B—H22E	0.9600
C22A—H22C	0.9600	C22B—H22F	0.9600
C11A—O1A—C18A	115.60 (19)	C11B—O1B—C18B	119.1 (2)
C13A—O2A—C20A	118.5 (2)	C13B—O2B—C20B	117.6 (2)
C15A—O3A—C21A	116.7 (2)	C15B—O3B—C21B	117.0 (2)
C16A—O4A—C22A	115.9 (2)	C16B—O4B—C22B	115.60 (19)
C11A—N1A—C7A	118.5 (2)	C11B—N1B—C7B	117.5 (2)
C2A—C1A—C6A	121.6 (2)	C2B—C1B—C6B	121.9 (2)
C2A—C1A—H1AA	119.2	C2B—C1B—H1BA	119.1
C6A—C1A—H1AA	119.2	C6B—C1B—H1BA	119.1
C3A—C2A—C1A	118.5 (2)	C1B—C2B—C3B	118.1 (2)
C3A—C2A—H2AA	120.8	C1B—C2B—H2BA	121.0
C1A—C2A—H2AA	120.8	C3B—C2B—H2BA	121.0
C2A—C3A—C4A	121.9 (2)	C4B—C3B—C2B	121.9 (2)
C2A—C3A—Br1A	119.47 (19)	C4B—C3B—Br1B	118.4 (2)
C4A—C3A—Br1A	118.59 (19)	C2B—C3B—Br1B	119.7 (2)
C5A—C4A—C3A	119.0 (2)	C5B—C4B—C3B	119.0 (2)
C5A—C4A—H4AA	120.5	C5B—C4B—H4BA	120.5
C3A—C4A—H4AA	120.5	C3B—C4B—H4BA	120.5
C4A—C5A—C6A	120.9 (2)	C4B—C5B—C6B	120.9 (2)
C4A—C5A—H5AA	119.6	C4B—C5B—H5BA	119.6
C6A—C5A—H5AA	119.6	C6B—C5B—H5BA	119.6
C1A—C6A—C5A	118.1 (2)	C1B—C6B—C5B	118.3 (2)
C1A—C6A—C7A	119.7 (2)	C1B—C6B—C7B	121.1 (2)
C5A—C6A—C7A	122.3 (2)	C5B—C6B—C7B	120.6 (2)
N1A—C7A—C8A	121.5 (2)	N1B—C7B—C8B	122.5 (2)
N1A—C7A—C6A	115.7 (2)	N1B—C7B—C6B	116.2 (2)
C8A—C7A—C6A	122.7 (2)	C8B—C7B—C6B	121.3 (2)
C7A—C8A—C9A	120.1 (2)	C9B—C8B—C7B	120.1 (2)
C7A—C8A—H8AA	119.9	C9B—C8B—H8BA	119.9
C9A—C8A—H8AA	119.9	C7B—C8B—H8BA	119.9
C10A—C9A—C8A	118.3 (2)	C8B—C9B—C10B	117.4 (2)
C10A—C9A—C12A	121.2 (2)	C8B—C9B—C12B	121.1 (2)
C8A—C9A—C12A	120.5 (2)	C10B—C9B—C12B	121.5 (2)

C9A—C10A—C11A	118.0 (2)	C9B—C10B—C11B	118.8 (2)
C9A—C10A—C23A	121.0 (2)	C9B—C10B—C23B	121.1 (2)
C11A—C10A—C23A	121.0 (2)	C11B—C10B—C23B	120.0 (2)
N1A—C11A—O1A	119.7 (2)	N1B—C11B—O1B	121.1 (2)
N1A—C11A—C10A	123.5 (2)	N1B—C11B—C10B	123.4 (2)
O1A—C11A—C10A	116.8 (2)	O1B—C11B—C10B	115.5 (2)
C13A—C12A—C17A	119.3 (2)	C13B—C12B—C17B	119.0 (2)
C13A—C12A—C9A	120.6 (2)	C13B—C12B—C9B	120.9 (2)
C17A—C12A—C9A	120.1 (2)	C17B—C12B—C9B	120.1 (2)
O2A—C13A—C12A	115.6 (2)	O2B—C13B—C14B	123.4 (2)
O2A—C13A—C14A	123.6 (2)	O2B—C13B—C12B	116.5 (2)
C12A—C13A—C14A	120.7 (2)	C14B—C13B—C12B	120.1 (2)
C15A—C14A—C13A	119.6 (2)	C15B—C14B—C13B	120.1 (2)
C15A—C14A—H14A	120.2	C15B—C14B—H14B	120.0
C13A—C14A—H14A	120.2	C13B—C14B—H14B	120.0
O3A—C15A—C14A	123.9 (2)	O3B—C15B—C14B	124.0 (2)
O3A—C15A—C16A	115.8 (2)	O3B—C15B—C16B	115.7 (2)
C14A—C15A—C16A	120.4 (2)	C14B—C15B—C16B	120.3 (2)
O4A—C16A—C17A	124.8 (2)	O4B—C16B—C17B	125.3 (2)
O4A—C16A—C15A	116.0 (2)	O4B—C16B—C15B	115.8 (2)
C17A—C16A—C15A	119.2 (2)	C17B—C16B—C15B	118.9 (2)
C16A—C17A—C12A	120.8 (2)	C16B—C17B—C12B	121.5 (2)
C16A—C17A—H17A	119.6	C16B—C17B—H17B	119.2
C12A—C17A—H17A	119.6	C12B—C17B—H17B	119.2
O1A—C18A—C19A	107.7 (2)	O1B—C18B—C19B	110.5 (2)
O1A—C18A—H18A	110.2	O1B—C18B—H18C	109.6
C19A—C18A—H18A	110.2	C19B—C18B—H18C	109.6
O1A—C18A—H18B	110.2	O1B—C18B—H18D	109.6
C19A—C18A—H18B	110.2	C19B—C18B—H18D	109.6
H18A—C18A—H18B	108.5	H18C—C18B—H18D	108.1
C18A—C19A—H19A	109.5	C18B—C19B—H19D	109.5
C18A—C19A—H19B	109.5	C18B—C19B—H19E	109.5
H19A—C19A—H19B	109.5	H19D—C19B—H19E	109.5
C18A—C19A—H19C	109.5	C18B—C19B—H19F	109.5
H19A—C19A—H19C	109.5	H19D—C19B—H19F	109.5
H19B—C19A—H19C	109.5	H19E—C19B—H19F	109.5
O2A—C20A—H20A	109.5	O2B—C20B—H20D	109.5
O2A—C20A—H20B	109.5	O2B—C20B—H20E	109.5
H20A—C20A—H20B	109.5	H20D—C20B—H20E	109.5
O2A—C20A—H20C	109.5	O2B—C20B—H20F	109.5
H20A—C20A—H20C	109.5	H20D—C20B—H20F	109.5
H20B—C20A—H20C	109.5	H20E—C20B—H20F	109.5
O3A—C21A—H21A	109.5	O3B—C21B—H21D	109.5
O3A—C21A—H21B	109.5	O3B—C21B—H21E	109.5
H21A—C21A—H21B	109.5	H21D—C21B—H21E	109.5
O3A—C21A—H21C	109.5	O3B—C21B—H21F	109.5
H21A—C21A—H21C	109.5	H21D—C21B—H21F	109.5
H21B—C21A—H21C	109.5	H21E—C21B—H21F	109.5
O4A—C22A—H22A	109.5	O4B—C22B—H22D	109.5

O4A—C22A—H22B	109.5	O4B—C22B—H22E	109.5
H22A—C22A—H22B	109.5	H22D—C22B—H22E	109.5
O4A—C22A—H22C	109.5	O4B—C22B—H22F	109.5
H22A—C22A—H22C	109.5	H22D—C22B—H22F	109.5
H22B—C22A—H22C	109.5	H22E—C22B—H22F	109.5
N2A—C23A—C10A	176.9 (3)	N2B—C23B—C10B	177.5 (3)
C6A—C1A—C2A—C3A	−0.1 (4)	C6B—C1B—C2B—C3B	0.0 (4)
C1A—C2A—C3A—C4A	−0.7 (4)	C1B—C2B—C3B—C4B	0.4 (4)
C1A—C2A—C3A—Br1A	−179.6 (2)	C1B—C2B—C3B—Br1B	−179.6 (2)
C2A—C3A—C4A—C5A	0.7 (4)	C2B—C3B—C4B—C5B	0.5 (5)
Br1A—C3A—C4A—C5A	179.6 (2)	Br1B—C3B—C4B—C5B	−179.5 (2)
C3A—C4A—C5A—C6A	−0.1 (4)	C3B—C4B—C5B—C6B	−1.8 (5)
C2A—C1A—C6A—C5A	0.7 (4)	C2B—C1B—C6B—C5B	−1.3 (4)
C2A—C1A—C6A—C7A	−180.0 (2)	C2B—C1B—C6B—C7B	176.5 (3)
C4A—C5A—C6A—C1A	−0.6 (4)	C4B—C5B—C6B—C1B	2.2 (4)
C4A—C5A—C6A—C7A	−180.0 (2)	C4B—C5B—C6B—C7B	−175.6 (3)
C11A—N1A—C7A—C8A	0.6 (4)	C11B—N1B—C7B—C8B	3.5 (4)
C11A—N1A—C7A—C6A	−178.3 (2)	C11B—N1B—C7B—C6B	−177.2 (2)
C1A—C6A—C7A—N1A	10.6 (4)	C1B—C6B—C7B—N1B	−27.5 (4)
C5A—C6A—C7A—N1A	−170.1 (2)	C5B—C6B—C7B—N1B	150.2 (3)
C1A—C6A—C7A—C8A	−168.3 (2)	C1B—C6B—C7B—C8B	151.9 (3)
C5A—C6A—C7A—C8A	11.0 (4)	C5B—C6B—C7B—C8B	−30.4 (4)
N1A—C7A—C8A—C9A	−1.8 (4)	N1B—C7B—C8B—C9B	0.1 (4)
C6A—C7A—C8A—C9A	177.1 (2)	C6B—C7B—C8B—C9B	−179.2 (2)
C7A—C8A—C9A—C10A	2.4 (4)	C7B—C8B—C9B—C10B	−3.1 (4)
C7A—C8A—C9A—C12A	−175.0 (2)	C7B—C8B—C9B—C12B	177.3 (2)
C8A—C9A—C10A—C11A	−1.8 (4)	C8B—C9B—C10B—C11B	2.6 (4)
C12A—C9A—C10A—C11A	175.5 (2)	C12B—C9B—C10B—C11B	−177.8 (2)
C8A—C9A—C10A—C23A	177.0 (3)	C8B—C9B—C10B—C23B	−173.4 (3)
C12A—C9A—C10A—C23A	−5.7 (4)	C12B—C9B—C10B—C23B	6.2 (4)
C7A—N1A—C11A—O1A	179.6 (2)	C7B—N1B—C11B—O1B	175.4 (2)
C7A—N1A—C11A—C10A	−0.1 (4)	C7B—N1B—C11B—C10B	−4.0 (4)
C18A—O1A—C11A—N1A	1.7 (3)	C18B—O1B—C11B—N1B	−1.4 (4)
C18A—O1A—C11A—C10A	−178.6 (2)	C18B—O1B—C11B—C10B	178.0 (2)
C9A—C10A—C11A—N1A	0.7 (4)	C9B—C10B—C11B—N1B	1.0 (4)
C23A—C10A—C11A—N1A	−178.1 (3)	C23B—C10B—C11B—N1B	177.1 (2)
C9A—C10A—C11A—O1A	−179.0 (2)	C9B—C10B—C11B—O1B	−178.5 (2)
C23A—C10A—C11A—O1A	2.2 (4)	C23B—C10B—C11B—O1B	−2.4 (4)
C10A—C9A—C12A—C13A	−61.4 (4)	C8B—C9B—C12B—C13B	−114.5 (3)
C8A—C9A—C12A—C13A	115.8 (3)	C10B—C9B—C12B—C13B	66.0 (4)
C10A—C9A—C12A—C17A	117.6 (3)	C8B—C9B—C12B—C17B	64.1 (4)
C8A—C9A—C12A—C17A	−65.2 (4)	C10B—C9B—C12B—C17B	−115.5 (3)
C20A—O2A—C13A—C12A	171.4 (3)	C20B—O2B—C13B—C14B	−5.5 (4)
C20A—O2A—C13A—C14A	−11.6 (4)	C20B—O2B—C13B—C12B	173.9 (2)
C17A—C12A—C13A—O2A	176.7 (2)	C17B—C12B—C13B—O2B	−176.9 (2)
C9A—C12A—C13A—O2A	−4.2 (4)	C9B—C12B—C13B—O2B	1.6 (4)
C17A—C12A—C13A—C14A	−0.3 (4)	C17B—C12B—C13B—C14B	2.5 (4)
C9A—C12A—C13A—C14A	178.7 (2)	C9B—C12B—C13B—C14B	−179.0 (3)
O2A—C13A—C14A—C15A	−177.5 (2)	O2B—C13B—C14B—C15B	179.6 (2)

C12A—C13A—C14A—C15A	−0.7 (4)	C12B—C13B—C14B—C15B	0.3 (4)
C21A—O3A—C15A—C14A	0.2 (4)	C21B—O3B—C15B—C14B	3.8 (4)
C21A—O3A—C15A—C16A	180.0 (2)	C21B—O3B—C15B—C16B	−175.5 (2)
C13A—C14A—C15A—O3A	−179.9 (2)	C13B—C14B—C15B—O3B	178.7 (2)
C13A—C14A—C15A—C16A	0.3 (4)	C13B—C14B—C15B—C16B	−2.1 (4)
C22A—O4A—C16A—C17A	8.1 (4)	C22B—O4B—C16B—C17B	−7.7 (4)
C22A—O4A—C16A—C15A	−172.8 (2)	C22B—O4B—C16B—C15B	173.3 (2)
O3A—C15A—C16A—O4A	2.1 (4)	O3B—C15B—C16B—O4B	−0.6 (3)
C14A—C15A—C16A—O4A	−178.0 (2)	C14B—C15B—C16B—O4B	−179.9 (2)
O3A—C15A—C16A—C17A	−178.7 (2)	O3B—C15B—C16B—C17B	−179.6 (2)
C14A—C15A—C16A—C17A	1.1 (4)	C14B—C15B—C16B—C17B	1.1 (4)
O4A—C16A—C17A—C12A	176.9 (2)	O4B—C16B—C17B—C12B	−177.2 (2)
C15A—C16A—C17A—C12A	−2.1 (4)	C15B—C16B—C17B—C12B	1.7 (4)
C13A—C12A—C17A—C16A	1.8 (4)	C13B—C12B—C17B—C16B	−3.5 (4)
C9A—C12A—C17A—C16A	−177.3 (2)	C9B—C12B—C17B—C16B	177.9 (2)
C11A—O1A—C18A—C19A	−174.0 (2)	C11B—O1B—C18B—C19B	92.8 (3)

Hydrogen-bond geometry (Å, °)

Cg1 and Cg2 are the centroids of C7A–C11A/N1A and C12A–C17A rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1A—H1AA···N1A	0.93	2.43	2.770 (3)	102
C18B—H18C···N1B	0.97	2.38	2.741 (4)	101
C22A—H22C···Cg2^{i}	0.96	2.74	3.585 (3)	147
C22B—H22E···Cg1^{ii}	0.96	2.68	3.511 (3)	145

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*, −*y*+1, −*z*+1.

Fig. 1

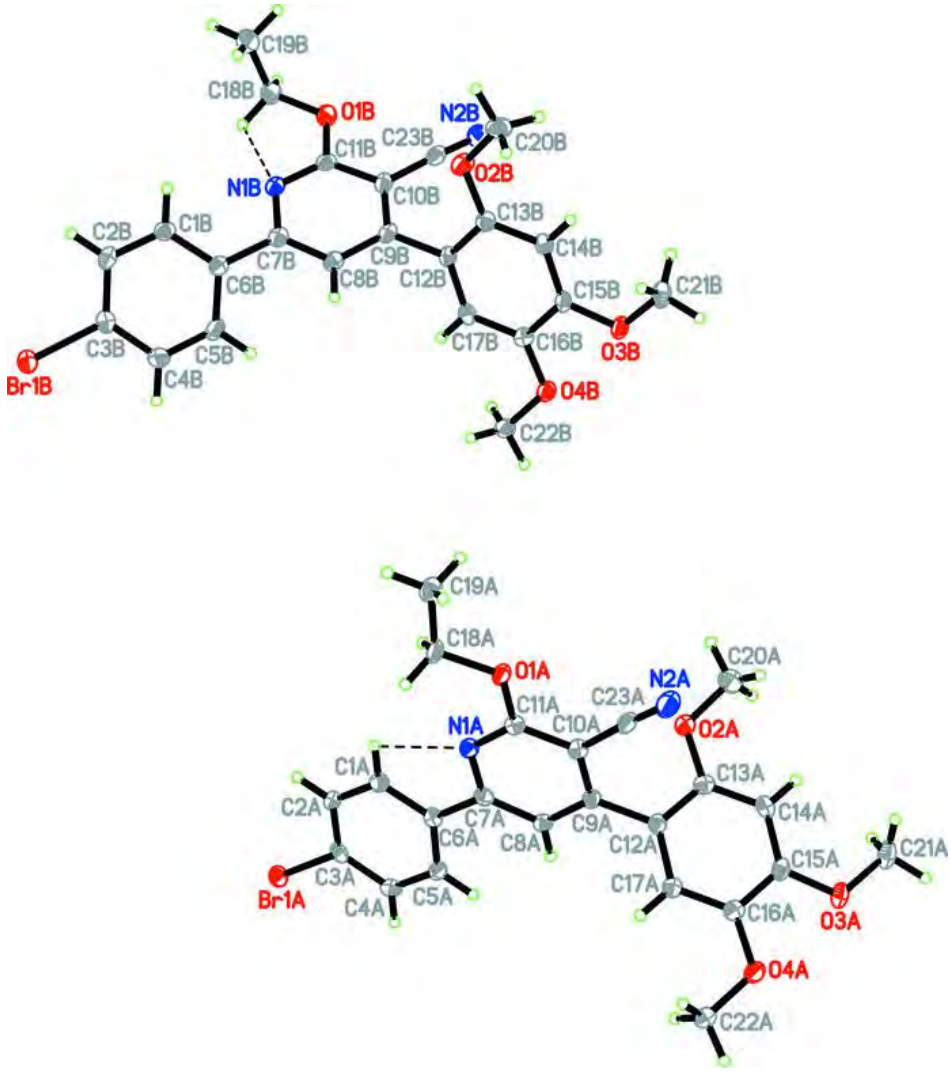
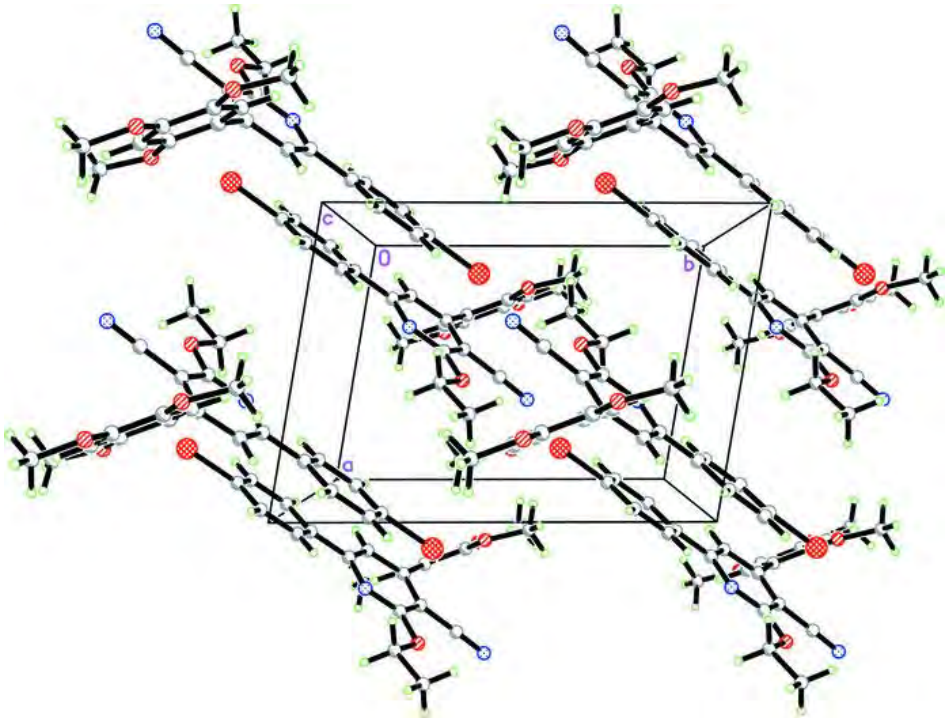


Fig. 2

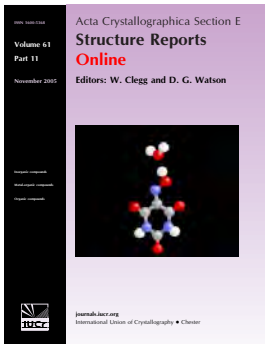


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-3-(Anthracen-9-yl)-1-(furan-2-yl)prop-2-en-1-one

Jirapa Horkaew, Thitipone Suwunwong, Suchada Chantrapromma, Chatchanok Karalai and Hoong-Kun Fun

Acta Cryst. (2010). E**66**, o800–o801



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-3-(Anthracen-9-yl)-1-(furan-2-yl)-prop-2-en-1-one[1]

Jirapa Horkaew,[a] Thitipone Suwunwong,[a] Suchada Chantrapromma,[a]*§ Chatchanok Karalai[a] and Hoong-Kun Fun[b]¶

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

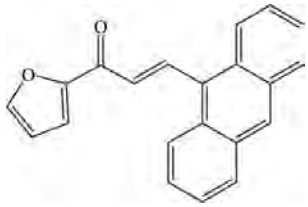
Received 1 February 2010; accepted 14 February 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.118; data-to-parameter ratio = 16.1.

In the molecule of the title heteroaryl chalcone derivative, $C_{21}H_{14}O_2$, the almost planar prop-2-en-1-one unit [r.m.s. deviation = 0.0087 (1) Å] forms dihedral angles of 5.81 (7) and 49.85 (6)°, respectively, with the furan ring and anthracene ring system. In the crystal structure, the molecules are linked into a two-dimensional network parallel to (100) by C—H···O hydrogen bonds and $\pi\cdots\pi$ interactions involving the furan rings [centroid–centroid distance = 3.7205 (6) Å].

Related literature

For background and applications of chalcones, see: Gaber *et al.* (2008); Niu *et al.* (2006); Xu *et al.* (2005). For related structures, see: Chantrapromma *et al.* (2009, 2010); Fun *et al.* (2009); Suwunwong *et al.* (2009). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{21}H_{14}O_2$
M_r = 298.32
Monoclinic, $P2_1/c$
a = 21.5743 (4) Å
b = 5.4571 (1) Å
c = 12.8394 (2) Å
β = 104.099 (1)°
V = 1466.09 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.55 × 0.25 × 0.07 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.955, T_{max} = 0.994
19468 measured reflections
4251 independent reflections
3549 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.118
S = 1.03
4251 reflections
264 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.98 (2)	2.34 (2)	3.2871 (14)	165 (1)
C6—H6···O1^{i}	0.94 (2)	2.40 (2)	3.3366 (13)	173 (1)
C19—H19···O1^{ii}	0.98 (1)	2.47 (1)	3.3419 (13)	148 (1)

Symmetry codes: (i) $x, y+1, z$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for research grant (RSA 5280033) and Prince of Songkla University for financial support. They also thank Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5032).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chantrapromma, S., Horkaew, J., Suwunwong, T. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o2673–o2674.
Chantrapromma, S., Suwunwong, T., Boonnak, N. & Fun, H.-K. (2010). *Acta Cryst.* E**66**, o312–o313.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Fun, H.-K., Suwunwong, T., Boonnak, N. & Chantrapromma, S. (2009). *Acta Cryst.* E**65**, o2168–o2169.
Gaber, M., El-Daly, S. A., Fayed, T. A. & El-Sayed, Y. S. (2008). *Opt. Laser Tech.* **40**, 528–537.
Niu, C.-G., Guan, A.-L., Zeng, G.-M., Liu, Y.-G. & Li, Z.-W. (2006). *Anal. Chim. Acta*, **577**, 264–270.

[1] This paper is dedicated to His Majesty King Bhumibol Adulyadej of Thailand (King Rama IX) for his sustainable development of the country.
§ Thomson Reuters ResearcherID: A-5085-2009.
¶ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suwunwong, T., Chantrapromma, S., Karalai, C., Pakdeevanich, P. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o420–o421.
Xu, Z., Bai, G. & Dong, C. (2005). *Spectrochim. Acta Part A*, **62**, 987–990.

supplementary materials

Acta Cryst. (2010). E**66**, o800-o801 [**doi:10.1107/S1600536810005982**]

(*E*)-3-(Anthracen-9-yl)-1-(furan-2-yl)prop-2-en-1-one

J. Horkaew, T. Suwunwong, S. Chantrapromma, C. Karalai and H.-K. Fun

Comment

Chalcones have been studied for their wide range of applications including laser activity (Gaber *et al.*, 2008) and fluorescence properties (Niu *et al.*, 2006; Xu *et al.*, 2005). We have previously reported crystal structures of several chalcone derivatives containing the anthracene moiety which exist in *E* configuration (Suwunwong *et al.*, 2009) or *Z* configuration (Chantrapromma *et al.*, 2009, 2010; Fun *et al.*, 2009). The title compound was synthesized on account of its fluorescence properties. The crystal structure determination was undertaken to elucidate its conformation and to study the structure and fluorescence activity relationship.

The molecule of the title chalcone derivative (Fig. 1) exists in an *E* configuration with respect to the C6═C7 ethenyl bond, with a C5—C6—C7—C8 torsion angle of 178.00 (9)°. The anthracene ring system (C8–C21) is essentially planar (r.m.s. deviation = 0.0258 (1) Å). The prop-2-en-1-one unit (C5–C7/O1) is also planar (r.m.s. deviation = 0.0087 (1) Å; O1—C5—C6—C7 = 2.92 (15)°) and it forms dihedral angles of 5.81 (7) and 49.85 (6)°, respectively, with the furan ring and anthracene ring system. The interplanar angle between the furan ring and anthracene ring system is 48.53 (5)°. The bond distances show normal values (Allen *et al.*, 1987) and are comparable with those in closely related structures (Chantrapromma, Horkaew *et al.*, 2009; Chantrapromma, Suwunwong *et al.*, 2010; Fun *et al.*, 2009; Suwunwong *et al.*, 2009).

In the crystal structure, the molecules are linked into a two-dimensional network parallel to the (100) by C—H···O hydrogen bonds (Fig. 2 and Table 1) and π···π interactions between the furan rings at (x, y, z) and (-x, 1-y, -z) [centroid···centroid distance = 3.7205 (6) Å].

Experimental

The title compound was synthesized by the condensation of anthracene-9-carbaldehyde (0.41 g, 2 mmol) with 2-furylmethylketone (0.22 g, 2 mmol) in ethanol (30 ml) in the presence of 30 % aqueous NaOH (5 ml) at room temperature. The reaction mixture was stirred at 278 K for 3 h and then a yellow solid appeared was collected by filtration, washed with acetone and dried in air. Yellow plate-shaped single crystals of the title compound suitable for *X*-ray structure determination were recrystalized from acetone-ethanol (1:1 v/v) by slow evaporation of the solvent at room temperature after several days (m.p. 423-424 K).

Refinement

All H atoms were located in a difference map and refined isotropically [C–H = 0.941 (15)–1.009 (14) Å].

Figures

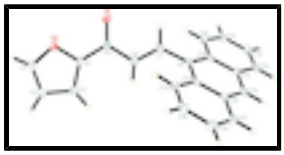


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

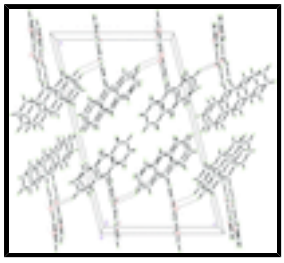


Fig. 2. The crystal packing of the title compound viewed along the *b* axis. C—H···O hydrogen bonds are shown as dashed lines.

(*E*)-3-(anthracen-9-yl)-1-(furan-2-yl)prop-2-en-1-one

Crystal data

$C_{21}H_{14}O_2$	$F(000) = 624$
$M_r = 298.32$	$D_x = 1.352$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point = 423–424 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 21.5743$ (4) Å	Cell parameters from 4251 reflections
$b = 5.4571$ (1) Å	$\theta = 2.9$–$30.0°$
$c = 12.8394$ (2) Å	$\mu = 0.09$ mm^{-1}
$\beta = 104.099$ (1)°	$T = 100$ K
$V = 1466.09$ (4) Å^3	Plate, yellow
$Z = 4$	0.55 × 0.25 × 0.07 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	4251 independent reflections
Radiation source: sealed tube	3549 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.029$
φ and ω scans	$\theta_{max} = 30.0°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -26 \rightarrow 30$
$T_{min} = 0.955$, $T_{max} = 0.994$	$k = -7 \rightarrow 7$
19468 measured reflections	$l = -17 \rightarrow 18$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.118$	All H-atom parameters refined
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.4737P]$ where $P = (F_o^2 + 2F_c^2)/3$
4251 reflections	$(\Delta/\sigma)_{max} = 0.001$
264 parameters	$\Delta\rho_{max} = 0.37$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor wR and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) etc. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.13071 (4)	0.32065 (14)	0.15881 (6)	0.02003 (17)
O2	0.00592 (3)	0.46137 (14)	0.12589 (6)	0.01846 (17)
C1	−0.04942 (5)	0.5903 (2)	0.11274 (9)	0.0201 (2)
H1	−0.0880 (7)	0.489 (3)	0.1018 (12)	0.030 (4)*
C2	−0.03863 (5)	0.8346 (2)	0.11454 (9)	0.0208 (2)
H2	−0.0701 (8)	0.964 (3)	0.1084 (13)	0.034 (4)*
C3	0.02854 (5)	0.8641 (2)	0.12983 (8)	0.0177 (2)
H3	0.0517 (7)	1.019 (3)	0.1342 (11)	0.027 (4)*
C4	0.05367 (5)	0.63332 (19)	0.13651 (8)	0.0152 (2)
C5	0.11877 (5)	0.54141 (19)	0.15040 (8)	0.0150 (2)
C6	0.16862 (5)	0.72918 (19)	0.15159 (8)	0.0163 (2)
C7	0.22914 (5)	0.66214 (19)	0.15739 (8)	0.0159 (2)
C8	0.28183 (5)	0.83333 (19)	0.15569 (8)	0.01475 (19)
C9	0.34004 (5)	0.80973 (19)	0.23544 (8)	0.0153 (2)
C10	0.34956 (5)	0.6249 (2)	0.31680 (8)	0.0183 (2)
C11	0.40571 (5)	0.6100 (2)	0.39433 (9)	0.0214 (2)
C12	0.45582 (5)	0.7804 (2)	0.39679 (9)	0.0231 (2)
C13	0.44862 (5)	0.9614 (2)	0.32146 (9)	0.0214 (2)
C14	0.39083 (5)	0.9814 (2)	0.23836 (8)	0.0166 (2)
C15	0.38256 (5)	1.1693 (2)	0.16251 (8)	0.0181 (2)
C16	0.32655 (5)	1.19033 (19)	0.08122 (8)	0.0164 (2)

C17	0.31916 (5)	1.3816 (2)	0.00291 (9)	0.0201 (2)
C18	0.26502 (6)	1.3994 (2)	−0.07801 (9)	0.0218 (2)
C19	0.21559 (5)	1.2226 (2)	−0.08673 (8)	0.0209 (2)
C20	0.22049 (5)	1.0396 (2)	−0.01267 (8)	0.0183 (2)
C21	0.27525 (5)	1.01872 (19)	0.07639 (8)	0.0154 (2)
H6	0.1563 (7)	0.895 (3)	0.1471 (11)	0.021 (3)*
H7	0.2399 (6)	0.482 (3)	0.1653 (11)	0.019 (3)*
H10	0.3158 (7)	0.507 (3)	0.3176 (11)	0.024 (4)*
H11	0.4105 (8)	0.479 (3)	0.4498 (13)	0.035 (4)*
H12	0.4960 (7)	0.766 (3)	0.4544 (13)	0.033 (4)*
H13	0.4831 (8)	1.086 (3)	0.3216 (13)	0.032 (4)*
H15	0.4171 (7)	1.291 (3)	0.1670 (11)	0.024 (4)*
H17	0.3534 (7)	1.501 (3)	0.0097 (12)	0.027 (4)*
H18	0.2611 (8)	1.533 (3)	−0.1306 (13)	0.036 (4)*
H19	0.1780 (7)	1.232 (3)	−0.1469 (11)	0.025 (4)*
H20	0.1864 (7)	0.916 (3)	−0.0210 (11)	0.025 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0184 (4)	0.0134 (4)	0.0265 (4)	−0.0007 (3)	0.0020 (3)	0.0008 (3)
O2	0.0144 (3)	0.0166 (4)	0.0236 (4)	−0.0032 (3)	0.0031 (3)	0.0005 (3)
C1	0.0134 (5)	0.0248 (5)	0.0216 (5)	−0.0011 (4)	0.0031 (4)	0.0001 (4)
C2	0.0154 (5)	0.0222 (5)	0.0244 (5)	0.0021 (4)	0.0042 (4)	0.0002 (4)
C3	0.0165 (5)	0.0165 (5)	0.0196 (5)	−0.0007 (4)	0.0034 (4)	−0.0002 (4)
C4	0.0142 (4)	0.0148 (5)	0.0161 (4)	−0.0026 (4)	0.0027 (3)	0.0000 (3)
C5	0.0147 (4)	0.0143 (5)	0.0149 (4)	−0.0017 (3)	0.0016 (3)	−0.0006 (3)
C6	0.0161 (5)	0.0125 (4)	0.0199 (4)	−0.0013 (4)	0.0038 (4)	−0.0004 (4)
C7	0.0163 (5)	0.0139 (5)	0.0170 (4)	−0.0011 (4)	0.0030 (3)	−0.0004 (3)
C8	0.0136 (4)	0.0137 (4)	0.0175 (4)	−0.0003 (3)	0.0047 (3)	−0.0019 (3)
C9	0.0140 (4)	0.0155 (5)	0.0173 (4)	0.0008 (4)	0.0051 (3)	−0.0012 (4)
C10	0.0162 (5)	0.0180 (5)	0.0212 (5)	0.0012 (4)	0.0054 (4)	0.0007 (4)
C11	0.0190 (5)	0.0232 (5)	0.0219 (5)	0.0047 (4)	0.0049 (4)	0.0032 (4)
C12	0.0156 (5)	0.0286 (6)	0.0232 (5)	0.0024 (4)	0.0011 (4)	0.0001 (4)
C13	0.0139 (5)	0.0248 (6)	0.0245 (5)	−0.0009 (4)	0.0027 (4)	−0.0015 (4)
C14	0.0135 (4)	0.0177 (5)	0.0192 (4)	−0.0002 (4)	0.0050 (4)	−0.0026 (4)
C15	0.0157 (5)	0.0184 (5)	0.0213 (5)	−0.0025 (4)	0.0067 (4)	−0.0012 (4)
C16	0.0167 (5)	0.0153 (5)	0.0190 (4)	−0.0003 (4)	0.0075 (4)	−0.0014 (4)
C17	0.0217 (5)	0.0176 (5)	0.0234 (5)	−0.0010 (4)	0.0105 (4)	0.0013 (4)
C18	0.0259 (6)	0.0213 (5)	0.0207 (5)	0.0027 (4)	0.0106 (4)	0.0039 (4)
C19	0.0204 (5)	0.0243 (6)	0.0175 (5)	0.0024 (4)	0.0038 (4)	0.0011 (4)
C20	0.0170 (5)	0.0193 (5)	0.0184 (4)	−0.0003 (4)	0.0037 (4)	−0.0003 (4)
C21	0.0150 (4)	0.0149 (5)	0.0168 (4)	0.0006 (4)	0.0049 (3)	−0.0011 (4)

Geometric parameters (Å, °)

O1—C5	1.2312 (13)	C10—H10	0.973 (15)
O2—C1	1.3606 (13)	C11—C12	1.4209 (17)
O2—C4	1.3754 (12)	C11—H11	0.997 (17)

C1—C2	1.3526 (16)	C12—C13	1.3642 (17)
C1—H1	0.981 (16)	C12—H12	0.996 (16)
C2—C3	1.4235 (15)	C13—C14	1.4342 (14)
C2—H2	0.968 (17)	C13—H13	1.007 (16)
C3—C4	1.3654 (14)	C14—C15	1.3950 (15)
C3—H3	0.974 (15)	C15—C16	1.3959 (14)
C4—C5	1.4609 (14)	C15—H15	0.988 (15)
C5—C6	1.4827 (14)	C16—C17	1.4310 (15)
C6—C7	1.3406 (14)	C16—C21	1.4396 (14)
C6—H6	0.941 (15)	C17—C18	1.3645 (16)
C7—C8	1.4757 (14)	C17—H17	0.972 (15)
C7—H7	1.009 (14)	C18—C19	1.4225 (17)
C8—C21	1.4176 (14)	C18—H18	0.984 (17)
C8—C9	1.4196 (14)	C19—C20	1.3653 (15)
C9—C10	1.4303 (14)	C19—H19	0.975 (14)
C9—C14	1.4353 (14)	C20—C21	1.4346 (14)
C10—C11	1.3701 (15)	C20—H20	0.984 (15)
C1—O2—C4	105.84 (8)	C10—C11—H11	118.7 (9)
C2—C1—O2	111.43 (10)	C12—C11—H11	120.6 (9)
C2—C1—H1	134.0 (9)	C13—C12—C11	120.23 (10)
O2—C1—H1	114.6 (9)	C13—C12—H12	120.8 (9)
C1—C2—C3	106.20 (10)	C11—C12—H12	119.0 (9)
C1—C2—H2	127.0 (10)	C12—C13—C14	120.72 (10)
C3—C2—H2	126.8 (10)	C12—C13—H13	122.1 (9)
C4—C3—C2	106.24 (9)	C14—C13—H13	117.2 (9)
C4—C3—H3	127.2 (9)	C15—C14—C13	121.04 (10)
C2—C3—H3	126.6 (9)	C15—C14—C9	119.63 (9)
C3—C4—O2	110.29 (9)	C13—C14—C9	119.30 (10)
C3—C4—C5	132.80 (9)	C14—C15—C16	121.51 (10)
O2—C4—C5	116.90 (9)	C14—C15—H15	119.0 (8)
O1—C5—C4	121.40 (9)	C16—C15—H15	119.5 (8)
O1—C5—C6	122.66 (9)	C15—C16—C17	120.89 (10)
C4—C5—C6	115.93 (9)	C15—C16—C21	119.67 (9)
C7—C6—C5	120.41 (10)	C17—C16—C21	119.43 (9)
C7—C6—H6	121.6 (9)	C18—C17—C16	121.01 (10)
C5—C6—H6	118.0 (9)	C18—C17—H17	120.7 (9)
C6—C7—C8	124.74 (10)	C16—C17—H17	118.3 (9)
C6—C7—H7	117.9 (8)	C17—C18—C19	119.86 (10)
C8—C7—H7	117.4 (8)	C17—C18—H18	119.4 (9)
C21—C8—C9	119.94 (9)	C19—C18—H18	120.8 (9)
C21—C8—C7	121.30 (9)	C20—C19—C18	120.81 (10)
C9—C8—C7	118.76 (9)	C20—C19—H19	120.0 (9)
C8—C9—C10	122.36 (9)	C18—C19—H19	119.2 (9)
C8—C9—C14	119.68 (9)	C19—C20—C21	121.48 (10)
C10—C9—C14	117.92 (9)	C19—C20—H20	119.7 (8)
C11—C10—C9	121.13 (10)	C21—C20—H20	118.8 (8)
C11—C10—H10	118.9 (8)	C8—C21—C20	123.23 (9)
C9—C10—H10	119.9 (8)	C8—C21—C16	119.46 (9)
C10—C11—C12	120.68 (10)	C20—C21—C16	117.25 (9)

C4—O2—C1—C2	0.05 (12)	C12—C13—C14—C15	178.54 (10)
O2—C1—C2—C3	0.07 (13)	C12—C13—C14—C9	0.46 (16)
C1—C2—C3—C4	−0.16 (12)	C8—C9—C14—C15	−0.25 (15)
C2—C3—C4—O2	0.20 (11)	C10—C9—C14—C15	−177.91 (9)
C2—C3—C4—C5	179.04 (10)	C8—C9—C14—C13	177.85 (9)
C1—O2—C4—C3	−0.16 (11)	C10—C9—C14—C13	0.20 (15)
C1—O2—C4—C5	−179.20 (8)	C13—C14—C15—C16	179.91 (10)
C3—C4—C5—O1	176.89 (11)	C9—C14—C15—C16	−2.01 (16)
O2—C4—C5—O1	−4.33 (14)	C14—C15—C16—C17	−178.97 (10)
C3—C4—C5—C6	−4.22 (16)	C14—C15—C16—C21	1.51 (15)
O2—C4—C5—C6	174.56 (8)	C15—C16—C17—C18	178.56 (10)
O1—C5—C6—C7	2.92 (15)	C21—C16—C17—C18	−1.92 (16)
C4—C5—C6—C7	−175.95 (9)	C16—C17—C18—C19	−1.51 (17)
C5—C6—C7—C8	178.00 (9)	C17—C18—C19—C20	2.45 (17)
C6—C7—C8—C21	−49.44 (15)	C18—C19—C20—C21	0.14 (17)
C6—C7—C8—C9	130.63 (11)	C9—C8—C21—C20	173.77 (9)
C21—C8—C9—C10	−179.46 (9)	C7—C8—C21—C20	−6.16 (15)
C7—C8—C9—C10	0.48 (15)	C9—C8—C21—C16	−3.49 (15)
C21—C8—C9—C14	2.99 (15)	C7—C8—C21—C16	176.57 (9)
C7—C8—C9—C14	−177.07 (9)	C19—C20—C21—C8	179.21 (10)
C8—C9—C10—C11	−178.45 (10)	C19—C20—C21—C16	−3.47 (15)
C14—C9—C10—C11	−0.86 (15)	C15—C16—C21—C8	1.27 (15)
C9—C10—C11—C12	0.87 (17)	C17—C16—C21—C8	−178.26 (9)
C10—C11—C12—C13	−0.18 (18)	C15—C16—C21—C20	−176.16 (9)
C11—C12—C13—C14	−0.48 (18)	C17—C16—C21—C20	4.31 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.98 (2)	2.34 (2)	3.2871 (14)	165 (1)
C6—H6···O1^{i}	0.94 (2)	2.40 (2)	3.3366 (13)	173 (1)
C19—H19···O1^{ii}	0.98 (1)	2.47 (1)	3.3419 (13)	148 (1)

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*, −*y*+3/2, *z*−1/2.

Fig. 1

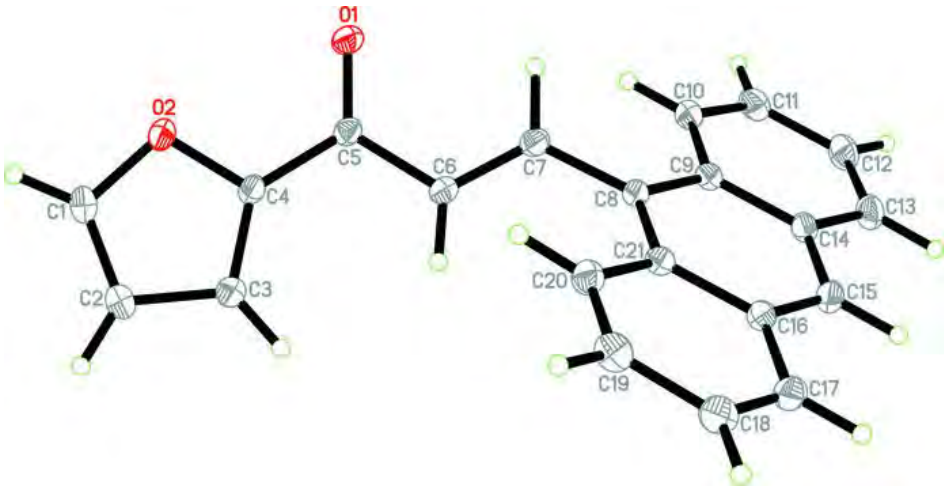
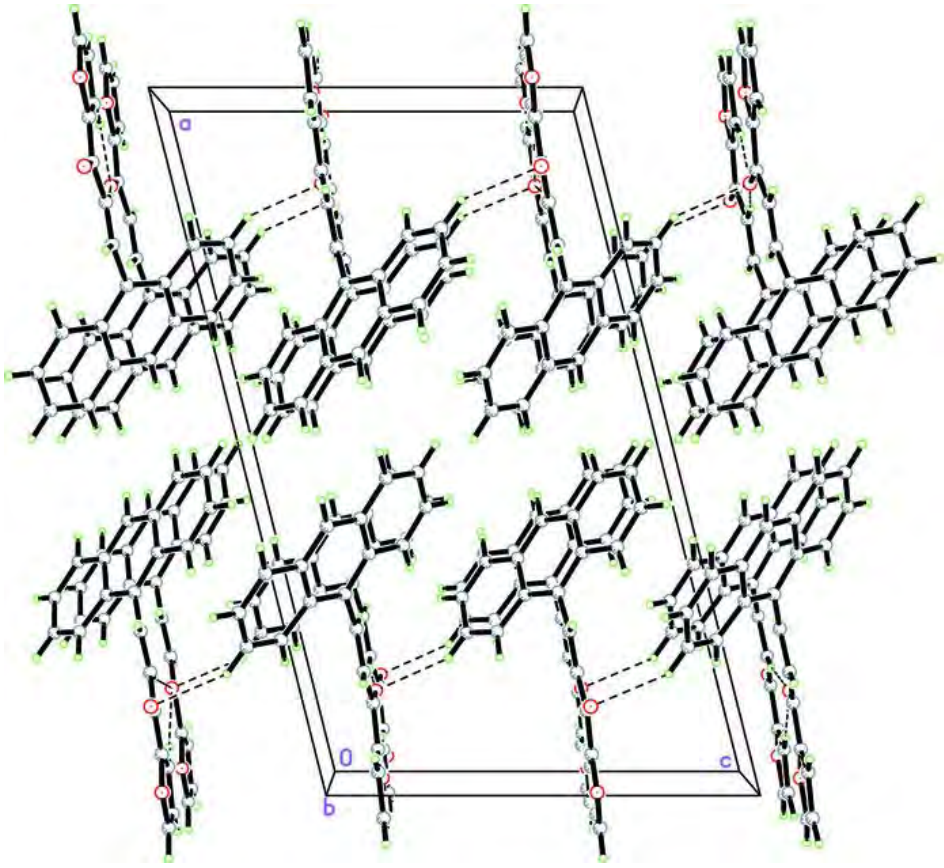


Fig. 2

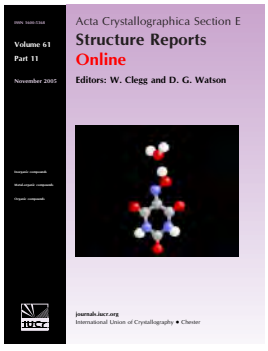


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-1-(4-Aminophenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun, Thawanrat Kobkeatthawin, Pumsak Ruanwas and Suchada Chantrapromma

Acta Cryst. (2010). E**66**, o1973–o1974



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(4-Aminophenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun,[a]*‡ Thawanrat Kobkeatthawin,[b] Pumsak Ruanwas[b] and Suchada Chantrapromma[b]§

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

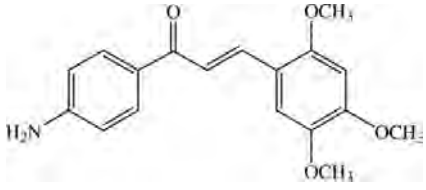
Received 2 July 2010; accepted 4 July 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.051; wR factor = 0.131; data-to-parameter ratio = 20.6.

Molecules of the title aminochalcone, $C_{18}H_{19}NO_4$, are twisted, with a dihedral angle of 11.26 (6)° between the 4-aminophenyl and 2,4,5-trimethoxyphenyl rings. The conformations of the three methoxy groups with respect to the benzene ring are slightly different. Two methoxy groups are almost coplanar with the attached benzene ring [C—O—C—C torsion angles of −1.45 (19) and 1.5 (2)°], while the third is (−)-synclinal with the attached benzene ring [C—O—C—C = −81.36 (17)°]. In the crystal structure, molecules are stacked into columns along the *b* axis and molecules in adjacent columns are linked by N—H···O hydrogen bonds into V-shaped double columns. Weak π–π interactions are also observed, with a centroid-centroid distance of 3.7532 (8) Å.

Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Chantrapromma *et al.* (2009, 2010); Suwunwong *et al.* (2009). For background to and applications of chalcones, see: Batovska *et al.* (2007); Jung *et al.* (2008); Kim *et al.* (2010); Nielsen *et al.* (2004); Niu *et al.* (2006); Romagnoli *et al.* (2008); Tewtrakul *et al.* (2003); Won *et al.* (2005); Xia *et al.* (2000).



Experimental

Crystal data

$C_{18}H_{19}NO_4$
M_r = 313.34
Monoclinic, $C2/c$
a = 13.6117 (2) Å
b = 10.3540 (2) Å
c = 22.3920 (4) Å
β = 100.879 (1)°
V = 3099.11 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.38 × 0.32 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.965, T_{max} = 0.991
19818 measured reflections
4506 independent reflections
3581 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.131
S = 1.05
4506 reflections
219 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···O1^{i}	0.86 (2)	2.12 (2)	2.9692 (16)	170.4 (17)
N1—H2*N*1···O1^{ii}	0.88 (2)	2.21 (2)	3.0176 (17)	153.4 (19)

Symmetry codes: (i) $x-\frac{1}{2}, y+\frac{1}{2}, z$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for the research grant (RSA5280033), Prince of Songkla University and Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012. TK and PR thank the Graduate School, Prince of Songkla University, for partial financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2326).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Batovska, D., Parushev, St., Slavova, A., Bankova, V., Tsvetkova, I., Ninova, M. & Najdenski, H. (2007). *Eur. J. Med. Chem.* **42**, 87–92.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chantrapromma, S., Suwunwong, T., Boonnak, N. & Fun, H.-K. (2010). *Acta Cryst.* E**66**, o312–o313.
Chantrapromma, S., Suwunwong, T., Karalai, C. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o893–o894.
Jung, Y. J., Son, K. I., Oh, Y. E. & Noh, D. Y. (2008). *Polyhedron*. **27**, 861–867.

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Kim, E.-J., Ryu, H. W., Curtis-Long, M. J., Han, J., Kim, J. Y., Cho, J. K., Kang, D. & Park, K. H. (2010). *Bioorg. Med. Chem. Lett*. **20**, 4237–4239.
Nielsen, S. F., Boesen, T., Larsen, M., Schønning, K. & Kromann, H. (2004). *Bioorg. Med. Chem*. **12**, 3047–3054.
Niu, C. G., Guan, A. L., Zeng, G. M., Liu, Y. G. & Li, Z. W. (2006). *Anal. Chim. Acta*, **577**, 264–270.
Romagnoli, R., Baraldi, P. G., Carrion, M. D., Cara, C. L., Cruz-Lopez, O., Preti, D., Tolomeo, M., Grimaudo, S., Cristina, A. D., Zonta, N., Balzarini, J., Brancale, A., Sarkar, T. & Hamel, E. (2008). *Bioorg. Med. Chem*. **16**, 5367–5376.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst*. D**65**, 148–155.
Suwunwong, T., Chantrapromma, S., Pakdeevanich, P. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o1575–o1576.
Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol*. **25**, 503–508.
Won, S. J., Liu, C. T., Tsao, L. T., Weng, J. R., Ko, H. H., Wang, J. P. & Lin, C. N. (2005). *Eur. J. Med. Chem*. **40**, 103–112.
Xia, Y., Yang, Z.-Y., Xia, P., Bastow, K. F., Nakanishi, Y. & Lee, K.-H. (2000). *Bioorg. Med. Chem. Lett*. **10**, 699–701.

supplementary materials

Acta Cryst. (2010). E**66**, o1973-o1974 [**doi:10.1107/S1600536810026346**]

(*E*)-1-(4-Aminophenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

H.-K. Fun, T. Kobkeatthawin, P. Ruanwas and S. Chantrapromma

Comment

Chalcones are important compounds which have considerable interesting applications involving biological activities such as antibacterial (Nielsen *et al.*, 2004), antifungal (Batovska *et al.*, 2007), anti-inflammatory (Won *et al.*, 2005), anti-HIV-1 protease inhibition (Tewtrakul *et al.*, 2003) and antitumor (Romagnoli *et al.*, 2008) properties. Moreover they also show electroactive fluorescent properties (Jung *et al.*, 2008; Niu *et al.*, 2006). From previous reports, aminochalcone showed good cytotoxicity in *vitro* against human tumor cell lines (Xia *et al.*, 2000) and was a new class of both activator and inhibitor of the TREK2 channel which turn out to be an important channel in charge of tuning neuronal transmitter and hormone levels (Kim *et al.*, 2010). In continuing our on-going research on bioactivity and fluorescence properties of chalcones (Chantrapromma *et al.*, 2009; 2010; Suwunwong *et al.*, 2009), the title aminochalcone (I) was synthesized. (I) doesn't possess antibacterial and cytotoxic activities. However (I) exhibits fluorescence with the maximum emission at 480 nm when the compound is excited at 320 nm in chloroform solution.

The molecule of the title aminochalcone, (Fig. 1), exists in an *E* configuration with respect to the C8═C9 double bond [1.3407 (19) Å] with torsion angle C7–C8–C9–C10 = 177.99 (13)°. The whole molecule is not planar indicated by the dihedral angle between 4-aminophenyl and 2,4,5-trimethoxyphenyl rings being 11.26 (6)°. The propenone unit (C7—C9/O1) is planar with the *r.m.s*. deviation 0.0031 (1) Å. The mean plane through the propenone unit makes dihedral angles of 11.70 (9) and 9.19 (9)° respectively with the planes of 4-aminophenyl and 2,4,5-trimethoxyphenyl rings. The three methoxy groups of 2,4,5-trimethoxyphenyl unit have two different orientations: two methoxy groups (at atom C11 and C13 positions) are co-planar with the attached benzene ring with torsion angles C16–O2–C11–C12 = -1.45 (19)° and C17–O3–C13–C12 = 1.5 (2)° whereas the one at atom C14 position is (-)-*syn*-clinally attached with the torsion angle C18–O4–C14–C13 = -81.36 (17)°. Weak C9—H9A···O1 and C9—H9A···O2 intramolecular interactions generate two S(5) motifs (Bernstein *et al.*, 1995) (Fig. 1 and Table 1). The bond distances and angles are of normal values (Allen *et al.*, 1987) and are comparable with the related structures (Chantrapromma *et al.*, 2009; 2010; Suwunwong *et al.*, 2009).

In the crystal packing (Fig. 2), the molecules are stacked into columns along the *b* axis. The molecules in the adjacent columns are linked by N—H···O hydrogen bonds (Table 1) into a V-shape double columns. π–π interaction was observed with the $Cg_1\cdots Cg_2$ distance of 3.7532 (8) Å (symmetry code -1/2 - *x*, 3/2 - *y*, 1 - *z*); Cg_1 and Cg_2 are the centroids of C1–C6 and C10–C15 rings, respectively.

Experimental

The title compound was synthesized by the condensation of 2,4,5-trimethoxybenzaldehyde (0.40 g, 2 mmol) with 4-amino-acetophenone (0.28 g, 2 mmol) in ethanol (30 ml) in the presence of 30% NaOH(aq) (5 ml). After stirring for 3 h at room temperature, the resulting orange solid appeared and was then collected by filtration, washed with distilled water, dried and purified by repeated recrystallization from acetone. Orange block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystallized from acetone by the slow evaporation of the solvent at room temperature after several days, Mp. 393–394 K.

Refinement

Amino H atoms were located in difference maps and refined isotropically. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C—H) = 0.93 Å for aromatic and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.74 Å from H18C and the deepest hole is located at 1.39 Å from C15.

Figures

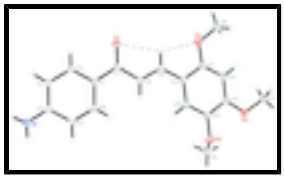


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. The dashed lines represent the intramolecular C—H···O interactions.

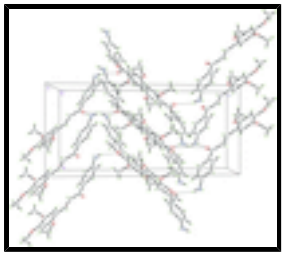


Fig. 2. The crystal packing of the title compound viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

(*E*)-1-(4-Aminophenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{18}H_{19}NO_4$	$F(000) = 1328$
$M_r = 313.34$	$D_x = 1.343$ Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point = 393–394 K
Hall symbol: -C 2yc	Mo *K*α radiation, λ = 0.71073 Å
a = 13.6117 (2) Å	Cell parameters from 4506 reflections
b = 10.3540 (2) Å	θ = 1.9–30.0°
c = 22.3920 (4) Å	μ = 0.10 mm^{-1}
β = 100.879 (1)°	*T* = 100 K
V = 3099.11 (9) Å^3	Block, orange
Z = 8	0.38 × 0.32 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	4506 independent reflections
Radiation source: sealed tube	3581 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.029$
φ and ω scans	$\theta_{max} = 30.0°$, $\theta_{min} = 1.9°$

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -19 \rightarrow 19$
$T_{min} = 0.965$, $T_{max} = 0.991$	$k = -14 \rightarrow 12$
19818 measured reflections	$l = -31 \rightarrow 31$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.051$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.131$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 3.4363P]$ where $P = (F_o^2 + 2F_c^2)/3$
4506 reflections	$(\Delta/\sigma)_{max} < 0.001$
219 parameters	$\Delta\rho_{max} = 0.43$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.38020 (7)	0.71239 (11)	0.65434 (5)	0.0236 (2)
O2	0.40102 (7)	0.40284 (10)	0.49618 (5)	0.0226 (2)
O3	0.10593 (7)	0.26403 (10)	0.35317 (5)	0.0237 (2)
O4	−0.00667 (7)	0.42294 (10)	0.40878 (5)	0.0230 (2)
N1	0.09228 (9)	1.16277 (13)	0.71073 (6)	0.0226 (3)
H1N1	0.0289 (15)	1.1700 (18)	0.6971 (8)	0.028 (5)*
H2N1	0.1136 (15)	1.197 (2)	0.7468 (9)	0.036 (5)*
C1	0.23640 (9)	0.84035 (13)	0.65403 (6)	0.0165 (3)
C2	0.28745 (9)	0.92608 (13)	0.69789 (6)	0.0169 (3)
H2A	0.3552	0.9129	0.7131	0.020*
C3	0.23973 (10)	1.02891 (13)	0.71892 (6)	0.0174 (3)

H3A	0.2749	1.0829	0.7486	0.021*
C4	0.13779 (10)	1.05267 (13)	0.69558 (6)	0.0171 (3)
C5	0.08494 (9)	0.96364 (14)	0.65447 (6)	0.0183 (3)
H5A	0.0166	0.9746	0.6407	0.022*
C6	0.13318 (10)	0.86001 (14)	0.63425 (6)	0.0182 (3)
H6A	0.0968	0.8020	0.6070	0.022*
C7	0.29247 (10)	0.73650 (13)	0.63014 (6)	0.0177 (3)
C8	0.24234 (10)	0.66396 (14)	0.57644 (6)	0.0201 (3)
H8A	0.1762	0.6835	0.5598	0.024*
C9	0.28853 (9)	0.57075 (13)	0.55064 (6)	0.0177 (3)
H9A	0.3553	0.5549	0.5673	0.021*
C10	0.24334 (9)	0.49221 (13)	0.49882 (6)	0.0163 (3)
C11	0.30112 (9)	0.40495 (13)	0.47178 (6)	0.0174 (3)
C12	0.25784 (10)	0.32686 (13)	0.42310 (6)	0.0185 (3)
H12A	0.2973	0.2697	0.4059	0.022*
C13	0.15546 (10)	0.33465 (13)	0.40038 (6)	0.0181 (3)
C14	0.09645 (9)	0.42119 (13)	0.42698 (6)	0.0181 (3)
C15	0.14011 (10)	0.49721 (13)	0.47490 (6)	0.0177 (3)
H15A	0.1002	0.5538	0.4921	0.021*
C16	0.46300 (10)	0.31421 (15)	0.47127 (7)	0.0227 (3)
H16A	0.5310	0.3231	0.4922	0.034*
H16B	0.4590	0.3324	0.4288	0.034*
H16C	0.4406	0.2276	0.4760	0.034*
C17	0.16178 (11)	0.17238 (15)	0.32512 (7)	0.0249 (3)
H17A	0.1189	0.1338	0.2908	0.037*
H17B	0.1877	0.1065	0.3540	0.037*
H17C	0.2162	0.2155	0.3118	0.037*
C18	−0.04143 (12)	0.49413 (19)	0.35457 (8)	0.0338 (4)
H18A	−0.1129	0.5017	0.3482	0.051*
H18B	−0.0228	0.4500	0.3207	0.051*
H18C	−0.0120	0.5787	0.3582	0.051*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0160 (5)	0.0295 (6)	0.0228 (5)	0.0039 (4)	−0.0025 (4)	−0.0032 (4)
O2	0.0130 (4)	0.0288 (6)	0.0253 (5)	0.0038 (4)	0.0019 (4)	−0.0025 (4)
O3	0.0197 (5)	0.0270 (5)	0.0244 (5)	−0.0026 (4)	0.0043 (4)	−0.0092 (4)
O4	0.0132 (4)	0.0286 (6)	0.0263 (5)	−0.0023 (4)	0.0012 (4)	−0.0023 (4)
N1	0.0168 (6)	0.0277 (7)	0.0220 (6)	0.0043 (5)	0.0001 (4)	−0.0045 (5)
C1	0.0151 (6)	0.0176 (6)	0.0160 (6)	−0.0007 (5)	0.0009 (4)	0.0023 (5)
C2	0.0126 (5)	0.0202 (6)	0.0164 (6)	−0.0009 (5)	−0.0009 (4)	0.0036 (5)
C3	0.0157 (6)	0.0201 (6)	0.0151 (6)	−0.0029 (5)	−0.0004 (4)	0.0012 (5)
C4	0.0162 (6)	0.0200 (6)	0.0152 (6)	−0.0005 (5)	0.0035 (4)	0.0034 (5)
C5	0.0116 (5)	0.0230 (7)	0.0195 (6)	−0.0014 (5)	0.0003 (4)	0.0023 (5)
C6	0.0151 (6)	0.0206 (6)	0.0176 (6)	−0.0038 (5)	−0.0002 (4)	0.0006 (5)
C7	0.0160 (6)	0.0194 (6)	0.0170 (6)	−0.0002 (5)	0.0012 (5)	0.0022 (5)
C8	0.0151 (6)	0.0225 (7)	0.0203 (6)	0.0011 (5)	−0.0024 (5)	−0.0015 (5)

C9	0.0137 (5)	0.0220 (7)	0.0170 (6)	−0.0018 (5)	0.0019 (4)	0.0029 (5)
C10	0.0145 (6)	0.0184 (6)	0.0161 (6)	−0.0008 (5)	0.0034 (4)	0.0018 (5)
C11	0.0135 (5)	0.0202 (6)	0.0186 (6)	−0.0002 (5)	0.0034 (5)	0.0035 (5)
C12	0.0178 (6)	0.0186 (6)	0.0209 (6)	0.0013 (5)	0.0080 (5)	0.0005 (5)
C13	0.0190 (6)	0.0186 (6)	0.0174 (6)	−0.0038 (5)	0.0052 (5)	−0.0006 (5)
C14	0.0129 (6)	0.0208 (7)	0.0206 (6)	−0.0023 (5)	0.0033 (5)	0.0008 (5)
C15	0.0148 (6)	0.0187 (6)	0.0200 (6)	0.0003 (5)	0.0045 (5)	−0.0005 (5)
C16	0.0166 (6)	0.0260 (7)	0.0265 (7)	0.0058 (5)	0.0071 (5)	0.0040 (6)
C17	0.0281 (7)	0.0221 (7)	0.0260 (7)	−0.0023 (6)	0.0091 (6)	−0.0064 (6)
C18	0.0208 (7)	0.0432 (10)	0.0345 (9)	0.0005 (7)	−0.0023 (6)	0.0071 (7)

Geometric parameters (Å, °)

O1—C7	1.2400 (16)	C8—C9	1.3407 (19)
O2—C11	1.3666 (15)	C8—H8A	0.9300
O2—C16	1.4293 (17)	C9—C10	1.4549 (18)
O3—C13	1.3557 (16)	C9—H9A	0.9300
O3—C17	1.4329 (17)	C10—C15	1.4071 (18)
O4—C14	1.3859 (15)	C10—C11	1.4076 (18)
O4—C18	1.4228 (19)	C11—C12	1.3953 (19)
N1—C4	1.3704 (18)	C12—C13	1.3927 (18)
N1—H1N1	0.862 (19)	C12—H12A	0.9300
N1—H2N1	0.88 (2)	C13—C14	1.4083 (19)
C1—C6	1.4060 (18)	C14—C15	1.3724 (19)
C1—C2	1.4064 (18)	C15—H15A	0.9300
C1—C7	1.4760 (19)	C16—H16A	0.9600
C2—C3	1.3758 (19)	C16—H16B	0.9600
C2—H2A	0.9300	C16—H16C	0.9600
C3—C4	1.4089 (18)	C17—H17A	0.9600
C3—H3A	0.9300	C17—H17B	0.9600
C4—C5	1.4016 (19)	C17—H17C	0.9600
C5—C6	1.378 (2)	C18—H18A	0.9600
C5—H5A	0.9300	C18—H18B	0.9600
C6—H6A	0.9300	C18—H18C	0.9600
C7—C8	1.4718 (19)		
C11—O2—C16	118.06 (11)	C11—C10—C9	121.02 (12)
C13—O3—C17	118.17 (11)	O2—C11—C12	123.00 (12)
C14—O4—C18	114.38 (11)	O2—C11—C10	115.65 (12)
C4—N1—H1N1	117.0 (13)	C12—C11—C10	121.35 (12)
C4—N1—H2N1	118.4 (13)	C13—C12—C11	119.84 (12)
H1N1—N1—H2N1	115.1 (18)	C13—C12—H12A	120.1
C6—C1—C2	117.47 (12)	C11—C12—H12A	120.1
C6—C1—C7	123.07 (12)	O3—C13—C12	124.72 (12)
C2—C1—C7	119.46 (11)	O3—C13—C14	115.69 (12)
C3—C2—C1	121.63 (12)	C12—C13—C14	119.59 (12)
C3—C2—H2A	119.2	C15—C14—O4	119.17 (12)
C1—C2—H2A	119.2	C15—C14—C13	119.88 (12)
C2—C3—C4	120.24 (12)	O4—C14—C13	120.72 (12)
C2—C3—H3A	119.9	C14—C15—C10	122.04 (12)

C4—C3—H3A	119.9	C14—C15—H15A	119.0
N1—C4—C5	120.65 (12)	C10—C15—H15A	119.0
N1—C4—C3	120.86 (12)	O2—C16—H16A	109.5
C5—C4—C3	118.44 (12)	O2—C16—H16B	109.5
C6—C5—C4	120.69 (12)	H16A—C16—H16B	109.5
C6—C5—H5A	119.7	O2—C16—H16C	109.5
C4—C5—H5A	119.7	H16A—C16—H16C	109.5
C5—C6—C1	121.26 (12)	H16B—C16—H16C	109.5
C5—C6—H6A	119.4	O3—C17—H17A	109.5
C1—C6—H6A	119.4	O3—C17—H17B	109.5
O1—C7—C8	120.93 (12)	H17A—C17—H17B	109.5
O1—C7—C1	120.63 (12)	O3—C17—H17C	109.5
C8—C7—C1	118.43 (11)	H17A—C17—H17C	109.5
C9—C8—C7	122.45 (12)	H17B—C17—H17C	109.5
C9—C8—H8A	118.8	O4—C18—H18A	109.5
C7—C8—H8A	118.8	O4—C18—H18B	109.5
C8—C9—C10	125.73 (12)	H18A—C18—H18B	109.5
C8—C9—H9A	117.1	O4—C18—H18C	109.5
C10—C9—H9A	117.1	H18A—C18—H18C	109.5
C15—C10—C11	117.31 (12)	H18B—C18—H18C	109.5
C15—C10—C9	121.64 (12)		
C6—C1—C2—C3	−2.81 (19)	C15—C10—C11—O2	179.96 (11)
C7—C1—C2—C3	176.14 (12)	C9—C10—C11—O2	−1.95 (18)
C1—C2—C3—C4	−1.5 (2)	C15—C10—C11—C12	0.21 (19)
C2—C3—C4—N1	−172.31 (13)	C9—C10—C11—C12	178.30 (12)
C2—C3—C4—C5	5.09 (19)	O2—C11—C12—C13	−179.69 (12)
N1—C4—C5—C6	173.09 (13)	C10—C11—C12—C13	0.0 (2)
C3—C4—C5—C6	−4.31 (19)	C17—O3—C13—C12	1.5 (2)
C4—C5—C6—C1	−0.1 (2)	C17—O3—C13—C14	−178.72 (12)
C2—C1—C6—C5	3.6 (2)	C11—C12—C13—O3	179.60 (13)
C7—C1—C6—C5	−175.30 (13)	C11—C12—C13—C14	−0.2 (2)
C6—C1—C7—O1	−170.62 (13)	C18—O4—C14—C15	104.12 (16)
C2—C1—C7—O1	10.5 (2)	C18—O4—C14—C13	−81.36 (17)
C6—C1—C7—C8	10.37 (19)	O3—C13—C14—C15	−179.72 (12)
C2—C1—C7—C8	−168.52 (12)	C12—C13—C14—C15	0.1 (2)
O1—C7—C8—C9	−1.0 (2)	O3—C13—C14—O4	5.79 (19)
C1—C7—C8—C9	177.98 (13)	C12—C13—C14—O4	−174.38 (12)
C7—C8—C9—C10	177.99 (13)	O4—C14—C15—C10	174.73 (12)
C8—C9—C10—C15	−7.9 (2)	C13—C14—C15—C10	0.2 (2)
C8—C9—C10—C11	174.10 (14)	C11—C10—C15—C14	−0.3 (2)
C16—O2—C11—C12	−1.45 (19)	C9—C10—C15—C14	−178.39 (13)
C16—O2—C11—C10	178.80 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O1^{i}	0.86 (2)	2.12 (2)	2.9692 (16)	170.4 (17)
N1—H2N1···O1^{ii}	0.88 (2)	2.21 (2)	3.0176 (17)	153.4 (19)

Symmetry codes: (i) $x-1/2, y+1/2, z$; (ii) $-x+1/2, y+1/2, -z+3/2$.

Fig. 1

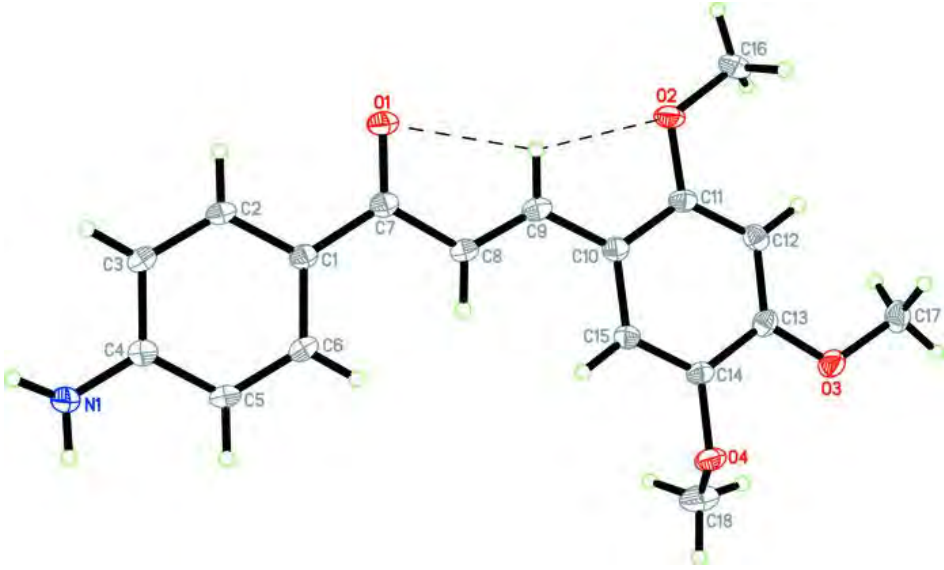
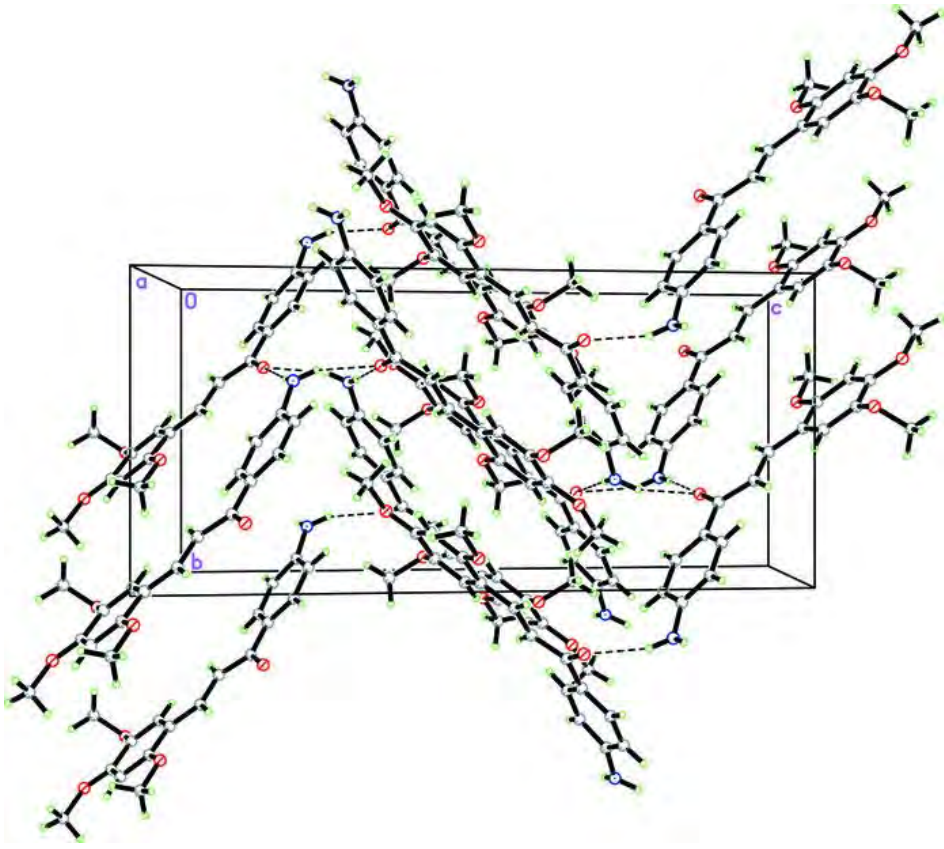


Fig. 2

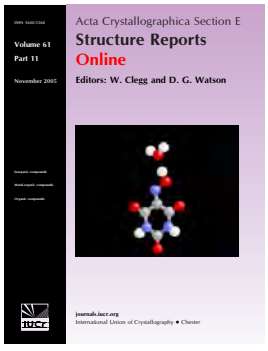


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-3-(4-Ethoxyphenyl)-1-(2-hydroxyphenyl)prop-2-en-1-one

Jirapa Horkaew, Suchada Chantrapromma, Nisakorn Saewan and Hoong-Kun Fun

Acta Cryst. (2010). E**66**, o2346–o2347



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-3-(4-Ethoxyphenyl)-1-(2-hydroxyphenyl)prop-2-en-1-one

Jirapa Horkaew,[a] Suchada Chantrapromma,[a]*‡ Nisakorn Saewan[b] and Hoong-Kun Fun[c]§

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]School of Cosmetic Science, Mae Fah Luang University, Muang, Chiang Rai 57100, Thailand, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

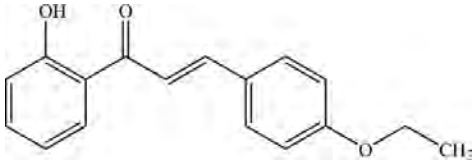
Received 28 July 2010; accepted 12 August 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.041; wR factor = 0.124; data-to-parameter ratio = 31.4.

In the title compound, $C_{17}H_{16}O_3$, the carbonyl group is in an *s-cis* configuration with respect to the olefinic double bond. The dihedral angle between the two benzene rings is 2.85 (3)°. The prop-2-en-1-one bridge makes dihedral angles of 4.77 (4) and 4.15 (4)°, respectively, with the 2-hydroxyphenyl and 4-ethoxyphenyl rings. The ethoxy group is coplanar with the attached phenyl ring [C_{ar}—O—C—C = 179.72 (5)°]. An intramolecular O—H···O hydrogen bond generates an S(6) ring motif. In the crystal structure, molecules are stacked in an antiparallel manner to form columns along the b axis. The columnar structure is stabilized by C—H···π interactions involving the 2-hydroxyphenyl ring.

Related literature

For background on the applications of chalcones, see: Jun *et al.* (2007); Nowakowska (2007); Patil & Dharmaprakash (2008); Saydam *et al.* (2003); Svetlichny *et al.* (2007); Tewtrakul *et al.* (2003). For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Fun *et al.* (2008); Patil *et al.* (2007). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{17}H_{16}O_3$
M_r = 268.30
Triclinic, $P\overline{1}$
a = 6.8305 (2) Å
b = 6.8790 (2) Å
c = 14.8188 (3) Å
α = 88.533 (1)°
β = 80.380 (1)°
γ = 77.469 (1)°
V = 670.11 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.60 × 0.38 × 0.36 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.948, T_{max} = 0.969
24772 measured reflections
5849 independent reflections
5179 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.124
S = 1.04
5848 reflections
186 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1*O2*···O1	0.93 (2)	1.66 (2)	2.5113 (7)	151 (1)
C16—H16*A*···*Cg*1^{i}	0.97	2.70	3.5762 (7)	151
C16—H16*B*···*Cg*1^{ii}	0.97	2.66	3.5339 (7)	151

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $-x+2, -y+2, -z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for the research grant (grant No. RSA 5280033) and the Prince of Songkla University for financial support. The authors also thank the Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012. Mr Teerasak Anantapong, Department of Biotechnology, Faculty of Agro-Industry, Prince of Songkla University is acknowledged for the bacterial assay.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5143).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

‡ Thomson Reuters ResearcherID: A-5085-2009.
§ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Fun, H.-K., Patil, P. S., Dharmaprakash, S. M. & Chantrapromma, S. (2008). *Acta Cryst.* E**64**, o1540–o1541.
Jun, N., Hong, G. & Jun, K. (2007). *Bioorg. Med. Chem.* **15**, 2396–2402.
Nowakowska, Z. (2007). *Eur. J. Med. Chem.* **42**, 125–137.
Patil, P. S. & Dharmaprakash, S. M. (2008). *Mater. Lett.* **62**, 451–453.
Patil, P. S., Fun, H.-K., Chantrapromma, S. & Dharmaprakash, S. M. (2007). *Acta Cryst.* E**63**, o2497–o2498.
Saydam, G., Aydin, H. H., Sahin, F., Kucukoglu, O., Erciyas, E., Terzioglu, E., Buyukkececi, F. & Omay, S. B. (2003). *Leuk. Res.* **27**, 57–64.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Svetlichny, V. Y., Merola, F., Dobretsov, G. E., Gularyan, S. K. & Syrejshchikova, T. I. (2007). *Chem. Phys. Lipids*, **145**, 13–26.
Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol.* **25**, 503–508.

supplementary materials

Acta Cryst. (2010). E**66**, o2346-o2347 [**doi:10.1107/S1600536810032514**]

(*E*)-3-(4-Ethoxyphenyl)-1-(2-hydroxyphenyl)prop-2-en-1-one

J. Horkaew, S. Chantrapromma, N. Saewan and H.-K. Fun

Comment

Chalcones is an interesting class of compounds which have been reported to posses various useful properties such as non-linear optical (NLO) (Patil & Dharmaprakash, 2008) and fluorescent properties (Svetlichny *et al.*, 2007). Synthetic and naturally occurring chalcones have been found to exhibit many useful biological activities, including anti-inflammatory, antileishmanial, antimicrobial, antioxidant (Nowakowska, 2007; Saydam *et al.*, 2003), HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003) and tyrosinase inhibitory (Jun *et al.*, 2007) activities. Our on going research on NLO properties and bioactivities of the synthetic chalcones led us to synthesize the title chalcone in order to study its NLO properties, antibacterial and tyrosinase inhibitory activities. The results show that the title compound, (I), crystallized in centrosymmetric $P\overline{1}$ space group which prohibits the second order NLO properties. Our biological testing found that (I) was inactive against the tested bacteria which are Gram-positive bacteria *i.e. Bacillus subtilis*, *Enterococcus faecalis*, *Staphylococcus aureus*, Methicillin-Resistant *Staphylococcus aureus* and Vancomycin-Resistant *Enterococcus faecalis* and Gram-negative bacteria *i.e. Pseudomonas aeruginosa*, *Salmonella typhi* and *Shigella sonnei*. Nevertheless (I) shows moderate tyrosinase inhibitory activity which will be reported elsewhere with some other chalcones. Herein the crystal structure of (I) is reported.

The title molecule (Fig. 1) exists in an *E* configuration with respect to the C8═C9 ethenyl bond [1.3475 (8) Å]; the C7–C8–C9–C10 torsion angle is -178.73 (6)°. The molecule is almost planar with the dihedral angle between the 2-hydroxyphenyl and 4-ethoxyphenyl rings being 2.85 (3)°. The substituted ethoxy group is coplanar with the attached phenyl ring with the torsion angle C13–O3–C16–C17 = 179.72 (5)°. The prop-2-en-1-one unit (C7–C9/O1) is planar [*r.m.s.* deviation 0.0098 (1) Å] and the torsion angle O1–C7–C8–C9 being 3.15 (10)°. This bridge makes dihedral angles of 4.77 (4) and 4.15 (4)° with the 2-hydroxyphenyl and 4-ethoxyphenyl rings, respectively. Intramolecular O2—H1O2···O1 hydrogen bond generates an S(6) ring motif (Fig. 1) (Bernstein *et al.*, 1995) which helps to stabilize the planarity of the chalcone skeleton. The bond distances are of normal values (Allen *et al.*, 1987) and are comparable with those observed in related structures (Fun *et al.*, 2008; Patil *et al.*, 2007).

In the crystal packing, the molecules are stacked in an antiparallel manner into columns along the *b* axis (Fig. 2). This arrangement is stabilized by C—H···π interactions (Table 1) involving the hydroxy phenyl ring. In addition C···O short contacts [3.2894 (8)–3.4003 (9) Å] were also observed.

Experimental

The title compound was synthesized by the condensation of 4-ethoxycarboxaldehyde (0.30 g, 2 mmol) with 2-hydroxyacetophenone (0.24 ml, 2 mmol) in ethanol (40 ml) in the presence of 30% aqueous NaOH (5 ml) at room temperature. After stirring for 3 h, 20% H_2SO_4(aq) (5 ml) was added dropwise into the solution. After the reaction mixture was kept at room temperature for a day, a yellow solid appeared and was then collected by filtration, washed with acetone and dried in air. Yellow block-shaped single crystals of the title compound suitable for X-ray structure determination were recrystalized from acetone by slow evaporation of the solvent at room temperature after several days (m.p. 387–388 K).

Refinement

Hydroxyl H atom was located in a difference map and refined isotropically. The remaining H atoms were placed in calculated positions, with C–H = 0.93 Å, U_{iso} = 1.2U_{eq}(C) for aromatic and CH, C–H = 0.96 Å, U_{iso} = 1.2U_{eq}(C) for CH_2, and C–H = 0.97 Å, U_{iso} = 1.5U_{eq}(C) for CH_3 atoms. A rotating group model was used for the methyl groups.

Figures

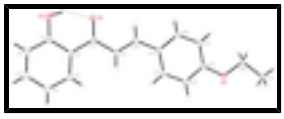


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. Intramolecular O—H···O hydrogen bond is shown as a dashed line.

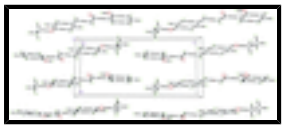


Fig. 2. The crystal packing of the title compound viewed along the *a* axis, showing antiparallel stacked columns running along the *b* axis.

(*E*)-3-(4-Ethoxyphenyl)-1-(2-hydroxyphenyl)prop-2-en-1-one

Crystal data

$C_{17}H_{16}O_3$	Z = 2
M_r = 268.30	F(000) = 284
Triclinic, $P\overline{1}$	D_x = 1.330 Mg m^{-3}
Hall symbol: -P 1	Melting point = 387–388 K
a = 6.8305 (2) Å	Mo $K\alpha$ radiation, λ = 0.71073 Å
b = 6.8790 (2) Å	Cell parameters from 5849 reflections
c = 14.8188 (3) Å	θ = 1.4–35.0°
α = 88.533 (1)°	μ = 0.09 mm^{-1}
β = 80.380 (1)°	T = 100 K
γ = 77.469 (1)°	Block, yellow
V = 670.11 (3) Å^3	0.60 × 0.38 × 0.36 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	5849 independent reflections
Radiation source: sealed tube	5179 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.020
φ and ω scans	θ_{max} = 35.0°, θ_{min} = 1.4°
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	h = −11→11
T_{min} = 0.948, T_{max} = 0.969	k = −10→11
24772 measured reflections	l = −23→23

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.124$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0744P)^2 + 0.1118P]$ where $P = (F_o^2 + 2F_c^2)/3$
5848 reflections	$(\Delta/\sigma)_{max} = 0.001$
186 parameters	$\Delta\rho_{max} = 0.39$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.44$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.46060 (7)	0.63044 (8)	0.13054 (3)	0.01858 (10)
O2	1.59166 (7)	0.61854 (8)	0.27959 (4)	0.01890 (11)
H1O2	1.589 (2)	0.614 (2)	0.2173 (11)	0.052 (4)*
O3	0.61756 (7)	0.84310 (8)	−0.23045 (3)	0.01749 (10)
C1	1.39580 (9)	0.65747 (9)	0.32040 (4)	0.01353 (11)
C2	1.35615 (10)	0.66707 (10)	0.41620 (4)	0.01724 (12)
H2A	1.4636	0.6474	0.4490	0.021*
C3	1.15843 (11)	0.70553 (11)	0.46221 (5)	0.02141 (13)
H3A	1.1333	0.7123	0.5258	0.026*
C4	0.99566 (11)	0.73437 (12)	0.41364 (5)	0.02221 (14)
H4A	0.8626	0.7594	0.4448	0.027*
C5	1.03359 (10)	0.72550 (10)	0.31877 (4)	0.01683 (12)
H5A	0.9248	0.7447	0.2869	0.020*
C6	1.23305 (9)	0.68816 (9)	0.26988 (4)	0.01237 (10)
C7	1.27920 (9)	0.67709 (9)	0.16865 (4)	0.01267 (10)

C8	1.11547 (9)	0.72198 (9)	0.11403 (4)	0.01350 (11)
H8A	0.9811	0.7630	0.1425	0.016*
C9	1.16056 (9)	0.70358 (9)	0.02217 (4)	0.01346 (11)
H9A	1.2972	0.6595	−0.0024	0.016*
C10	1.01927 (9)	0.74506 (9)	−0.04275 (4)	0.01229 (10)
C11	0.80665 (9)	0.79158 (9)	−0.01582 (4)	0.01357 (11)
H11A	0.7517	0.7999	0.0461	0.016*
C12	0.67867 (9)	0.82501 (9)	−0.08002 (4)	0.01398 (11)
H12A	0.5386	0.8566	−0.0611	0.017*
C13	0.75873 (9)	0.81164 (9)	−0.17369 (4)	0.01315 (10)
C14	0.96900 (9)	0.76806 (10)	−0.20234 (4)	0.01477 (11)
H14A	1.0233	0.7606	−0.2643	0.018*
C15	1.09656 (9)	0.73581 (9)	−0.13654 (4)	0.01429 (11)
H15A	1.2367	0.7074	−0.1555	0.017*
C16	0.68663 (9)	0.83005 (9)	−0.32736 (4)	0.01484 (11)
H16A	0.7685	0.6985	−0.3445	0.018*
H16B	0.7680	0.9278	−0.3465	0.018*
C17	0.49767 (11)	0.87050 (11)	−0.37121 (5)	0.01895 (12)
H17A	0.5358	0.8690	−0.4366	0.028*
H17B	0.4155	0.9986	−0.3514	0.028*
H17C	0.4217	0.7696	−0.3536	0.028*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.01206 (19)	0.0280 (3)	0.0151 (2)	−0.00354 (17)	−0.00144 (15)	−0.00028 (17)
O2	0.01199 (19)	0.0265 (2)	0.0186 (2)	−0.00291 (17)	−0.00501 (16)	−0.00171 (18)
O3	0.01373 (19)	0.0271 (3)	0.01164 (19)	−0.00273 (17)	−0.00437 (14)	0.00013 (16)
C1	0.0135 (2)	0.0129 (2)	0.0151 (2)	−0.00301 (18)	−0.00482 (18)	0.00005 (18)
C2	0.0209 (3)	0.0179 (3)	0.0145 (2)	−0.0041 (2)	−0.0076 (2)	0.00091 (19)
C3	0.0244 (3)	0.0268 (3)	0.0130 (2)	−0.0054 (2)	−0.0034 (2)	0.0009 (2)
C4	0.0174 (3)	0.0329 (4)	0.0149 (3)	−0.0044 (2)	−0.0001 (2)	0.0007 (2)
C5	0.0130 (2)	0.0228 (3)	0.0145 (2)	−0.0035 (2)	−0.00232 (19)	0.0006 (2)
C6	0.0120 (2)	0.0133 (2)	0.0122 (2)	−0.00281 (17)	−0.00315 (17)	0.00025 (17)
C7	0.0127 (2)	0.0131 (2)	0.0129 (2)	−0.00335 (17)	−0.00318 (17)	0.00039 (17)
C8	0.0128 (2)	0.0150 (2)	0.0130 (2)	−0.00247 (18)	−0.00374 (18)	−0.00015 (18)
C9	0.0138 (2)	0.0146 (2)	0.0129 (2)	−0.00401 (18)	−0.00363 (17)	0.00107 (18)
C10	0.0129 (2)	0.0128 (2)	0.0116 (2)	−0.00302 (17)	−0.00291 (17)	0.00046 (17)
C11	0.0136 (2)	0.0159 (2)	0.0113 (2)	−0.00358 (18)	−0.00181 (17)	0.00037 (17)
C12	0.0124 (2)	0.0167 (2)	0.0125 (2)	−0.00295 (18)	−0.00149 (17)	−0.00021 (18)
C13	0.0127 (2)	0.0145 (2)	0.0126 (2)	−0.00236 (18)	−0.00379 (17)	0.00019 (18)
C14	0.0134 (2)	0.0191 (3)	0.0111 (2)	−0.00229 (19)	−0.00181 (17)	−0.00019 (18)
C15	0.0122 (2)	0.0177 (3)	0.0124 (2)	−0.00208 (18)	−0.00191 (17)	−0.00048 (18)
C16	0.0169 (2)	0.0151 (2)	0.0129 (2)	−0.00269 (19)	−0.00430 (18)	0.00006 (18)
C17	0.0206 (3)	0.0204 (3)	0.0173 (3)	−0.0031 (2)	−0.0091 (2)	0.0003 (2)

Geometric parameters (Å, °)

O1—C7	1.2491 (7)	C9—C10	1.4543 (8)

O2—C1	1.3452 (8)	C9—H9A	0.93
O2—H1O2	0.928 (16)	C10—C15	1.3998 (8)
O3—C13	1.3621 (7)	C10—C11	1.4078 (8)
O3—C16	1.4334 (7)	C11—C12	1.3791 (8)
C1—C2	1.4006 (9)	C11—H11A	0.93
C1—C6	1.4174 (8)	C12—C13	1.4019 (8)
C2—C3	1.3803 (10)	C12—H12A	0.93
C2—H2A	0.93	C13—C14	1.3960 (8)
C3—C4	1.3993 (10)	C14—C15	1.3957 (8)
C3—H3A	0.93	C14—H14A	0.93
C4—C5	1.3868 (9)	C15—H15A	0.93
C4—H4A	0.93	C16—C17	1.5102 (9)
C5—C6	1.4053 (8)	C16—H16A	0.97
C5—H5A	0.93	C16—H16B	0.97
C6—C7	1.4808 (8)	C17—H17A	0.96
C7—C8	1.4632 (8)	C17—H17B	0.96
C8—C9	1.3475 (8)	C17—H17C	0.96
C8—H8A	0.93		
C1—O2—H1O2	105.5 (10)	C15—C10—C9	118.97 (5)
C13—O3—C16	118.56 (5)	C11—C10—C9	123.04 (5)
O2—C1—C2	117.49 (5)	C12—C11—C10	120.91 (5)
O2—C1—C6	122.28 (5)	C12—C11—H11A	119.5
C2—C1—C6	120.23 (6)	C10—C11—H11A	119.5
C3—C2—C1	120.32 (6)	C11—C12—C13	120.33 (5)
C3—C2—H2A	119.8	C11—C12—H12A	119.8
C1—C2—H2A	119.8	C13—C12—H12A	119.8
C2—C3—C4	120.34 (6)	O3—C13—C14	125.05 (5)
C2—C3—H3A	119.8	O3—C13—C12	114.98 (5)
C4—C3—H3A	119.8	C14—C13—C12	119.97 (5)
C5—C4—C3	119.71 (6)	C15—C14—C13	119.03 (5)
C5—C4—H4A	120.1	C15—C14—H14A	120.5
C3—C4—H4A	120.1	C13—C14—H14A	120.5
C4—C5—C6	121.34 (6)	C14—C15—C10	121.76 (5)
C4—C5—H5A	119.3	C14—C15—H15A	119.1
C6—C5—H5A	119.3	C10—C15—H15A	119.1
C5—C6—C1	118.06 (5)	O3—C16—C17	106.17 (5)
C5—C6—C7	122.80 (5)	O3—C16—H16A	110.5
C1—C6—C7	119.14 (5)	C17—C16—H16A	110.5
O1—C7—C8	120.46 (5)	O3—C16—H16B	110.5
O1—C7—C6	118.86 (5)	C17—C16—H16B	110.5
C8—C7—C6	120.67 (5)	H16A—C16—H16B	108.7
C9—C8—C7	119.61 (5)	C16—C17—H17A	109.5
C9—C8—H8A	120.2	C16—C17—H17B	109.5
C7—C8—H8A	120.2	H17A—C17—H17B	109.5
C8—C9—C10	127.21 (5)	C16—C17—H17C	109.5
C8—C9—H9A	116.4	H17A—C17—H17C	109.5
C10—C9—H9A	116.4	H17B—C17—H17C	109.5
C15—C10—C11	117.98 (5)		

O2—C1—C2—C3	−179.69 (6)	C7—C8—C9—C10	−178.73 (5)
C6—C1—C2—C3	0.31 (10)	C8—C9—C10—C15	173.80 (6)
C1—C2—C3—C4	0.31 (11)	C8—C9—C10—C11	−7.18 (10)
C2—C3—C4—C5	−0.43 (12)	C15—C10—C11—C12	0.63 (9)
C3—C4—C5—C6	−0.06 (11)	C9—C10—C11—C12	−178.39 (5)
C4—C5—C6—C1	0.65 (10)	C10—C11—C12—C13	0.49 (9)
C4—C5—C6—C7	179.82 (6)	C16—O3—C13—C14	0.40 (9)
O2—C1—C6—C5	179.22 (6)	C16—O3—C13—C12	−179.35 (5)
C2—C1—C6—C5	−0.78 (9)	C11—C12—C13—O3	178.52 (5)
O2—C1—C6—C7	0.03 (9)	C11—C12—C13—C14	−1.24 (9)
C2—C1—C6—C7	−179.97 (5)	O3—C13—C14—C15	−178.90 (6)
C5—C6—C7—O1	−175.37 (6)	C12—C13—C14—C15	0.83 (9)
C1—C6—C7—O1	3.79 (9)	C13—C14—C15—C10	0.32 (10)
C5—C6—C7—C8	5.26 (9)	C11—C10—C15—C14	−1.04 (9)
C1—C6—C7—C8	−175.59 (5)	C9—C10—C15—C14	178.03 (6)
O1—C7—C8—C9	3.15 (9)	C13—O3—C16—C17	179.71 (5)
C6—C7—C8—C9	−177.48 (5)		

Hydrogen-bond geometry (Å, °)

*Cg*1 is the centroid of the O2,C1–C6 ring?

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1O2···O1	0.93 (2)	1.66 (2)	2.5113 (7)	151 (1)
C16—H16A···Cg1^{i}	0.97	2.70	3.5762 (7)	151
C16—H16B···Cg1^{ii}	0.97	2.66	3.5339 (7)	151

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*; (ii) −*x*+2, −*y*+2, −*z*.

Fig. 1

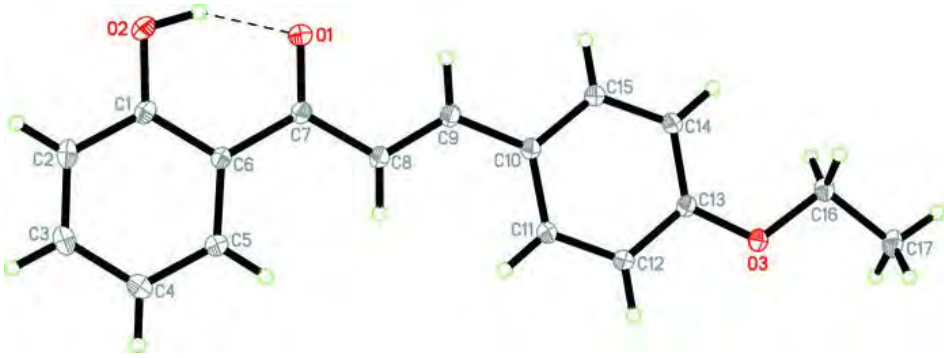
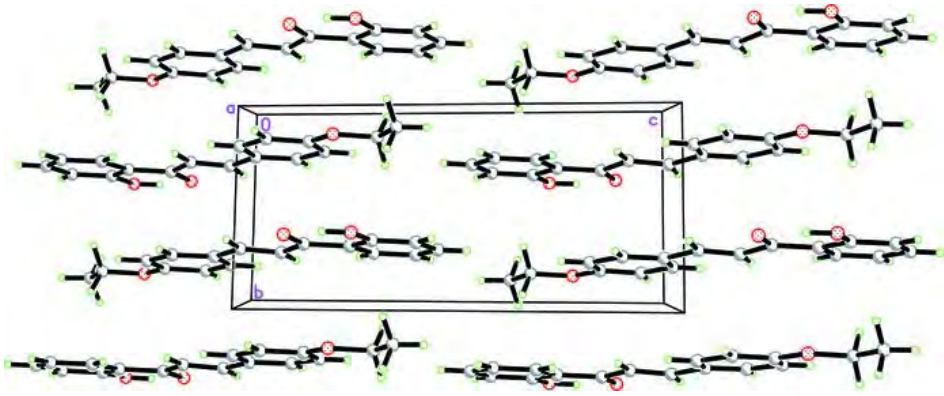


Fig. 2

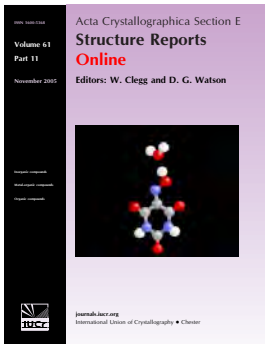


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-1-(2-Furyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun, Thitipone Suwunwong, Suchada Chantrapromma and Chatchanok Karalai

Acta Cryst. (2010). E**66**, o2559–o2560



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(2-Furyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun,[a]*‡ Thitipone Suwunwong,[b] Suchada Chantrapromma[b]§ and Chatchanok Karalai[b]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

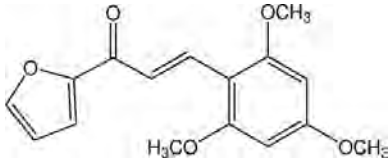
Received 31 August 2010; accepted 6 September 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.113; data-to-parameter ratio = 15.5.

In the title heteroaryl chalcone derivative, $C_{16}H_{16}O_5$, the dihedral angle between the furan and benzene rings is 14.45 (6)°. The three methoxy groups are almost coplanar with their attached benzene ring [C—C—O—C torsion angles = 2.07 (17), −5.04 (17) and 2.85 (16)°]. An intramolecular C—H···O hydrogen bond occurs. In the crystal, adjacent molecules are linked into X-shaped chains along the c axis by weak C—H···O(enone) interactions. These chains are stacked along the b axis. C···O [3.3308 (13)–3.4123 (14) Å] short contacts are also observed.

Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Chantrapromma *et al.* (2009); Suwunwong *et al.* (2009. For background to and applications of chalcones and heteroaryl chalcones, see: Gaber *et al.* (2008); Go *et al.* (2005); Jung *et al.* (2008); Ng *et al.* (2009); Ni *et al.* (2004); Nowakowska (2007); Patil & Dharmaprakash (2008) and Tewtrakul *et al.* (2003). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{16}H_{16}O_5$
M_r = 288.29
Monoclinic, $C2/c$
a = 38.5688 (5) Å
b = 3.93493 (5) Å
c = 18.2638 (3) Å
β = 103.901 (1)°
V = 2690.68 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.41 × 0.15 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.957, T_{max} = 0.990
20657 measured reflections
3941 independent reflections
3077 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.113
S = 1.06
3941 reflections
254 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2^{i}	0.956 (15)	2.496 (15)	3.3512 (14)	148.9 (13)
C6—H6···O5	0.965 (15)	2.260 (14)	2.8197 (15)	116.0 (11)
C14—H14*A*···O4^{ii}	0.975 (15)	2.589 (16)	3.4462 (14)	146.7 (11)
C15—H15*A*···O1^{iii}	0.989 (16)	2.546 (16)	3.4293 (18)	148.6 (12)
C16—H16*A*···O1^{iii}	0.982 (16)	2.575 (16)	3.4120 (16)	143.0 (12)

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+2$; (ii) $-x, -y, -z+1$; (iii) $x, -y, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for a research grant (RSA 5280033) and the Prince of Songkla University for financial support. The authors also thank Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811160. Mr Teerasak Anantapong, Department of Biotechnology, Faculty of Agro-Industry, Prince of Songkla University, is acknowledged for the bacterial assay.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5630).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chantrapromma, S., Suwunwong, T., Karalai, C. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o893–o894.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Gaber, M., El-Daly, S. A., Fayed, T. A. & El-Sayed, Y. S. (2008). *Opt. Laser Technol.* **40**, 528–537.
Go, M.-L., Wu, X. & Liu, X.-L. (2005). *Curr. Med. Chem.* **12**, 483–499.
Jung, Y. J., Son, K. I., Oh, Y. E. & Noh, D. Y. (2008). *Polyhedron*. **27**, 861–867.
Ng, L.-T., Ko, H.-H. & Lu, T.-M. (2009). *Bioorg. Med. Chem.* **17**, 4360–4366.
Ni, L., Meng, C. Q. & Sikorski, J. A. (2004). *Exp. Opin. Ther. Patents*, **14**, 1669–1691.
Nowakowska, Z. (2007). *Eur. J. Med. Chem.* **42**, 125–137.
Patil, P. S. & Dharmaprakash, S. M. (2008). *Mater. Lett.* **62**, 451–453.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suwunwong, T., Chantrapromma, S., Pakdeevanich, P. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o1575–o1576.
Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol.* **25**, 503–508.

supplementary materials

Acta Cryst. (2010). E**66**, o2559-o2560 [**doi:10.1107/S1600536810035762**]

(*E*)-1-(2-Furyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

H.-K. Fun, T. Suwunwong, S. Chantrapromma and C. Karalai

Comment

Chalcone and heteroaryl chalcones are very interesting due to their variety of applications with biological activities. Many of them possess analgesic, anti-inflammatory and antibacterial properties (Go *et al.*, 2005; Ni *et al.*, 2004; Nowakowska, 2007) as well as HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003) and tyrosinase inhibitory (Ng *et al.*, 2009) activities. Moreover synthetic chalcones and heteroaryl chalcones have also been found to exhibit non-linear optical (Patil & Dharmaprakash, 2008), fluorescent (Jung *et al.*, 2008) and laser properties (Gaber *et al.*, 2008) . In continuing our on-going research on anti-bacterial activities and fluorescence properties of chalcones and heteroaryl chalcone derivatives, the title heteroaryl chalcone was synthesized in order to study its antibacterial and fluorescence properties. However our results show that (I) do not possess fluorescence property. In addition our biological testing found that (I) was inactive against the tested bacteria strains which are *Bacillus subtilis*, *Enterococcus faecalis*, *Staphylococcus aureus*, Methicillin-Resistant *Staphylococcus aureus*, Vancomycin-Resistant *Enterococcus faecalis*, *Pseudomonas aeruginosa*, *Salmonella typhi* and *Shigella sonnei*. Herein we report the crystal structure of (I).

The molecule of the title heteroaryl chalcone (Fig. 1) exists in an *E* configuration with respect to the C6═C7 double bond [1.3512 (16) Å] with the C5–C6–C7–C8 torsion angle being -176.44 (12)°. The whole molecule is slightly twisted with the dihedral angle between the furan and benzene rings being 14.45 (6)°. Atoms of the propenone unit (C5, C6, C7 and O1) lie on the same plane [*r.m.s*. 0.0179 (1)]. This plane makes dihedral angles of 11.38 (8) and 9.12 (8)° with furan and phenyl rings, respectively. All the three substituted methoxy groups of 2,4,6-trimethoxyphenyl unit are almost co-planar with the phenyl ring as indicated by torsion angles C14–O3–C9–C10 = 2.07 (17)°, C15–O4–C11–C12 = -5.04 (17)° and C16–O5–C13–C12 = 2.85 (16)°. In the structure, a weak intramolecular C6—H6···O5 interaction generates an S(6) ring motif (Bernstein *et al.*, 1995) (Table 1). The bond lengths have normal values (Allen *et al.*, 1987) and bond lengths and angles are comparable with its related structures (Chantrapromma *et al.*, 2009; Suwunwong *et al.*, 2009).

In the crystal packing, all the three methoxy groups involve in weak intermolecular C—H···O interactions (Table 1). The adjacent molecules are linked into X-shape chains along the *c* axis through the enone unit by weak C—H···O interactions (Fig. 2, Table 1). The adjacent chains are arranged into face-to-face manner (Fig. 3) and stacked along the *b* axis (Fig. 3). The crystal is further stabilized by C···O[3.3308 (13)-3.4123 (14) Å] short contacts.

Experimental

The title compound was prepared by the condensation of the solution of 2-furyl methylketone (2 mmol, 0.22 g) in ethanol (15 ml) and 2,4,6-trimethoxybenzaldehyde (2 mmol, 0.40 g) in ethanol (15 ml) in the presence of 20% NaOH (aq) 5 ml at 278 K for 5 hr. The resulting solid which was obtained was further collected by filtration, washed with distilled water and dried in air. Colorless blocks of (I) were recrystalized from acetone/ethanol (1:1 *v*/*v*) by the slow evaporation of the solvent at room temperature after several days, Mp. 390–391 K.

Refinement

All H atoms were located in difference maps and refined isotropically. The highest residual electron density peak is located at 0.63 Å from C10 and the deepest hole is located at 1.12 Å from C2.

Figures

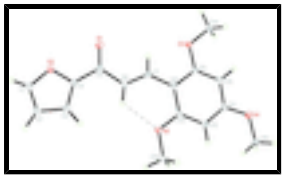


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids. Weak intramolecular interactions are shown as dashed lines.

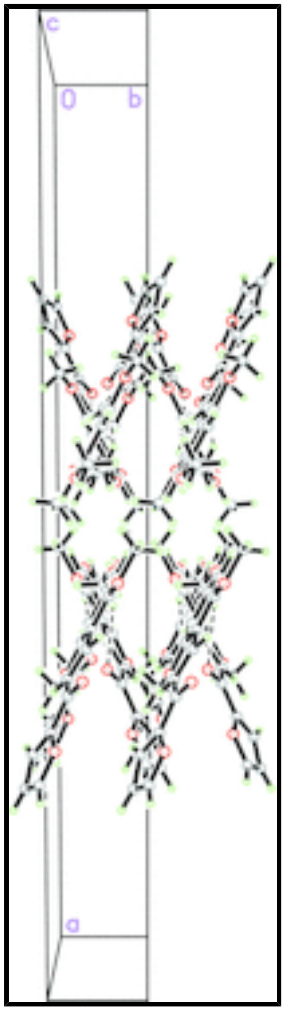


Fig. 2. The crystal packing of (I) viewed along the *c* axis, showing X-chains running along the *c* axis. Weak C—H···O interactions are shown as dashed lines.

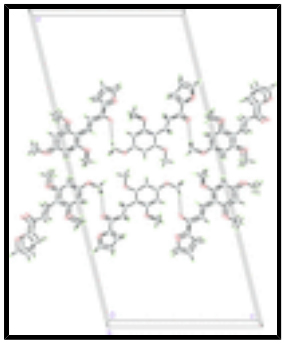


Fig. 3. The crystal packing of (I) viewed along the *b* axis, showing chains stacking along the *b* axis. Weak C—H···O interactions are shown as dashed lines.

(*E*)-1-(2-Furyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{16}H_{16}O_5$	$F(000) = 1216$
$M_r = 288.29$	$D_x = 1.423$ Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point = 390–391 K
Hall symbol: -C 2yc	Mo *K*α radiation, λ = 0.71073 Å
a = 38.5688 (5) Å	Cell parameters from 3941 reflections
b = 3.93493 (5) Å	θ = 1.1–30.0°
c = 18.2638 (3) Å	μ = 0.11 mm^{-1}
β = 103.901 (1)°	*T* = 100 K
V = 2690.68 (6) Å^3	Block, colorless
Z = 8	0.41 × 0.15 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer	3941 independent reflections
Radiation source: sealed tube	3077 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.038$
φ and ω scans	$\theta_{max} = 30.0°$, $\theta_{min} = 1.1°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	*h* = −54→54
$T_{min} = 0.957$, $T_{max} = 0.990$	*k* = −5→5
20657 measured reflections	*l* = −25→25

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.113$	All H-atom parameters refined
S = 1.06	$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 1.4768P]$ where $P = (F_o^2 + 2F_c^2)/3$

3941 reflections	$(\Delta/\sigma)_{max} = 0.001$
254 parameters	$\Delta\rho_{max} = 0.35$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.15077 (2)	0.0988 (3)	0.84899 (5)	0.0215 (2)
O2	0.21727 (2)	0.3478 (2)	0.90839 (4)	0.01663 (19)
O3	0.05690 (2)	−0.2197 (2)	0.65467 (5)	0.01694 (19)
O4	0.04708 (2)	−0.1724 (2)	0.39117 (5)	0.01735 (19)
O5	0.15024 (2)	0.2851 (2)	0.56781 (5)	0.01639 (19)
C1	0.25039 (3)	0.4913 (3)	0.92049 (7)	0.0174 (2)
H1	0.2651 (4)	0.474 (4)	0.9706 (8)	0.018 (4)*
C2	0.25591 (3)	0.6274 (3)	0.85645 (7)	0.0188 (3)
H2	0.2775 (4)	0.741 (4)	0.8513 (9)	0.027 (4)*
C3	0.22403 (3)	0.5659 (3)	0.79963 (7)	0.0174 (2)
H3	0.2194 (4)	0.630 (4)	0.7461 (9)	0.021 (4)*
C4	0.20144 (3)	0.3929 (3)	0.83315 (6)	0.0141 (2)
C5	0.16581 (3)	0.2391 (3)	0.80482 (6)	0.0150 (2)
C6	0.15171 (3)	0.2521 (3)	0.72275 (7)	0.0153 (2)
H6	0.1649 (4)	0.381 (4)	0.6935 (8)	0.019 (4)*
C7	0.12181 (3)	0.0762 (3)	0.69077 (6)	0.0145 (2)
H7	0.1101 (4)	−0.049 (4)	0.7240 (8)	0.020 (4)*
C8	0.10376 (3)	0.0332 (3)	0.61194 (6)	0.0133 (2)
C9	0.06997 (3)	−0.1318 (3)	0.59416 (6)	0.0132 (2)
C10	0.05156 (3)	−0.1931 (3)	0.52043 (7)	0.0150 (2)
H10	0.0282 (4)	−0.310 (4)	0.5082 (9)	0.027 (4)*
C11	0.06682 (3)	−0.0939 (3)	0.46185 (6)	0.0140 (2)
C12	0.09968 (3)	0.0689 (3)	0.47557 (6)	0.0139 (2)
H12	0.1100 (4)	0.127 (4)	0.4359 (8)	0.018 (4)*
C13	0.11766 (3)	0.1321 (3)	0.55034 (6)	0.0131 (2)
C14	0.02326 (3)	−0.3920 (3)	0.63942 (7)	0.0177 (2)
H14A	0.0040 (4)	−0.255 (4)	0.6091 (8)	0.018 (4)*

H14B	0.0253 (4)	−0.612 (4)	0.6129 (8)	0.021 (4)*
H14C	0.0183 (4)	−0.431 (4)	0.6887 (9)	0.023 (4)*
C15	0.06015 (4)	−0.0546 (4)	0.32866 (7)	0.0206 (3)
H15A	0.0833 (4)	−0.158 (4)	0.3269 (9)	0.024 (4)*
H15B	0.0411 (5)	−0.122 (4)	0.2834 (9)	0.032 (4)*
H15C	0.0620 (4)	0.196 (4)	0.3296 (9)	0.024 (4)*
C16	0.16511 (3)	0.3953 (3)	0.50694 (7)	0.0158 (2)
H16A	0.1703 (4)	0.200 (4)	0.4778 (9)	0.021 (4)*
H16B	0.1490 (4)	0.555 (4)	0.4762 (8)	0.015 (3)*
H16C	0.1875 (4)	0.511 (4)	0.5307 (8)	0.020 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0194 (4)	0.0314 (5)	0.0145 (4)	−0.0052 (4)	0.0057 (3)	0.0020 (4)
O2	0.0151 (4)	0.0227 (5)	0.0111 (4)	−0.0018 (3)	0.0012 (3)	0.0000 (3)
O3	0.0143 (4)	0.0231 (5)	0.0142 (4)	−0.0047 (3)	0.0049 (3)	0.0000 (3)
O4	0.0162 (4)	0.0239 (5)	0.0111 (4)	−0.0038 (3)	0.0015 (3)	−0.0018 (3)
O5	0.0130 (4)	0.0242 (5)	0.0119 (4)	−0.0051 (3)	0.0029 (3)	0.0009 (3)
C1	0.0152 (5)	0.0204 (6)	0.0157 (6)	−0.0013 (4)	0.0017 (4)	−0.0018 (5)
C2	0.0174 (6)	0.0211 (6)	0.0182 (6)	−0.0039 (5)	0.0049 (5)	−0.0022 (5)
C3	0.0193 (6)	0.0193 (6)	0.0133 (5)	−0.0018 (4)	0.0037 (4)	−0.0014 (5)
C4	0.0152 (5)	0.0171 (6)	0.0094 (5)	0.0016 (4)	0.0016 (4)	−0.0009 (4)
C5	0.0149 (5)	0.0171 (6)	0.0131 (5)	0.0011 (4)	0.0038 (4)	−0.0007 (4)
C6	0.0155 (5)	0.0172 (6)	0.0131 (5)	−0.0002 (4)	0.0033 (4)	0.0004 (4)
C7	0.0146 (5)	0.0169 (6)	0.0122 (5)	0.0018 (4)	0.0038 (4)	−0.0003 (4)
C8	0.0129 (5)	0.0147 (5)	0.0124 (5)	0.0009 (4)	0.0032 (4)	−0.0001 (4)
C9	0.0135 (5)	0.0139 (5)	0.0129 (5)	0.0006 (4)	0.0048 (4)	0.0008 (4)
C10	0.0125 (5)	0.0170 (6)	0.0150 (5)	−0.0007 (4)	0.0027 (4)	−0.0006 (4)
C11	0.0132 (5)	0.0153 (6)	0.0121 (5)	0.0011 (4)	0.0003 (4)	−0.0012 (4)
C12	0.0133 (5)	0.0163 (6)	0.0121 (5)	0.0014 (4)	0.0031 (4)	0.0006 (4)
C13	0.0106 (5)	0.0133 (5)	0.0152 (5)	−0.0001 (4)	0.0028 (4)	0.0002 (4)
C14	0.0142 (5)	0.0197 (6)	0.0199 (6)	−0.0029 (4)	0.0058 (5)	0.0018 (5)
C15	0.0232 (6)	0.0265 (7)	0.0117 (5)	−0.0043 (5)	0.0036 (5)	0.0000 (5)
C16	0.0143 (5)	0.0205 (6)	0.0133 (5)	−0.0023 (4)	0.0043 (4)	0.0024 (5)

Geometric parameters (Å, °)

O1—C5	1.2315 (14)	C7—C8	1.4507 (16)
O2—C1	1.3652 (14)	C7—H7	0.974 (15)
O2—C4	1.3745 (13)	C8—C13	1.4123 (15)
O3—C9	1.3648 (13)	C8—C9	1.4220 (15)
O3—C14	1.4307 (14)	C9—C10	1.3844 (16)
O4—C11	1.3673 (14)	C10—C11	1.3949 (16)
O4—C15	1.4317 (15)	C10—H10	0.988 (16)
O5—C13	1.3602 (13)	C11—C12	1.3883 (16)
O5—C16	1.4352 (14)	C12—C13	1.3974 (16)
C1—C2	1.3492 (17)	C12—H12	0.936 (15)
C1—H1	0.956 (15)	C14—H14A	0.976 (15)

C2—C3	1.4264 (17)	C14—H14B	1.003 (16)
C2—H2	0.970 (16)	C14—H14C	0.977 (16)
C3—C4	1.3617 (17)	C15—H15A	0.989 (16)
C3—H3	0.984 (15)	C15—H15B	1.001 (17)
C4—C5	1.4766 (16)	C15—H15C	0.990 (17)
C5—C6	1.4675 (16)	C16—H16A	0.982 (16)
C6—C7	1.3512 (16)	C16—H16B	0.963 (15)
C6—H6	0.967 (15)	C16—H16C	0.982 (16)
C1—O2—C4	106.39 (9)	C9—C10—C11	119.01 (10)
C9—O3—C14	117.18 (9)	C9—C10—H10	121.8 (9)
C11—O4—C15	117.18 (9)	C11—C10—H10	119.2 (9)
C13—O5—C16	118.08 (9)	O4—C11—C12	123.50 (10)
C2—C1—O2	111.12 (10)	O4—C11—C10	114.76 (10)
C2—C1—H1	132.6 (9)	C12—C11—C10	121.74 (10)
O2—C1—H1	116.2 (9)	C11—C12—C13	118.39 (10)
C1—C2—C3	105.98 (11)	C11—C12—H12	120.9 (9)
C1—C2—H2	125.8 (10)	C13—C12—H12	120.7 (9)
C3—C2—H2	128.2 (10)	O5—C13—C12	121.43 (10)
C4—C3—C2	106.90 (11)	O5—C13—C8	116.18 (10)
C4—C3—H3	126.4 (9)	C12—C13—C8	122.37 (10)
C2—C3—H3	126.7 (9)	O3—C14—H14A	112.3 (9)
C3—C4—O2	109.60 (10)	O3—C14—H14B	109.3 (8)
C3—C4—C5	133.59 (11)	H14A—C14—H14B	109.8 (12)
O2—C4—C5	116.74 (10)	O3—C14—H14C	105.3 (9)
O1—C5—C6	124.57 (11)	H14A—C14—H14C	108.5 (12)
O1—C5—C4	119.95 (10)	H14B—C14—H14C	111.5 (13)
C6—C5—C4	115.40 (10)	O4—C15—H15A	112.7 (9)
C7—C6—C5	119.48 (11)	O4—C15—H15B	104.0 (10)
C7—C6—H6	122.6 (9)	H15A—C15—H15B	110.8 (13)
C5—C6—H6	117.9 (9)	O4—C15—H15C	110.3 (9)
C6—C7—C8	130.17 (11)	H15A—C15—H15C	110.6 (13)
C6—C7—H7	117.8 (9)	H15B—C15—H15C	108.2 (14)
C8—C7—H7	112.0 (9)	O5—C16—H16A	110.8 (9)
C13—C8—C9	116.51 (10)	O5—C16—H16B	109.1 (8)
C13—C8—C7	125.09 (10)	H16A—C16—H16B	112.5 (12)
C9—C8—C7	118.36 (10)	O5—C16—H16C	105.8 (8)
O3—C9—C10	122.72 (10)	H16A—C16—H16C	109.3 (12)
O3—C9—C8	115.31 (10)	H16B—C16—H16C	109.2 (13)
C10—C9—C8	121.96 (10)		
C4—O2—C1—C2	0.63 (14)	C7—C8—C9—O3	3.17 (16)
O2—C1—C2—C3	−0.01 (15)	C13—C8—C9—C10	0.06 (17)
C1—C2—C3—C4	−0.63 (14)	C7—C8—C9—C10	−177.77 (11)
C2—C3—C4—O2	1.03 (14)	O3—C9—C10—C11	179.79 (11)
C2—C3—C4—C5	−175.73 (13)	C8—C9—C10—C11	0.80 (18)
C1—O2—C4—C3	−1.03 (13)	C15—O4—C11—C12	−5.04 (17)
C1—O2—C4—C5	176.35 (10)	C15—O4—C11—C10	175.53 (11)
C3—C4—C5—O1	−178.75 (13)	C9—C10—C11—O4	178.49 (10)
O2—C4—C5—O1	4.67 (17)	C9—C10—C11—C12	−0.95 (18)

C3—C4—C5—C6	4.4 (2)	O4—C11—C12—C13	−179.17 (11)
O2—C4—C5—C6	−172.18 (10)	C10—C11—C12—C13	0.22 (18)
O1—C5—C6—C7	−6.08 (19)	C16—O5—C13—C12	2.85 (16)
C4—C5—C6—C7	170.60 (11)	C16—O5—C13—C8	−178.91 (10)
C5—C6—C7—C8	−176.44 (12)	C11—C12—C13—O5	178.81 (10)
C6—C7—C8—C13	10.2 (2)	C11—C12—C13—C8	0.69 (18)
C6—C7—C8—C9	−172.18 (12)	C9—C8—C13—O5	−179.03 (10)
C14—O3—C9—C10	2.07 (17)	C7—C8—C13—O5	−1.37 (17)
C14—O3—C9—C8	−178.87 (10)	C9—C8—C13—C12	−0.82 (17)
C13—C8—C9—O3	−179.00 (10)	C7—C8—C13—C12	176.84 (11)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2^{i}	0.956 (15)	2.496 (15)	3.3512 (14)	148.9 (13)
C6—H6···O5	0.965 (15)	2.260 (14)	2.8197 (15)	116.0 (11)
C14—H14A···O4^{ii}	0.975 (15)	2.589 (16)	3.4462 (14)	146.7 (11)
C15—H15A···O1^{iii}	0.989 (16)	2.546 (16)	3.4293 (18)	148.6 (12)
C16—H16A···O1^{iii}	0.982 (16)	2.575 (16)	3.4120 (16)	143.0 (12)

Symmetry codes: (i) −*x*+1/2, −*y*+1/2, −*z*+2; (ii) −*x*, −*y*, −*z*+1; (iii) *x*, −*y*, *z*−1/2.

Fig. 1

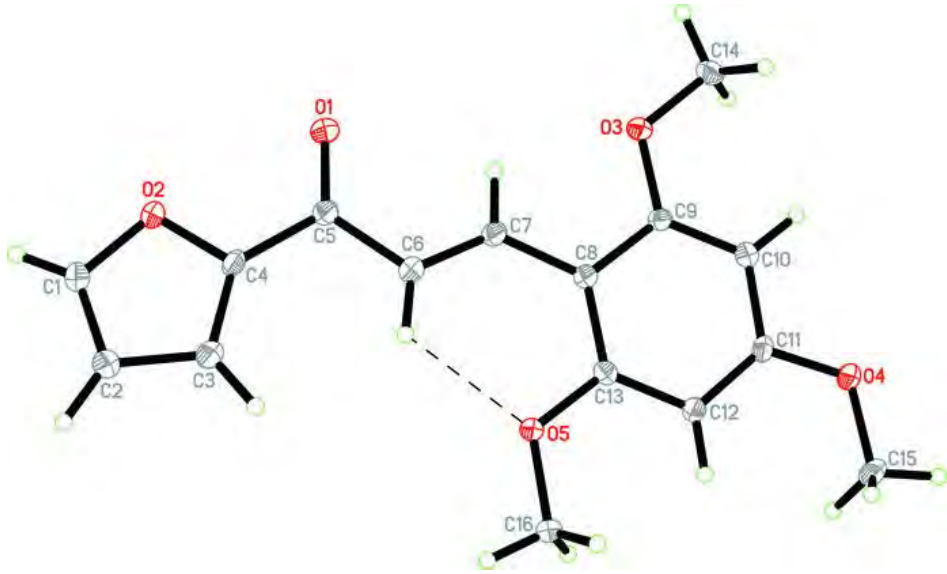


Fig. 2

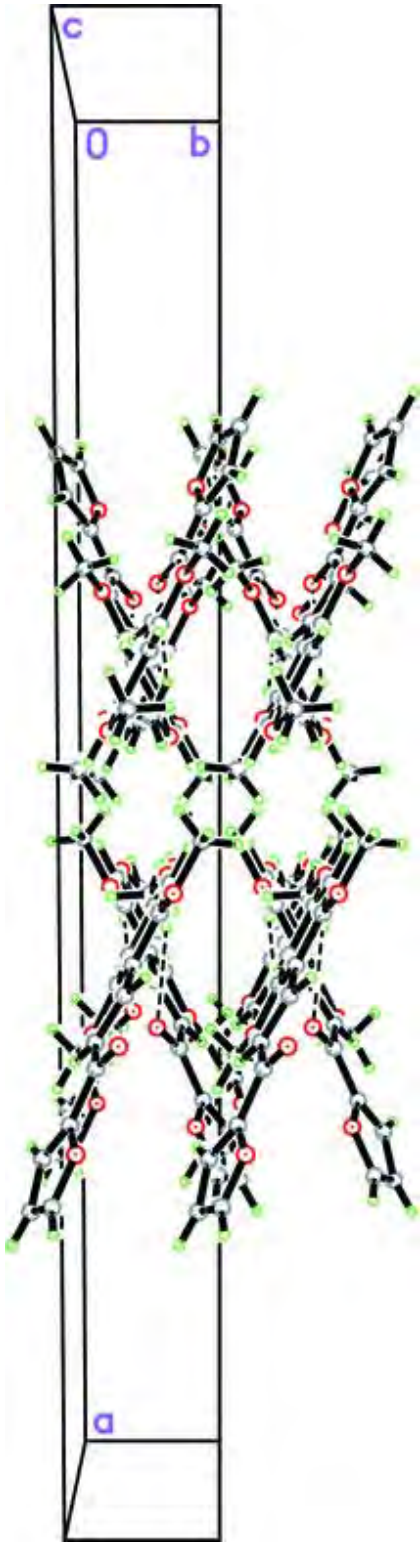
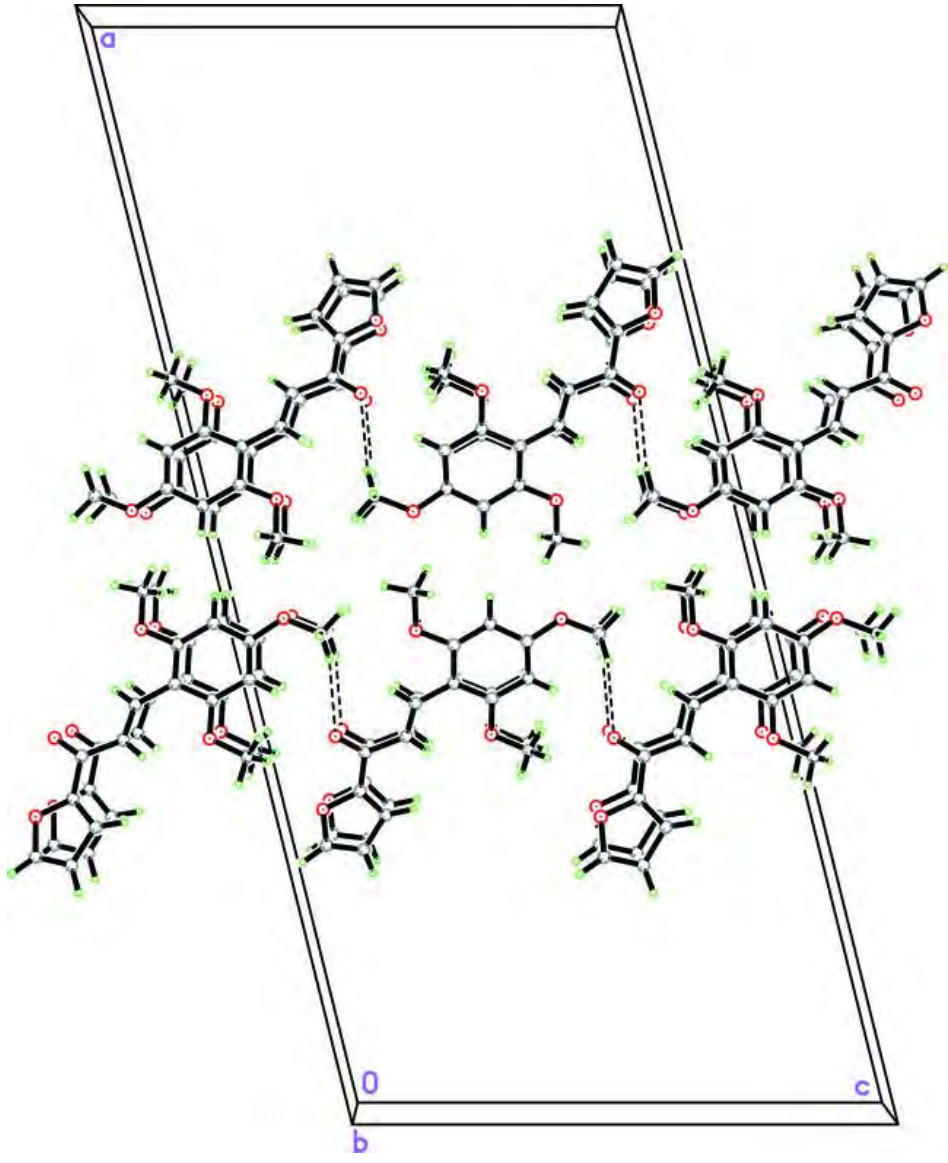


Fig. 3

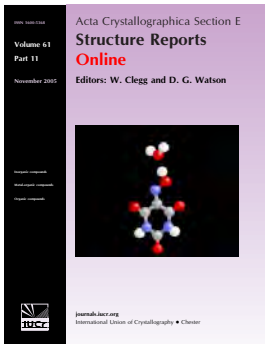


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-1-(4-Aminophenyl)-3-(pyridin-3-yl)prop-2-en-1-one

Suchada Chantrapromma, Thawanrat Kobkeatthawin, Kullapa Chanawanno, Pitikan Wisitsak and Hoong-Kun Fun

Acta Cryst. (2011). E**67**, o1770–o1771



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(4-Aminophenyl)-3-(pyridin-3-yl)-prop-2-en-1-one

Suchada Chantrapromma,[a]*‡ Thawanrat Kobkeatthawin,[a] Kullapa Chanawanno,[a] Pitikan Wisitsak[b] and Hoong-Kun Fun[c]§

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]Excellence Center, Mae Fah Luang University, Thasud, Muang, Chaing Rai 57100, Thailand, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

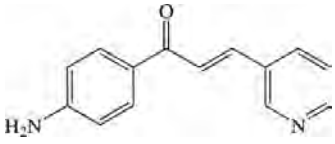
Received 13 June 2011; accepted 16 June 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.048; wR factor = 0.132; data-to-parameter ratio = 15.7.

The title chalcone derivative, $C_{14}H_{12}N_2O$, consists of 4-aminophenyl and pyridine rings bridged by a prop-2-en-1-one unit and exists in a *trans* configuration with respect to the C=C double bond. The molecule is slightly twisted with a dihedral angle of 29.38 (7)° between the benzene and pyridine rings. The prop-2-en-1-one bridge is nearly planar with an r.m.s. deviation of 0.0384 (1) Å and makes dihedral angles of 15.40 (9) and 16.30 (9)°, respectively, with the benzene and pyridine rings. In the crystal, molecules are linked by N—H···N and N—H···O hydrogen bonds into a layer parallel to the *ab* plane. A π–π interaction with a centroid–centroid distance of 3.6946 (10) Å is also observed.

Related literature

For bond-length data, see: Allen *et al.* (1987). For a related structure, see: Horkaew *et al.* (2010). For background to and applications of chalcones, see: Gaber *et al.* (2008); Ávila *et al.* (2008); Mei *et al.* (2001); Ohad *et al.* (2004); Patil *et al.* (2007); Svetlichny *et al.* (2007); Tewtrakul *et al.* (2003); Wu *et al.* (2006); Xu *et al.* (2005). For the stability of the temperature controller used in the data collection, see Cosier & Glazer (1986).



Experimental

Crystal data

$C_{14}H_{12}N_2O$
M_r = 224.26
Orthorhombic, *Pbca*
a = 12.0046 (12) Å
b = 7.9329 (9) Å
c = 22.925 (3) Å
V = 2183.2 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.52 × 0.32 × 0.18 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.956, T_{max} = 0.985
12726 measured reflections
3177 independent reflections
2433 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.132
S = 1.03
3177 reflections
202 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···O1^{i}	0.88 (2)	2.13 (2)	2.9920 (16)	170 (2)
N1—H2*N*1···N2^{ii}	0.93 (2)	2.26 (2)	3.1471 (17)	161.7 (19)

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for a research grant (RSA 5280033) and the Prince of Songkla University for financial support. The authors also thank Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2731).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Ávila, H. P., Smânia, E. de F. A., Delle Monache, F. & Smânia, A. Jr (2008). *Bioorg. Med. Chem.* **16**, 9790–9794.
Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Gaber, M., El-Daly, S. A., Fayed, T. A. & El-Sayed, Y. S. (2008). *J. Opt. Laser Techol.* **40**, 528–537.
Horkaew, J., Chantrapromma, S., Saewan, N. & Fun, H.-K. (2010). *Acta Cryst.* E**66**, o2346–o2347.
Mei, L., Prapon, W. & Mei, L. G. (2001). *J. Med. Chem.* **44**, 4443–4452.
Ohad, N., Ramadan, M., Soliman, K., Snait, T. & Jacob, V. (2004). *J. Photochem.* **65**, 1389–1395.
Patil, P. S., Dharmaprakash, S. M., Ramakrishna, K., Fun, H.-K., Kumar, R. S. S. & Narayana Rao, D. (2007). *J. Cryst. Growth*, **303**, 520–524.

‡ Thomson Reuters ResearcherID: A-5085-2009.
§ Thomson Reuters ResearcherID: A-3561-2009. Additional correspondence author, e-mail: hkfun@usm.my.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Svetlichny, V. Y., Merola, F., Dobretsov, G. E., Gularyan, S. K. & Syrejshchikova, T. I. (2007). *Chem. Phys. Lipids*, **145**, 13–26.
Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol.* **25**, 503–508.
Wu, X., Tiekink, E. R. T., Kostetski, I., Kocherginsky, N., Tan, A. L. C., Khoo, S. B., Wilairat, P. & Go, M.-L. (2006). *Eur. J. Pharm. Sci.* **27**, 175–187.
Xu, Z., Bai, G. & Dong, C. (2005). *Bioorg. Med. Chem.* **13**, 5694–5699.

supplementary materials

Acta Cryst. (2011). E**67**, o1770-o1771 [**doi:10.1107/S1600536811023634**]

(*E*)-1-(4-Aminophenyl)-3-(pyridin-3-yl)prop-2-en-1-one

S. Chantrapromma, T. Kobkeatthawin, K. Chanawanno, P. Wisitsak and H.-K. Fun

Comment

Chalcones are 1,3-diaryl-2-propen-1-ones which can be obtained from both synthetic and natural sources. They have a wide variety of biological activities such as antimalarial (Mei *et al.*, 2001), HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003), antityrosinase (Ohad *et al.*, 2004), antibacterial (Ávila *et al.*, 2008) and antiplasmodial (Wu *et al.*, 2006) properties. Moreover, chalcones have also been studied for non-linear optical (NLO) (Patil *et al.*, 2007) and fluorescent materials (Gaber *et al.*, 2008). These compounds have also been used for sensor, liquid crystal display and fluorescence probe for sensing of DNA or proteins (Svetlichny *et al.*, 2007; Xu *et al.*, 2005). These interesting properties has lead us to synthesize the title compound (I), which contains the amino and pyridine groups in order to study its bioactivity and fluorescent properties. Our results show that (I) was inactive for antibacterial and tyrosinase inhibitory activities. However (I) exhibits weak fluorescence with the maximum emission at 437 nm when was excited at 310 nm. Herein the crystal structure of (I) is reported.

The molecule of the title chalcone derivative (Fig. 1), $C_{14}H_{12}N_2O$, exists in a *E* configuration with respect to the C8═C9 ethenyl bond [1.332 (2) Å] and the torsion angle C7–C8–C9–C10 = -176.57 (13)°. The molecule is twisted with a dihedral angle between the phenyl and pyridine rings being 29.38 (7)°. The prop-2-en-1-one unit (C7-C9/O1) is nearly planar [*r.m.s.* of 0.0384 (1) Å] and the torsion angle O1–C7–C8–C9 being -12.5 (2)°. This middle bridge makes the dihedral angles of 15.40 (9) and 16.30 (9)° with the phenyl and pyridine rings, respectively. The bond distances are of normal values (Allen *et al.*, 1987) and are comparable with the related structure (Horkaew *et al.*, 2010).

In the crystal packing, the molecules are linked by N—H···N and N—H···O hydrogen bonds (Table 1) into sheets parallel to the *ab* plane (Fig. 2). A π–π interaction with a *Cg*1···*Cg*1 distance of 3.6946 (10) Å is observed in the crystal; *Cg*1 is the centroid of the C10–C14/N1 ring. In addition C···C [3.3505 (19) Å; symmetry code 3/2-x, 1/2+y, z and 3.3776 (19) Å: symmetry code 3/2-x, -1/2+y, z], C···O [3.1312 (18) Å; symmetry code 3/2-x, -1/2+y, z] and N···O [2.9920 (16) Å; symmetry code 2-x,1/2+y,3/2-z] short contacts are also observed.

Experimental

The title compound was synthesized by condensation of 4-aminoacetophenone (0.40 g, 3 mmol) with 3-pyridinecarboxaldehyde (0.18 ml, 3 mmol) in ethanol (15 ml) in the presence of 10% NaOH (aq) (5 ml). After stirring for 2 hr at room temperature, the resulting yellow solid was collected by filtration, washed with distilled diethyl ether, dried and purified by repeated recrysallization from acetone. Yellow block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystalized from methanol by the slow evaporation of the solvent at room temperature after several days, Mp. 453-454 K.

Refinement

All H atoms were located in a difference Fourier map and refined isotropically. The highest residual electron density peak is located at 0.74 Å from C8 and the deepest hole is located at 1.35 Å from C14.

Figures

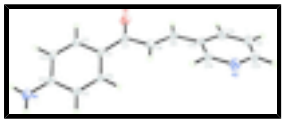


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

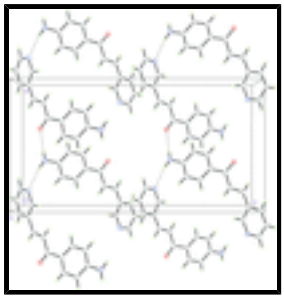


Fig. 2. The crystal packing of the title compound viewed along the *b* axis, Hydrogen bonds were shown as dashed lines.

(*E*)-1-(4-Aminophenyl)-3-(pyridin-3-yl)prop-2-en-1-one

Crystal data

$C_{14}H_{12}N_2O$	D_x = 1.365 Mg m^{-3}
M_r = 224.26	Melting point = 453–454 K
Orthorhombic, *Pbca*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 3177 reflections
a = 12.0046 (12) Å	θ = 2.5–30.0°
b = 7.9329 (9) Å	μ = 0.09 mm^{-1}
c = 22.925 (3) Å	*T* = 100 K
V = 2183.2 (4) Å^3	Block, yellow
Z = 8	0.52 × 0.32 × 0.18 mm
F(000) = 944	

Data collection

Bruker APEX DUO CCD area-detector diffractometer	3177 independent reflections
Radiation source: sealed tube	2433 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.043
φ and ω scans	θ_{max} = 30.0°, θ_{min} = 2.5°
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	*h* = −16→14
T_{min} = 0.956, T_{max} = 0.985	*k* = −11→10
12726 measured reflections	*l* = −32→23

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.132$	All H-atom parameters refined
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.5761P]$ where $P = (F_o^2 + 2F_c^2)/3$
3177 reflections	$(\Delta/\sigma)_{max} = 0.001$
202 parameters	$\Delta\rho_{max} = 0.37$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.87481 (8)	−0.00829 (13)	0.62116 (4)	0.0274 (2)
N1	0.88749 (10)	−0.40307 (16)	0.86417 (5)	0.0262 (3)
H1N1	0.9537 (17)	−0.447 (3)	0.8695 (9)	0.037 (5)*
H2N1	0.8278 (17)	−0.431 (3)	0.8878 (9)	0.043 (5)*
N2	0.34284 (10)	0.06758 (18)	0.57106 (5)	0.0317 (3)
C1	0.82037 (9)	−0.14511 (16)	0.70829 (6)	0.0195 (3)
C2	0.92774 (10)	−0.20798 (16)	0.72041 (6)	0.0210 (3)
H2A	0.9864 (14)	−0.193 (2)	0.6922 (7)	0.023 (4)*
C3	0.95058 (10)	−0.29288 (16)	0.77139 (6)	0.0214 (3)
H3A	1.0254 (14)	−0.340 (2)	0.7786 (7)	0.029 (4)*
C4	0.86642 (10)	−0.31820 (17)	0.81357 (6)	0.0208 (3)
C5	0.75956 (10)	−0.25227 (18)	0.80220 (6)	0.0234 (3)
H5A	0.7024 (17)	−0.265 (3)	0.8321 (9)	0.045 (5)*
C6	0.73797 (10)	−0.16943 (17)	0.75074 (6)	0.0214 (3)
H6A	0.6624 (14)	−0.121 (2)	0.7442 (7)	0.025 (4)*
C7	0.79831 (10)	−0.05897 (16)	0.65285 (6)	0.0205 (3)
C8	0.68037 (10)	−0.03488 (18)	0.63425 (6)	0.0226 (3)
H8A	0.6213 (16)	−0.097 (3)	0.6564 (9)	0.042 (5)*
C9	0.65435 (10)	0.06655 (17)	0.58997 (6)	0.0232 (3)
H9A	0.7163 (14)	0.126 (2)	0.5705 (7)	0.031 (4)*
C10	0.54235 (10)	0.10884 (17)	0.56899 (6)	0.0219 (3)

C11	0.52786 (11)	0.2397 (2)	0.52922 (6)	0.0284 (3)
H11A	0.5968 (15)	0.303 (2)	0.5147 (8)	0.030 (4)*
C12	0.42183 (12)	0.2841 (2)	0.51084 (7)	0.0313 (3)
H12A	0.4105 (16)	0.374 (3)	0.4827 (9)	0.040 (5)*
C13	0.33284 (12)	0.1966 (2)	0.53375 (7)	0.0313 (3)
H13A	0.2553 (16)	0.229 (2)	0.5245 (8)	0.041 (5)*
C14	0.44619 (11)	0.0255 (2)	0.58784 (6)	0.0267 (3)
H14A	0.4500 (15)	−0.070 (3)	0.6168 (8)	0.034 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0178 (4)	0.0354 (6)	0.0292 (5)	−0.0004 (4)	0.0009 (4)	0.0037 (4)
N1	0.0177 (5)	0.0342 (7)	0.0267 (6)	0.0028 (4)	−0.0018 (4)	0.0020 (5)
N2	0.0186 (5)	0.0466 (8)	0.0299 (6)	−0.0020 (5)	−0.0015 (5)	0.0051 (5)
C1	0.0148 (5)	0.0193 (6)	0.0245 (6)	0.0002 (4)	−0.0024 (4)	−0.0034 (5)
C2	0.0143 (5)	0.0216 (6)	0.0271 (6)	−0.0001 (4)	0.0004 (5)	−0.0027 (5)
C3	0.0135 (5)	0.0219 (6)	0.0286 (7)	0.0010 (4)	−0.0020 (4)	−0.0031 (5)
C4	0.0169 (5)	0.0219 (6)	0.0237 (6)	0.0004 (4)	−0.0026 (4)	−0.0043 (5)
C5	0.0156 (5)	0.0296 (7)	0.0248 (6)	0.0029 (5)	0.0011 (5)	−0.0027 (5)
C6	0.0142 (5)	0.0243 (6)	0.0255 (6)	0.0023 (4)	−0.0015 (4)	−0.0051 (5)
C7	0.0158 (5)	0.0210 (6)	0.0246 (6)	0.0004 (4)	−0.0013 (4)	−0.0038 (5)
C8	0.0159 (5)	0.0263 (7)	0.0257 (6)	−0.0015 (5)	−0.0013 (5)	−0.0001 (5)
C9	0.0162 (5)	0.0267 (7)	0.0267 (6)	0.0002 (5)	0.0014 (5)	−0.0009 (5)
C10	0.0186 (6)	0.0265 (7)	0.0205 (6)	0.0019 (4)	−0.0003 (4)	−0.0019 (5)
C11	0.0214 (6)	0.0339 (8)	0.0298 (7)	0.0001 (5)	0.0019 (5)	0.0049 (6)
C12	0.0264 (7)	0.0358 (8)	0.0316 (7)	0.0036 (5)	−0.0038 (6)	0.0071 (6)
C13	0.0210 (6)	0.0440 (9)	0.0290 (7)	0.0039 (6)	−0.0043 (5)	0.0027 (6)
C14	0.0190 (6)	0.0348 (8)	0.0262 (7)	−0.0019 (5)	−0.0015 (5)	0.0039 (6)

Geometric parameters (Å, °)

O1—C7	1.2381 (16)	C5—H5A	0.97 (2)
N1—C4	1.3649 (18)	C6—H6A	0.996 (17)
N1—H1N1	0.88 (2)	C7—C8	1.4909 (17)
N1—H2N1	0.93 (2)	C8—C9	1.332 (2)
N2—C13	1.339 (2)	C8—H8A	1.00 (2)
N2—C14	1.3410 (17)	C9—C10	1.4668 (17)
C1—C6	1.4011 (18)	C9—H9A	0.986 (18)
C1—C2	1.4097 (16)	C10—C11	1.392 (2)
C1—C7	1.4671 (18)	C10—C14	1.3986 (19)
C2—C3	1.3767 (19)	C11—C12	1.3864 (19)
C2—H2A	0.963 (16)	C11—H11A	1.023 (18)
C3—C4	1.4127 (18)	C12—C13	1.378 (2)
C3—H3A	0.988 (17)	C12—H12A	0.97 (2)
C4—C5	1.4097 (17)	C13—H13A	0.99 (2)
C5—C6	1.3749 (19)	C14—H14A	1.01 (2)
C4—N1—H1N1	118.9 (13)	O1—C7—C8	119.67 (12)

C4—N1—H2N1	118.2 (12)	C1—C7—C8	118.61 (11)
H1N1—N1—H2N1	121.7 (19)	C9—C8—C7	121.19 (12)
C13—N2—C14	117.14 (13)	C9—C8—H8A	121.1 (11)
C6—C1—C2	117.38 (12)	C7—C8—H8A	117.8 (11)
C6—C1—C7	122.58 (11)	C8—C9—C10	127.07 (12)
C2—C1—C7	120.04 (11)	C8—C9—H9A	117.1 (10)
C3—C2—C1	121.50 (12)	C10—C9—H9A	115.8 (10)
C3—C2—H2A	119.0 (10)	C11—C10—C14	116.83 (12)
C1—C2—H2A	119.5 (10)	C11—C10—C9	119.98 (12)
C2—C3—C4	120.55 (11)	C14—C10—C9	123.18 (12)
C2—C3—H3A	120.7 (10)	C12—C11—C10	120.21 (13)
C4—C3—H3A	118.8 (10)	C12—C11—H11A	121.3 (10)
N1—C4—C5	120.58 (12)	C10—C11—H11A	118.5 (10)
N1—C4—C3	121.28 (11)	C13—C12—C11	117.90 (14)
C5—C4—C3	118.13 (12)	C13—C12—H12A	121.0 (12)
C6—C5—C4	120.50 (12)	C11—C12—H12A	121.1 (12)
C6—C5—H5A	121.3 (12)	N2—C13—C12	123.98 (13)
C4—C5—H5A	118.2 (12)	N2—C13—H13A	115.0 (11)
C5—C6—C1	121.92 (11)	C12—C13—H13A	121.0 (11)
C5—C6—H6A	119.0 (10)	N2—C14—C10	123.89 (14)
C1—C6—H6A	119.0 (10)	N2—C14—H14A	114.7 (10)
O1—C7—C1	121.71 (11)	C10—C14—H14A	121.4 (10)
C6—C1—C2—C3	1.23 (18)	O1—C7—C8—C9	−12.5 (2)
C7—C1—C2—C3	−178.45 (12)	C1—C7—C8—C9	168.37 (12)
C1—C2—C3—C4	−0.56 (19)	C7—C8—C9—C10	−176.57 (13)
C2—C3—C4—N1	179.56 (12)	C8—C9—C10—C11	168.40 (14)
C2—C3—C4—C5	−0.85 (19)	C8—C9—C10—C14	−10.3 (2)
N1—C4—C5—C6	−178.84 (13)	C14—C10—C11—C12	1.1 (2)
C3—C4—C5—C6	1.57 (19)	C9—C10—C11—C12	−177.71 (14)
C4—C5—C6—C1	−0.9 (2)	C10—C11—C12—C13	0.9 (2)
C2—C1—C6—C5	−0.50 (19)	C14—N2—C13—C12	1.8 (2)
C7—C1—C6—C5	179.18 (12)	C11—C12—C13—N2	−2.5 (2)
C6—C1—C7—O1	165.18 (12)	C13—N2—C14—C10	0.5 (2)
C2—C1—C7—O1	−15.15 (19)	C11—C10—C14—N2	−1.9 (2)
C6—C1—C7—C8	−15.74 (19)	C9—C10—C14—N2	176.92 (14)
C2—C1—C7—C8	163.92 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O1^{i}	0.88 (2)	2.13 (2)	2.9920 (16)	170 (2)
N1—H2N1···N2^{ii}	0.93 (2)	2.26 (2)	3.1471 (17)	161.7 (19)

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+3/2; (ii) −*x*+1, *y*−1/2, −*z*+3/2.

Fig. 1

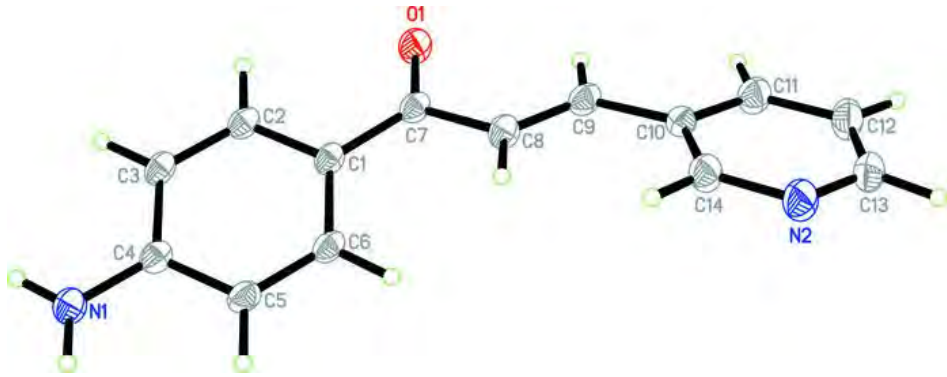
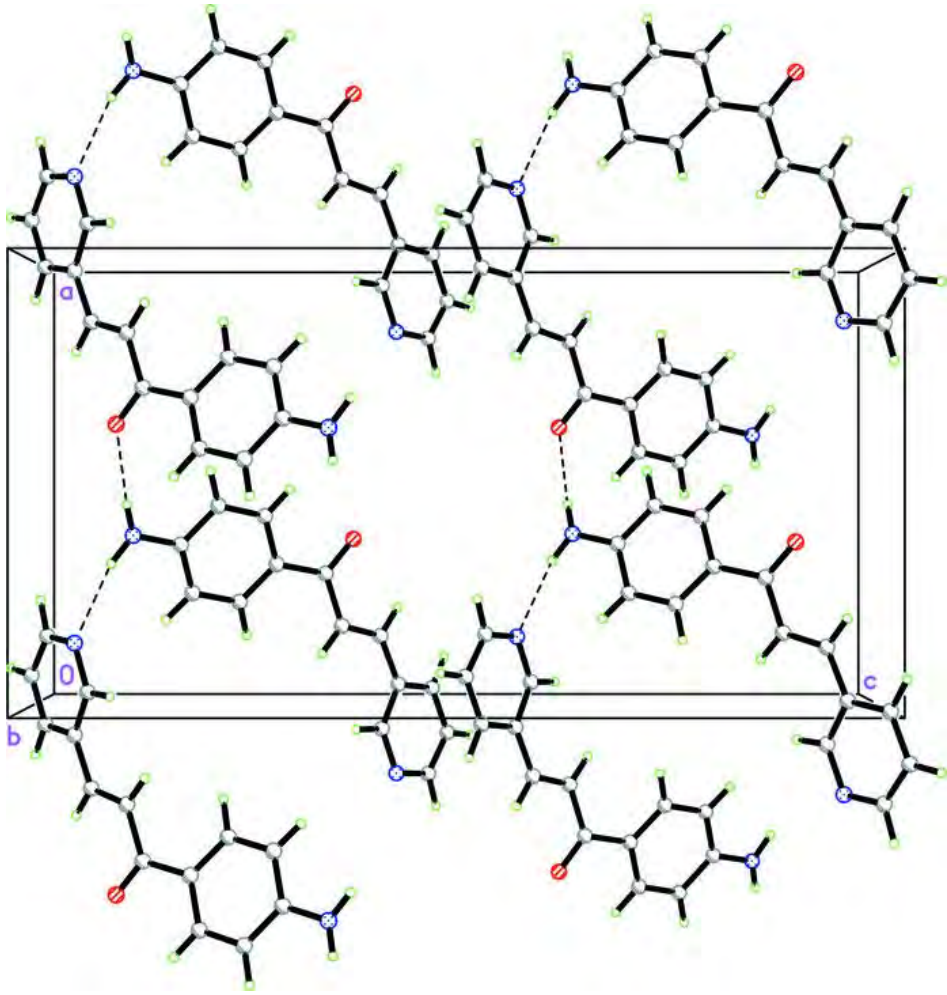


Fig. 2

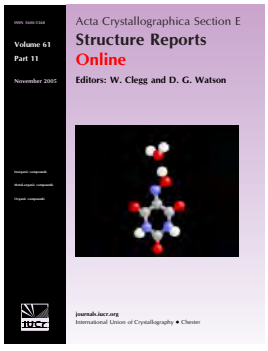


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-1-(4-Aminophenyl)-3-(naphthalen-2-yl)prop-2-en-1-one

Thawanrat Kobkeatthawin, Suchada Chantrapromma, Nisakorn Saewan and Hoong-Kun Fun

Acta Cryst. (2011). E**67**, o1204–o1205



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(4-Aminophenyl)-3-(naphthalen-2-yl)prop-2-en-1-one

Thawanrat Kobkeatthawin,[a] Suchada Chantrapromma,[a]*‡ Nisakorn Saewan[b] and Hoong-Kun Fun[c]§

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]School of Cosmetic Science, Mae Fah Luang University, Muang, Chiang Rai 57100, Thailand, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

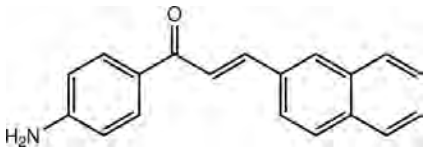
Received 29 March 2011; accepted 13 April 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.057; wR factor = 0.119; data-to-parameter ratio = 9.8.

The molecule of the title chalcone derivative, $C_{19}H_{15}NO$, exists in a *trans* configuration with respect to the C=C double bond. The molecule is slightly twisted with a dihedral angle of 6.12 (12)° between the benzene ring and the naphthalene ring system. The prop-2-en-1-one bridge is nearly planar, with an r.m.s. deviation of 0.0194 (2), and makes dihedral angles of 8.05 (19) and 11.47 (18)° with the benzene ring and the naphthalene ring system, respectively. In the crystal, molecules are linked by N—H···O hydrogen bonds into chains along the *b* axis. Weak N—H···π and C—H···π interactions and a short N···O contact [2.974 (4) Å] are also observed.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related structures, see: Fun *et al.* (2008); Horkaew *et al.* (2010). For background to and applications of chalcones, see: Bandgar & Gawande (2010); Cheng *et al.* (2008); Gaber *et al.* (2008); Nerya *et al.* (2004); Nowakowska *et al.* (2008); Patil *et al.* (2007); Svetlichny *et al.* (2007); Tewtrakul *et al.* (2003); Xu *et al.* (2005). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{19}H_{15}NO$
M_r = 273.32
Orthorhombic, $P2_12_12_1$
a = 5.7422 (6) Å
b = 9.8022 (10) Å
c = 25.504 (3) Å
V = 1435.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 100 K
0.32 × 0.28 × 0.07 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.976, T_{max} = 0.994
8109 measured reflections
1940 independent reflections
1633 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.119
S = 1.14
1940 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1, *Cg*2 and *Cg*3 are the centroids of the C1–C6, C10–C12/C17–C19 and C12–C17 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···O1^{i}	0.90 (4)	2.12 (4)	2.974 (4)	159 (4)
N1—H2*N*1···*Cg*1^{ii}	0.86 (4)	2.99 (4)	3.475 (3)	118 (3)
C5—H5*A*···*Cg*3^{iii}	0.93	2.82	3.513 (3)	132
C11—H11*A*···*Cg*3^{iv}	0.93	2.92	3.631 (3)	135
C13—H13*A*···*Cg*2^{iv}	0.93	2.86	3.551 (3)	132
C16—H16*A*···*Cg*1^{iii}	0.93	2.87	3.603 (4)	136

Symmetry codes: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (iii) $-x-1, y+\frac{3}{2}, -z+\frac{5}{2}$; (iv) $-x, y+\frac{1}{2}, -z+\frac{5}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for the research grant (RSA 5280033) and the Prince of Songkla University for financial support. The authors also thank Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2579).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bandgar, B. P. & Gawande, S. S. (2010). *J. Bioorg. Med. Chem.* **18**, 2060–2065.
Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cheng, J. H., Hung, C.-F., Yang, S. C., Wang, J.-P., Won, S.-J. & Lin, S.-J. (2008). *J. Bioorg. Med. Chem.* **16**, 7270–7276.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

‡ Thomson Reuters ResearcherID: A-5085-2009.
§ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Fun, H.-K., Patil, P. S., Dharmaprakash, S. M. & Chantrapromma, S. (2008). *Acta Cryst.* E**64**, o1540–o1541.
Gaber, M., El-Daly, S. A., Fayed, T. A. & El-Sayed, Y. S. (2008). *J. Opt. Laser Techol.* **40**, 528–537.
Horkaew, J., Chantrapromma, S., Saewan, N. & Fun, H.-K. (2010). *Acta Cryst.* E**66**, o2346–o2347.
Nerya, O., Musa, R., Khatib, S., Tamir, S. & Vaya, J. (2004). *Phytochemistry*, **65**, 1389–1395.
Nowakowska, Z., Kedzia, B. & Schroeder, G. (2008). *Eur. J. Med. Chem.* **43**, 707–713.
Patil, P. S., Dharmaprakash, S. M., Ramakrishna, K., Fun, H.-K., Kumar, R. S. S. & Narayana Rao, D. (2007). *J. Cryst. Growth.* **303**, 520–524.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Svetlichny, V. Y., Merola, F., Dobretsov, G. E., Gularyan, S. K. & Syrejshchikova, T. I. (2007). *J. Chem. Phys. Lipids*, **145**, 13–26.
Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol.* **25**, 503–508.
Xu, Z., Bai, G. & Dong, C. (2005). *J. Bioorg. Med. Chem.* **13**, 5694–5699.

supplementary materials

Acta Cryst. (2011). E**67**, o1204-o1205 [**doi:10.1107/S1600536811014024**]

(*E*)-1-(4-Aminophenyl)-3-(naphthalen-2-yl)prop-2-en-1-one

T. Kobkeatthawin, S. Chantrapromma, N. Saewan and H.-K. Fun

Comment

Chalcones are the precursors of flavonoids and antiflavonoids which are available in plenty in ferns and higher plants. Their derivatives are known to display a variety of biological properties such as analgesic, anti-inflammatory, antibacterial and antifungal activities (Bandgar & Gawande, 2010; Cheng *et al.*, 2008; Nowakowska *et al.*, 2008), HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003) and tyrosinase inhibitory (Nerya *et al.*, 2004) activities. Moreover, some of them have also been studied for fluorescent property (Gaber *et al.*, 2008) and used for sensor, liquid crystal display and fluorescence probe for sensing of DNA or proteins (Svetlichny *et al.*, 2007; Xu *et al.*, 2005). In addition, some of them exhibit second harmonic generation (SHG), and hence are used in non-linear optical (NLO) applications (Patil *et al.*, 2007). These interesting properties of chalcones have lead us to synthesize the title compound, (I), which contains the amino and fluorophore (naphthalene) groups in order to study its NLO and fluorescent properties. Compound (I) crystallizes in the chiral orthorhombic $P2_12_12_1$ space group and should therefore exhibit second-order nonlinear optical properties. (I) also shows fluorescent emission at 440 nm when excited at 380 nm. Our results also showed that (I) was inactive for tyrosinase inhibitory. Herein the crystal structure of (I) is reported.

The molecule of the title chalcone derivative (Fig. 1), $C_{19}H_{15}NO$, exists in an *E* configuration with respect to the C8═C9 ethenyl bond [1.330 (4) Å], as indicated by the torsion angle C7–C8–C9–C10 = 179.1 (3)°. The molecule is slightly twisted with the dihedral angle between the benzene and naphthalene rings of 6.12 (12)°. The prop-2-en-1-one unit (C7—C9/O1) is nearly planar [*r.m.s*. of 0.0194 (2) Å] and the torsion angle O1–C7–C8–C9 is 6.4 (5)°. This middle bridge makes dihedral angles of 8.05 (19) and 11.47 (18) ° with the benzene and naphthalene rings, respectively. The bond distances are of normal values (Allen *et al.*, 1987) and are comparable with those found in related structures (Fun *et al.*, 2008; Horkaew *et al.*, 2010).

In the crystal packing, the molecules are linked by N—H···O hydrogen bonds (Table 1) into chains along the *b* axis (Fig. 2). N—H···π and C—H···π weak interactions (Table 1) are present in the crystal. In addition, a N···O short contact [2.974 (4) Å; symmetry code 2 - *x*, 1/2 + *y*, 3/2 - *z*] is also observed.

Experimental

The title compound was synthesized by condensation of 4-aminoacetophenone (0.40 g, 3 mmol) with 2-naphthaldehyde (0.46 g, 3 mmol) in ethanol (25 ml) in the presence of 20% NaOH(aq) (5 ml). After stirring for 6 h at room temperature, the resulting yellow solid was collected by filtration, washed with distilled diethylether, dried and purified by repeated recrysallization from acetone. Yellow plate-shaped single crystals of the title compound suitable for X-ray structure determination were recrystalized from acetone by slow evaporation of the solvent at room temperature after several days. M.p. 416–417 K.

Refinement

Anime H atoms were located in a difference Fourier map and refined isotropically. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C—H) = 0.93 Å and $U_{iso} = 1.2U_{eq}$(C). The highest residual

electron density peak is located at 0.73 Å from C11 and the deepest hole is located at 1.23 Å from C5. A total of 1345 Friedel pairs were merged as there is no significant anomalous dispersion to determine the absolute cofiguration.

Figures

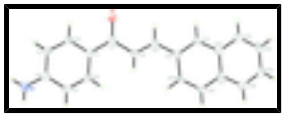


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

Fig. 2. The crystal packing of the title compound viewed along the *a* axis, showing chains running along the [010] direction.

(*E*)-1-(4-Aminophenyl)-3-(naphthalen-2-yl)prop-2-en-1-one

Crystal data

$C_{19}H_{15}NO$	D_x = 1.265 Mg m^{-3}
M_r = 273.32	Melting point = 416–417 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 1940 reflections
a = 5.7422 (6) Å	θ = 2.2–27.5°
b = 9.8022 (10) Å	μ = 0.08 mm^{-1}
c = 25.504 (3) Å	T = 100 K
V = 1435.5 (3) Å^3	Plate, yellow
Z = 4	0.32 × 0.28 × 0.07 mm
F(000) = 576	

Data collection

Bruker APEX DUO CCD area-detector diffractometer	1940 independent reflections
Radiation source: sealed tube	1633 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.042
φ and ω scans	θ_{max} = 27.5°, θ_{min} = 2.2°
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	h = −6→7
T_{min} = 0.976, T_{max} = 0.994	k = −9→12
8109 measured reflections	l = −32→33

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.057$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.119$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.14$	$w = 1/[\sigma^2(F_o^2) + (0.0233P)^2 + 1.3561P]$ where $P = (F_o^2 + 2F_c^2)/3$
1940 reflections	$(\Delta/\sigma)_{max} = 0.001$
198 parameters	$\Delta\rho_{max} = 0.29$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.1598 (4)	0.7032 (2)	0.85128 (9)	0.0266 (5)
N1	0.5373 (7)	1.1585 (3)	0.74175 (13)	0.0339 (8)
H2N1	0.409 (8)	1.195 (4)	0.7515 (15)	0.045 (13)*
H1N1	0.599 (7)	1.187 (4)	0.7112 (14)	0.033 (11)*
C1	0.8419 (6)	0.8481 (3)	0.83133 (11)	0.0201 (7)
C2	0.9516 (6)	0.9000 (3)	0.78648 (12)	0.0224 (7)
H2A	1.0945	0.8641	0.7763	0.027*
C3	0.8535 (6)	1.0026 (3)	0.75714 (12)	0.0230 (7)
H3A	0.9314	1.0356	0.7278	0.028*
C4	0.6354 (6)	1.0583 (3)	0.77112 (12)	0.0233 (7)
C5	0.5252 (6)	1.0072 (3)	0.81610 (12)	0.0232 (7)
H5A	0.3827	1.0434	0.8264	0.028*
C6	0.6244 (6)	0.9045 (3)	0.84519 (12)	0.0219 (7)
H6A	0.5466	0.8715	0.8746	0.026*
C7	0.9597 (6)	0.7405 (3)	0.86224 (13)	0.0222 (7)
C8	0.8373 (6)	0.6782 (3)	0.90735 (12)	0.0221 (7)
H8A	0.6818	0.7000	0.9133	0.027*
C9	0.9441 (6)	0.5920 (3)	0.93962 (11)	0.0198 (7)
H9A	1.0988	0.5718	0.9321	0.024*
C10	0.8433 (6)	0.5257 (3)	0.98562 (12)	0.0187 (7)
C11	0.9577 (6)	0.4164 (3)	1.00866 (11)	0.0204 (7)

H11A	1.1021	0.3900	0.9956	0.025*
C12	0.8612 (6)	0.3438 (3)	1.05134 (12)	0.0199 (7)
C13	0.9733 (6)	0.2294 (3)	1.07446 (12)	0.0234 (7)
H13A	1.1171	0.2010	1.0617	0.028*
C14	0.8731 (7)	0.1609 (3)	1.11511 (13)	0.0275 (8)
H14A	0.9490	0.0864	1.1299	0.033*
C15	0.6544 (7)	0.2022 (3)	1.13492 (13)	0.0276 (8)
H15A	0.5864	0.1540	1.1624	0.033*
C16	0.5423 (7)	0.3123 (3)	1.11405 (12)	0.0263 (7)
H16A	0.3998	0.3395	1.1279	0.032*
C17	0.6402 (6)	0.3855 (3)	1.07164 (11)	0.0196 (7)
C18	0.5282 (6)	0.4987 (3)	1.04803 (12)	0.0220 (7)
H18A	0.3853	0.5277	1.0611	0.026*
C19	0.6240 (6)	0.5658 (3)	1.00685 (12)	0.0211 (7)
H19A	0.5453	0.6395	0.9922	0.025*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0249 (13)	0.0254 (11)	0.0297 (12)	0.0044 (12)	0.0068 (11)	0.0020 (10)
N1	0.0305 (19)	0.0385 (18)	0.0326 (17)	0.0104 (17)	0.0064 (16)	0.0122 (15)
C1	0.0240 (18)	0.0180 (15)	0.0185 (14)	−0.0039 (15)	−0.0014 (14)	−0.0037 (12)
C2	0.0215 (17)	0.0213 (15)	0.0244 (16)	−0.0025 (15)	0.0041 (14)	−0.0062 (13)
C3	0.0257 (18)	0.0216 (15)	0.0216 (15)	−0.0037 (17)	0.0047 (15)	−0.0005 (13)
C4	0.0236 (17)	0.0226 (15)	0.0237 (15)	−0.0024 (16)	−0.0032 (15)	−0.0039 (13)
C5	0.0183 (17)	0.0272 (16)	0.0242 (15)	0.0003 (15)	0.0017 (14)	−0.0039 (14)
C6	0.0196 (17)	0.0233 (15)	0.0229 (15)	−0.0050 (15)	0.0028 (14)	−0.0033 (13)
C7	0.0227 (17)	0.0184 (15)	0.0256 (16)	−0.0021 (15)	0.0001 (15)	−0.0051 (13)
C8	0.0242 (18)	0.0152 (14)	0.0269 (15)	−0.0016 (15)	0.0052 (15)	−0.0045 (13)
C9	0.0203 (16)	0.0113 (13)	0.0278 (16)	−0.0040 (13)	0.0037 (14)	−0.0071 (12)
C10	0.0207 (16)	0.0121 (14)	0.0234 (15)	−0.0012 (13)	−0.0001 (14)	−0.0065 (11)
C11	0.0153 (15)	0.0220 (15)	0.0240 (15)	−0.0012 (15)	0.0005 (14)	−0.0075 (13)
C12	0.0202 (17)	0.0171 (14)	0.0225 (14)	−0.0037 (15)	−0.0026 (14)	−0.0074 (12)
C13	0.0234 (17)	0.0188 (15)	0.0281 (16)	−0.0019 (15)	−0.0033 (15)	−0.0065 (13)
C14	0.035 (2)	0.0181 (15)	0.0297 (17)	0.0039 (16)	−0.0075 (17)	−0.0003 (14)
C15	0.035 (2)	0.0226 (16)	0.0255 (16)	−0.0092 (17)	0.0008 (17)	0.0016 (13)
C16	0.0245 (18)	0.0296 (17)	0.0249 (16)	−0.0078 (17)	0.0015 (15)	−0.0075 (14)
C17	0.0198 (16)	0.0196 (14)	0.0196 (14)	−0.0024 (14)	0.0009 (14)	−0.0085 (12)
C18	0.0188 (16)	0.0220 (15)	0.0251 (15)	0.0022 (15)	0.0027 (14)	−0.0069 (13)
C19	0.0221 (17)	0.0152 (14)	0.0261 (15)	0.0084 (15)	−0.0014 (14)	−0.0020 (13)

Geometric parameters (Å, °)

O1—C7	1.238 (4)	C9—H9A	0.9300
N1—C4	1.358 (4)	C10—C11	1.388 (4)
N1—H2N1	0.86 (4)	C10—C19	1.426 (5)
N1—H1N1	0.90 (4)	C11—C12	1.413 (4)
C1—C2	1.402 (4)	C11—H11A	0.9300
C1—C6	1.411 (5)	C12—C13	1.421 (4)

C1—C7	1.480 (4)	C12—C17	1.430 (5)
C2—C3	1.374 (4)	C13—C14	1.363 (5)
C2—H2A	0.9300	C13—H13A	0.9300
C3—C4	1.412 (5)	C14—C15	1.413 (5)
C3—H3A	0.9300	C14—H14A	0.9300
C4—C5	1.403 (4)	C15—C16	1.364 (5)
C5—C6	1.374 (4)	C15—H15A	0.9300
C5—H5A	0.9300	C16—C17	1.415 (4)
C6—H6A	0.9300	C16—H16A	0.9300
C7—C8	1.480 (4)	C17—C18	1.417 (4)
C8—C9	1.330 (4)	C18—C19	1.356 (4)
C8—H8A	0.9300	C18—H18A	0.9300
C9—C10	1.461 (4)	C19—H19A	0.9300
C4—N1—H2N1	120 (3)	C11—C10—C19	118.1 (3)
C4—N1—H1N1	123 (2)	C11—C10—C9	119.7 (3)
H2N1—N1—H1N1	117 (4)	C19—C10—C9	122.1 (3)
C2—C1—C6	117.4 (3)	C10—C11—C12	121.9 (3)
C2—C1—C7	119.2 (3)	C10—C11—H11A	119.0
C6—C1—C7	123.4 (3)	C12—C11—H11A	119.0
C3—C2—C1	121.7 (3)	C11—C12—C13	122.6 (3)
C3—C2—H2A	119.1	C11—C12—C17	118.9 (3)
C1—C2—H2A	119.1	C13—C12—C17	118.5 (3)
C2—C3—C4	120.6 (3)	C14—C13—C12	120.9 (3)
C2—C3—H3A	119.7	C14—C13—H13A	119.6
C4—C3—H3A	119.7	C12—C13—H13A	119.6
N1—C4—C5	121.5 (3)	C13—C14—C15	120.4 (3)
N1—C4—C3	120.6 (3)	C13—C14—H14A	119.8
C5—C4—C3	117.9 (3)	C15—C14—H14A	119.8
C6—C5—C4	121.1 (3)	C16—C15—C14	120.4 (3)
C6—C5—H5A	119.4	C16—C15—H15A	119.8
C4—C5—H5A	119.4	C14—C15—H15A	119.8
C5—C6—C1	121.2 (3)	C15—C16—C17	120.8 (3)
C5—C6—H6A	119.4	C15—C16—H16A	119.6
C1—C6—H6A	119.4	C17—C16—H16A	119.6
O1—C7—C8	119.6 (3)	C16—C17—C18	122.8 (3)
O1—C7—C1	121.0 (3)	C16—C17—C12	118.9 (3)
C8—C7—C1	119.4 (3)	C18—C17—C12	118.2 (3)
C9—C8—C7	121.6 (3)	C19—C18—C17	121.7 (3)
C9—C8—H8A	119.2	C19—C18—H18A	119.2
C7—C8—H8A	119.2	C17—C18—H18A	119.2
C8—C9—C10	126.7 (3)	C18—C19—C10	121.2 (3)
C8—C9—H9A	116.7	C18—C19—H19A	119.4
C10—C9—H9A	116.7	C10—C19—H19A	119.4
C6—C1—C2—C3	0.5 (4)	C9—C10—C11—C12	−176.3 (3)
C7—C1—C2—C3	−178.1 (3)	C10—C11—C12—C13	178.3 (3)
C1—C2—C3—C4	−0.7 (5)	C10—C11—C12—C17	−0.7 (4)
C2—C3—C4—N1	−179.2 (3)	C11—C12—C13—C14	−179.0 (3)
C2—C3—C4—C5	1.0 (5)	C17—C12—C13—C14	0.0 (4)

N1—C4—C5—C6	179.1 (3)	C12—C13—C14—C15	0.2 (5)
C3—C4—C5—C6	−1.0 (5)	C13—C14—C15—C16	−0.8 (5)
C4—C5—C6—C1	0.9 (5)	C14—C15—C16—C17	1.2 (5)
C2—C1—C6—C5	−0.6 (4)	C15—C16—C17—C18	178.7 (3)
C7—C1—C6—C5	178.0 (3)	C15—C16—C17—C12	−1.0 (5)
C2—C1—C7—O1	5.3 (4)	C11—C12—C17—C16	179.4 (3)
C6—C1—C7—O1	−173.3 (3)	C13—C12—C17—C16	0.4 (4)
C2—C1—C7—C8	−175.6 (3)	C11—C12—C17—C18	−0.3 (4)
C6—C1—C7—C8	5.8 (4)	C13—C12—C17—C18	−179.3 (3)
O1—C7—C8—C9	6.4 (5)	C16—C17—C18—C19	−178.9 (3)
C1—C7—C8—C9	−172.7 (3)	C12—C17—C18—C19	0.8 (4)
C7—C8—C9—C10	179.1 (3)	C17—C18—C19—C10	−0.3 (5)
C8—C9—C10—C11	165.8 (3)	C11—C10—C19—C18	−0.6 (4)
C8—C9—C10—C19	−11.5 (5)	C9—C10—C19—C18	176.8 (3)
C19—C10—C11—C12	1.1 (4)		

Hydrogen-bond geometry (Å, °)

*Cg*1, *Cg*2 and *Cg*3 are the centroids of the C1–C6, C10–C12/C17–C19 and C12–C17 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O1^{i}	0.90 (4)	2.12 (4)	2.974 (4)	159 (4)
N1—H2N1···Cg1^{ii}	0.86 (4)	2.99 (4)	3.475 (3)	118 (3)
C5—H5A···Cg3^{iii}	0.93	2.82	3.513 (3)	132
C11—H11A···Cg3^{iv}	0.93	2.92	3.631 (3)	135
C13—H13A···Cg2^{iv}	0.93	2.86	3.551 (3)	132
C16—H16A···Cg1^{iii}	0.93	2.87	3.603 (4)	136

Symmetry codes: (i) −*x*+2, *y*+1/2, −*z*+3/2; (ii) *x*+3/2, −*y*+1/2, −*z*+1; (iii) −*x*−1, *y*+3/2, −*z*+5/2; (iv) −*x*, *y*+1/2, −*z*+5/2.

Fig. 1

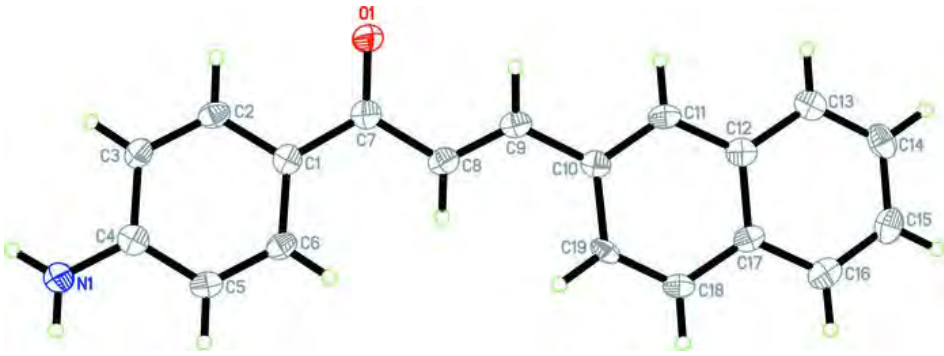
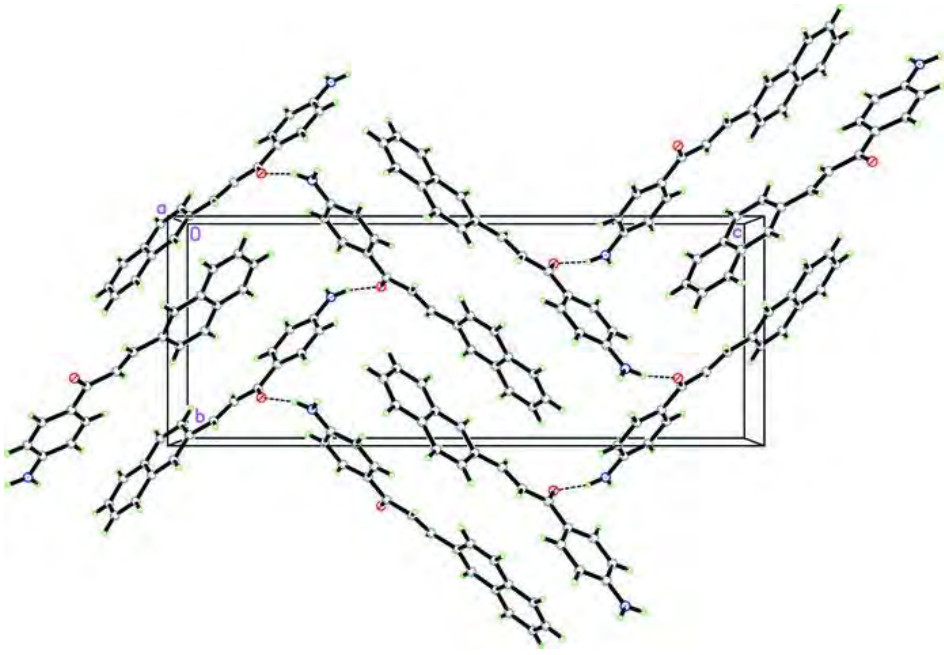


Fig. 2

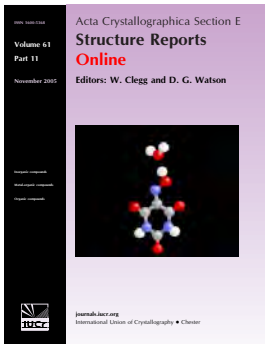


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

Redetermination and absolute configuration of pruniflorone M monohydrate

Hoong-Kun Fun, Suchada Chantrapromma, Nawong Boonnak, Chatchanok Karalai and Kan Chantrapromma

Acta Cryst. (2011). E**67**, o1916–o1917



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination and absolute configuration of pruniflorone M monohydrate

Hoong-Kun Fun,[a]*‡ Suchada Chantrapromma,[b]§ Nawong Boonnak,[b] Chatchanok Karalai[b] and Kan Chantrapromma[c]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [c]Research Unit of Natural Products Utilization, Walailak University, Thasala, Nakhon Si Thammarat 80160, Thailand
Correspondence e-mail: hkfun@usm.my

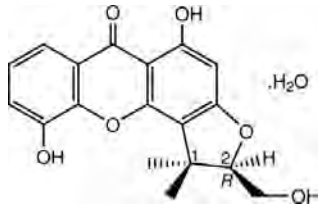
Received 23 June 2011; accepted 27 June 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.120; data-to-parameter ratio = 10.9.

The title xanthone known as pruniflorone M (systematic name: (2*R*)-5,10-dihydroxy-2-hydroxymethyl-1,1-dimethyl-1*H*-furo[2,3-*c*]xanthen-6-one), crystallized in a monohydrate form, $C_{18}H_{16}O_6\cdot H_2O$. It was isolated from the green fruits of *Cratoxylum formosum* ssp. *pruniflorum*. The structure of the title compound has been reported previously [Boonnak *et al.* (2010). *Aust. J. Chem.* **63**, 1550–1556], but we report here the absolute configuration determined using Cu Kα radiation. There are two crystallograpically independent molecules in the asymmetric unit, which differ slightly in the bond angles. The hydroxymethyl substituents at position 2 of the furan rings of both pruniflorone M molecules adopt *R* configurations. In both molecules, the three rings of the xanthone skeleton are approximately coplanar, with an r.m.s. deviation of 0.0124 (2) Å for one molecule and 0.0289 (2) Å for the other, and the furan ring adopts an envelope conformation. In the crystal, molecules of pruniflorone M and water are linked into a two-dimensional network by O—H···O hydrogen bonds and weak C—H···O interactions. The crystal structure is further consolidated by π–π interactions with centroid–centroid distances in the range 3.5987 (13)–3.7498 (14) Å. Short C···C [3.378 (3) Å] and O···O [2.918 (3) Å] contacts are also observed.

Related literature

For details of hydrogen-bond motifs, see: Bernstein *et al.* (1995) and for ring conformations, see: Cremer & Pople (1975). For bond-length data, see: Allen *et al.* (1987). For background to xanthones and their biological activity, see: Boonnak, Karalai *et al.* (2007); Boonnak *et al.* (2009, 2010); Hay *et al.* (2008); Marques *et al.* (2000); Molinar-Toribio *et al.* (2006); Phongpaichit *et al.* (1994); Yu *et al.* (2007). For related structures, see: Boonnak *et al.* (2006); Boonnak, Fun *et al.* (2007). For the stability of the temperature controller used in the data collection, see Cosier & Glazer (1986).



Experimental

Crystal data

$C_{18}H_{16}O_6\cdot H_2O$
M_r = 346.32
Orthorhombic, $P2_12_12_1$
a = 9.8887 (3) Å
b = 15.6028 (4) Å
c = 20.4857 (5) Å
V = 3160.77 (15) Å^3
Z = 8
Cu Kα radiation
μ = 0.95 mm^{-1}
T = 100 K
0.54 × 0.17 × 0.10 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.627, T_{max} = 0.913
13449 measured reflections
4981 independent reflections
4753 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.120
S = 1.02
4981 reflections
456 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 2102 Friedel pairs
Flack parameter: 0.06 (19)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3*A*—H3*OA*···O2*A*	0.98	1.62	2.530 (3)	152
O4*A*—H4*OA*···O1*WA*	0.92	1.75	2.672 (3)	175
O6*A*—H6*OA*···O4*B*^{i}	0.82	2.12	2.918 (3)	165
O3*B*—H3*OB*···O2*B*	1.06	1.57	2.529 (3)	148
O4*B*—H4*OB*···O1*WB*	1.02	1.62	2.639 (3)	175
O6*B*—H6*OB*···O4*A*^{ii}	0.82	2.25	3.059 (4)	167
O1*WA*—H1*WA*···O3*A*^{iii}	0.83	2.06	2.889 (3)	173
O1*WA*—H2*WA*···O6*B*	0.92	1.84	2.737 (5)	165
O1*WB*—H1*WB*···O6*A*	0.89	1.86	2.691 (3)	153
O1*WB*—H2*WB*···O3*B*^{iv}	0.82	2.06	2.868 (3)	164
C16*B*—H16*C*···O2*B*^{v}	0.96	2.46	3.389 (3)	163

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

The authors thank the Thailand Research Fund (TRF) for research grant (RSA 5280033) and the Prince of Songkla University for financial support. The authors also thank the Koshinocorporation Group, Japan, and Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5174).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Boonnak, N., Chantrapromma, S. & Fun, H.-K. (2006). *Acta Cryst.* E**62**, o2034–o2036.

Boonnak, N., Fun, H.-K., Chantrapromma, S. & Karalai, C. (2007). *Acta Cryst.* E**63**, o3958–o3959.

Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Fun, H.-K., Kanjana-Opas, A., Chantrapromma, K. & Kato, S. (2009). *Tetrahedron*, **65**, 3003–3013.

Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Kanjana-Opas, A., Chantrapromma, K. & Fun, H.-K. (2007). *Can. J. Chem.* **85**, 341–345.

Boonnak, N., Khamthip, A., Karalai, C., Chantrapromma, S., Ponglimanont, C., Kanjana-Opas, A., Tewtrakul, S., Chantrapromma, K., Fun, H.-K. & Kato, S. (2010). *Aust. J. Chem.* **63**, 1550–1556.

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Hay, A. E., Merza, J., Landreau, A., Litaudon, M., Pagniez, F., Pape, P. L. & Richomme, P. (2008). *Fitoterapia*, **79**, 42–46.

Marques, V. L. L., Oliveira, F. M. D., Conserva, L. M., Brito, R. G. L. & Guilhon, G. M. S. P. (2000). *Phytochemistry*, **55**, 815–818.

Molinar-Toribio, E., González, J., Ortega-Barría, E., Capson, T. L., Coley, P. D., Kursar, T. A., McPhail, K. & Cubilla-Rios, L. (2006). *Pharm. Biol.* **44**, 550–553.

Phongpaichit, S., Nilrat, L., Tharavichitkul, P., Bunchoo, S., Chuaprapaisilp, T. & Wiriyachitra, P. (1994). *Songklanakarin J. Sci. Technol.* **16**, 399–405.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Yu, L., Zhao, M., Yang, B., Zhao, Q. & Jiang, Y. (2007). *Food Chem.* **104**, 176–181.

supplementary materials

Acta Cryst. (2011). E**67**, o1916-o1917 [**doi:10.1107/S1600536811025177**]

Redetermination and absolute configuration of pruniflorone M monohydrate

H.-K. Fun, S. Chantrapromma, N. Boonnak, C. Karalai and K. Chantrapromma

Comment

Xanthones are secondary metabolites of several plants and exhibit considerable biological activities such as antibacterial, antioxidant, antiprotozoal, cytotoxic and nitric oxide inhibitory activities (Boonnak, Karalai *et al.* 2007; Boonnak *et al.* 2009, 2010; Hay *et al.* 2008; Marques *et al.* 2000; Molinar-Toribio *et al.*, 2006; Phongpaichit *et al.*, 1994; Yu *et al.*, 2007). During the course of our research on the chemical constituents and bioactive compounds from the green fruits of *Cratoxylum formosum* ssp. *pruniflorum*, which were collected from Pha Yao province in the northern part of Thailand, the title xanthone (I) known as pruniflorone M (Boonnak *et al.*, 2010) was isolated. The previous report showed that (I) possess nitric oxide inhibitory activity (Boonnak *et al.*, 2010). The absolute configuration of (I) was determined by making use of the anomalous scattering of Cu Kα *X*-radiation with the Flack parameter being refined to 0.06 (19). We report herein the crystal structure of (I).

There are two crystallograpically independent molecules *A* and *B* in the asymmetric unit of (I), $C_{18}H_{16}O_6.H_2O$, (Fig. 1) with the same conformation but with slight differences in bond angles. In the structure of (I), the three ring system [C1–C13/O1] is essentially planar with *r.m.s*. deviations of 0.0124 (2) Å for molecule *A* [0.0289 (2) Å for molecule *B*] from the plane through 14 non-hydrogen atoms of the three rings. The O3 and O4 hydroxy O atoms lie close to this plane with deviations +0.038 (2) for O3 and +0.004 (2) Å for O4 for molecule *A* [the corresponding values are +0.043 (2) and -0.024 (2) Å for molecule *B*]. The furan ring (C3–C4/C14–C15/O5) is in an envelope conformation with the puckering atom C15 of 0.148 (3) Å, and puckering parameter Q = 0.239 (2) Å and φ = 132.0 (6)° (Cremer & Pople, 1975) for molecule *A* and the corresponding values are 0.134 (3) Å, 0.213 (3) Å and φ = 137.8 (7)° for molecule *B*. The orientation of the hydroxymethyl moiety at atom C15 can be indicated by the torsion angle of C14–C15–C16–O6 = -73.3 (3)° for molecule *A* [165.0 (3)° for molecule *B*]. Intramolecular O3A—H3OA···O2A and O3B—H3OB..O2B hydrogen bonds (Table 1) generate S(6) ring motifs (Bernstein *et al.*, 1995). The bond distances in (I) are within normal ranges (Allen *et al.*, 1987) and comparable to the related structures (Boonnak *et al.*, 2006; Boonnak, Fun *et al.*, 2007). The hydroxymethyl substituents at position 2 (on atoms C15A and C15B) of the furan rings of both pruniflorone M molecules adopt *R* configurations.

In the crystal packing of (I) (Fig. 2), the molecules of pruniflorone M and water are linked into a two dimensional network by O—H···O hydrogen bonds and weak C—H···O interactions (Table 1). π···π interactions were also observed with centroid···centroid distances: $Cg_1\cdots Cg_5^{v}$ = 3.7453 (13) Å; $Cg_1\cdots Cg_6^{vi}$ = 3.6847 (13) Å; $Cg_2\cdots Cg_4^{vi}$ = 3.7189 (12) Å; $Cg_2\cdots Cg_6^{vi}$ = 3.6940 (14) Å; $Cg_3\cdots Cg_4^{v}$ = 3.5987 (13) Å and $Cg_3\cdots Cg_5^{v}$ = 3.7498 (14) Å; Cg_1, Cg_2, Cg_3, Cg_4, Cg_5 and Cg_6 are the centroids of C9A–C13A/O1A, C1A–C4A/C11A–C12A, C5A–C9A/C13A, C9B–C13B/O1B, C1B–C4B/C11B–C12B and C5B–C9B/C13B rings, respectively. C···C^{v}[3.378 (3) Å;] and O···O^{i}[2.918 (3) Å short contacts were also observed; [symmetry codes: (i) -1/2+x, 3/2-y, 1-z; (v) 3/2-x, 1-y, 1/2+z and (vi) 1/2-x, 1-y, 1/2-z].

Experimental

The green fruits of *C. formosum* ssp. *pruniflorum* (5.00 kg) were extracted with CH_2Cl_2 (2x20 L, for a week) successively at room temperature and were further evaporated under reduced pressure to afford the crude CH_2Cl_2 extracts (31.42 g). The crude extract was further subjected to QCC (Quick Column Chromatography) on silica gel using hexane as a first eluent and then increasing the polarity with acetone to give 14 fractions (F1-F14). Fraction F10 was separated by QCC eluting with a gradient of acetone-hexane to give 17 subfractions (F10A-F10Q). Subfractions F10N was further separated by CC and eluted with a gradient of EtOAc-hexane to give 8 subfractions (F10N1-F10N8). Subfraction F10N2 was further separated by CC and eluted with $CHCl_3$ to give the title compound as yellow powder (28.0 mg). Yellow block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystallized from $CHCl_3$ by the slow evaporation of the solvent at room temperature after several days, Mp. 508-510 K.

Refinement

All H atoms were placed in calculated positions with (O—H) = 0.82-1.06 Å for OH, (C—H) = 0.93 for aromatic and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 1.34 Å from O6B and the deepest hole is located at 0.51 Å from O6B. 2102 Friedel pairs were used to determine the absolute configuration. There is no pseudo-symmetry observed in the crystal structure.

Figures

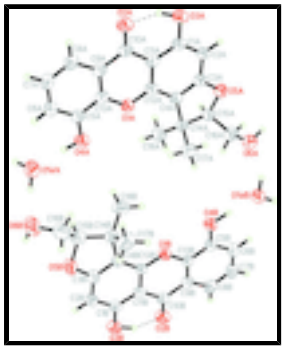


Fig. 1. The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

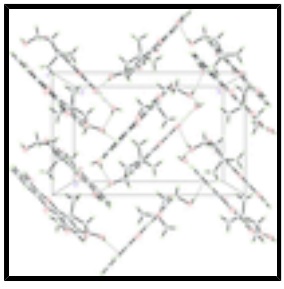


Fig. 2. The crystal packing of (I) viewed along the *c* axis, showing two dimensional network. Hydrogen bonds are shown as dashed lines.

(2*R*)-5,10-Dihydroxy-2-hydroxymethyl-1,1-dimethyl-1*H*- furo[2,3-*c*]xanthen-6-one monohydrate

Crystal data

$C_{18}H_{16}O_6 \cdot H_2O$	$D_x = 1.456$ Mg m^{-3}
$M_r = 346.32$	Melting point = 508–510 K
Orthorhombic, $P2_12_12_1$	Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 4981 reflections
$a = 9.8887$ (3) Å	$\theta = 5.3$–$63.5°$
$b = 15.6028$ (4) Å	$\mu = 0.95$ mm^{-1}
$c = 20.4857$ (5) Å	$T = 100$ K
$V = 3160.77$ (15) Å^3	Block, yellow
$Z = 8$	0.54 × 0.17 × 0.10 mm
$F(000) = 1456$	

Data collection

Bruker APEX DUO CCD area-detector diffractometer	4981 independent reflections
Radiation source: sealed tube	4753 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.021$
φ and ω scans	$\theta_{max} = 63.5°$, $\theta_{min} = 5.3°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	$h = -7 \rightarrow 11$
$T_{min} = 0.627$, $T_{max} = 0.913$	$k = -18 \rightarrow 18$
13449 measured reflections	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.120$	$w = 1/[\sigma^2(F_o^2) + (0.0751P)^2 + 0.9688P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
4981 reflections	$\Delta\rho_{max} = 0.56$ e Å^{-3}
456 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
0 restraints	Absolute structure: Flack (1983), 2102 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.06 (19)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.40973 (17)	0.43207 (10)	0.67334 (7)	0.0370 (4)
O2A	0.3650 (2)	0.44183 (12)	0.87235 (8)	0.0512 (5)
O3A	0.18470 (19)	0.55552 (12)	0.85896 (8)	0.0483 (4)
H3OA	0.2444	0.5118	0.8781	0.072*
O4A	0.58566 (19)	0.32867 (12)	0.61541 (8)	0.0484 (4)
H4OA	0.6449	0.2869	0.6019	0.062 (9)*
O5A	0.06909 (18)	0.63151 (11)	0.64104 (8)	0.0444 (4)
O6A	0.1454 (2)	0.74151 (11)	0.54195 (9)	0.0551 (5)
H6OA	0.0952	0.7697	0.5655	0.083*
C1A	0.2000 (2)	0.54898 (15)	0.79348 (11)	0.0357 (5)
C2A	0.1201 (2)	0.59682 (17)	0.75260 (11)	0.0381 (5)
H2A	0.0538	0.6334	0.7688	0.046*
C3A	0.1426 (2)	0.58815 (14)	0.68612 (11)	0.0353 (5)
C4A	0.2403 (2)	0.53548 (14)	0.65853 (11)	0.0339 (5)
C5A	0.5798 (2)	0.32706 (15)	0.68149 (11)	0.0378 (5)
C6A	0.6618 (3)	0.27595 (16)	0.71967 (13)	0.0426 (6)
H6A	0.7249	0.2404	0.6997	0.051*
C7A	0.6521 (3)	0.27649 (16)	0.78758 (13)	0.0460 (6)
H7A	0.7088	0.2418	0.8123	0.055*
C8A	0.5592 (3)	0.32806 (16)	0.81792 (12)	0.0428 (6)
H8A	0.5519	0.3275	0.8632	0.051*
C9A	0.4758 (3)	0.38129 (15)	0.78110 (11)	0.0375 (5)
C10A	0.3778 (2)	0.43875 (15)	0.81172 (11)	0.0374 (5)
C11A	0.2983 (2)	0.49098 (15)	0.76823 (11)	0.0341 (5)
C12A	0.3168 (2)	0.48597 (14)	0.70018 (11)	0.0336 (5)
C13A	0.4870 (2)	0.38085 (15)	0.71280 (11)	0.0345 (5)
C14A	0.2429 (2)	0.54925 (16)	0.58514 (11)	0.0376 (5)
C15A	0.1020 (3)	0.59373 (16)	0.57746 (11)	0.0409 (5)
H15A	0.0355	0.5485	0.5691	0.049*
C16A	0.0818 (3)	0.66176 (16)	0.52624 (12)	0.0476 (6)
H16A	0.1176	0.6411	0.4851	0.057*
H16B	−0.0144	0.6714	0.5205	0.057*
C17A	0.3638 (3)	0.6064 (2)	0.56656 (13)	0.0528 (7)
H17A	0.4466	0.5770	0.5764	0.079*
H17B	0.3598	0.6589	0.5910	0.079*

H17C	0.3604	0.6190	0.5207	0.079*
C18A	0.2474 (3)	0.46718 (18)	0.54526 (13)	0.0535 (7)
H18A	0.3326	0.4391	0.5520	0.080*
H18B	0.2370	0.4807	0.4998	0.080*
H18C	0.1754	0.4299	0.5588	0.080*
O1B	0.59342 (16)	0.57000 (10)	0.32981 (7)	0.0359 (4)
O2B	0.64340 (19)	0.55237 (12)	0.13115 (7)	0.0489 (4)
O3B	0.82577 (19)	0.44097 (12)	0.14837 (8)	0.0470 (4)
H3OB	0.7592	0.4841	0.1249	0.070*
O4B	0.42557 (18)	0.67981 (11)	0.38433 (7)	0.0433 (4)
H4OB	0.3510	0.7205	0.3997	0.065*
O5B	0.9092 (2)	0.35711 (12)	0.36836 (9)	0.0535 (5)
O6B	0.9636 (4)	0.2762 (3)	0.49615 (14)	0.1384 (16)
H6OB	0.9941	0.2549	0.4626	0.208*
C1B	0.8046 (2)	0.44754 (15)	0.21360 (11)	0.0356 (5)
C2B	0.8789 (3)	0.39760 (16)	0.25614 (11)	0.0395 (5)
H2B	0.9461	0.3606	0.2415	0.047*
C3B	0.8480 (3)	0.40551 (15)	0.32155 (11)	0.0386 (5)
C4B	0.7515 (2)	0.46058 (14)	0.34767 (11)	0.0353 (5)
C5B	0.4287 (2)	0.67673 (14)	0.31800 (11)	0.0348 (5)
C6B	0.3467 (3)	0.72657 (16)	0.27867 (13)	0.0444 (6)
H6B	0.2848	0.7640	0.2975	0.053*
C7B	0.3563 (3)	0.72109 (17)	0.21084 (13)	0.0496 (7)
H7B	0.2996	0.7545	0.1850	0.059*
C8B	0.4466 (3)	0.66814 (16)	0.18191 (12)	0.0437 (6)
H8B	0.4534	0.6663	0.1367	0.052*
C9B	0.5303 (2)	0.61584 (15)	0.22073 (11)	0.0348 (5)
C10B	0.6281 (2)	0.55735 (15)	0.19175 (10)	0.0359 (5)
C11B	0.7058 (2)	0.50642 (15)	0.23627 (11)	0.0336 (5)
C12B	0.6838 (2)	0.51255 (14)	0.30405 (11)	0.0313 (5)
C13B	0.5192 (2)	0.61998 (14)	0.28842 (11)	0.0328 (5)
C14B	0.7477 (2)	0.45210 (16)	0.42106 (11)	0.0396 (5)
C15B	0.8276 (3)	0.36851 (19)	0.42911 (13)	0.0539 (7)
H15B	0.7623	0.3214	0.4312	0.065*
C16B	0.9217 (3)	0.35831 (15)	0.48366 (10)	0.0792 (11)
H16C	0.8832	0.3818	0.5229	0.095*
H16D	1.0059	0.3875	0.4745	0.095*
C17B	0.8116 (3)	0.53045 (15)	0.45315 (10)	0.0629 (8)
H17D	0.9028	0.5369	0.4379	0.094*
H17E	0.8119	0.5231	0.4997	0.094*
H17F	0.7604	0.5806	0.4421	0.094*
C18B	0.6051 (3)	0.4398 (2)	0.44913 (14)	0.0662 (8)
H18D	0.5559	0.4927	0.4460	0.099*
H18E	0.6115	0.4230	0.4941	0.099*
H18F	0.5588	0.3961	0.4249	0.099*
O1WA	0.7567 (3)	0.21065 (14)	0.56948 (11)	0.0822 (8)
H1WA	0.7741	0.1639	0.5869	0.123*
H2WA	0.8243	0.2244	0.5407	0.123*
O1WB	0.2432 (2)	0.79095 (14)	0.42531 (11)	0.0745 (7)

H1WB	0.1943	0.7889	0.4620	0.112*
H2WB	0.2087	0.8328	0.4072	0.112*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0379 (9)	0.0434 (8)	0.0296 (8)	0.0054 (8)	0.0003 (7)	0.0028 (7)
O2A	0.0608 (11)	0.0647 (11)	0.0282 (8)	0.0099 (10)	−0.0001 (8)	0.0017 (8)
O3A	0.0581 (11)	0.0559 (10)	0.0308 (8)	0.0056 (9)	0.0069 (8)	−0.0025 (8)
O4A	0.0507 (10)	0.0568 (10)	0.0378 (9)	0.0077 (9)	0.0027 (8)	0.0010 (8)
O5A	0.0495 (10)	0.0472 (9)	0.0366 (8)	0.0105 (8)	−0.0005 (7)	0.0036 (7)
O6A	0.0697 (13)	0.0465 (9)	0.0491 (10)	0.0035 (9)	0.0140 (9)	0.0032 (8)
C1A	0.0389 (12)	0.0386 (12)	0.0297 (11)	−0.0064 (10)	0.0026 (9)	−0.0022 (10)
C2A	0.0401 (13)	0.0396 (12)	0.0346 (12)	−0.0005 (11)	0.0056 (10)	−0.0039 (9)
C3A	0.0337 (12)	0.0364 (11)	0.0358 (12)	−0.0034 (10)	−0.0022 (10)	0.0030 (10)
C4A	0.0358 (12)	0.0363 (11)	0.0296 (12)	−0.0027 (10)	−0.0005 (9)	−0.0001 (9)
C5A	0.0377 (13)	0.0385 (11)	0.0371 (12)	−0.0047 (11)	0.0006 (10)	0.0021 (10)
C6A	0.0409 (14)	0.0387 (12)	0.0482 (14)	0.0029 (12)	−0.0041 (11)	−0.0003 (11)
C7A	0.0492 (15)	0.0405 (13)	0.0484 (14)	0.0062 (13)	−0.0079 (12)	0.0060 (11)
C8A	0.0475 (15)	0.0425 (12)	0.0385 (13)	−0.0009 (12)	−0.0093 (11)	0.0020 (11)
C9A	0.0388 (13)	0.0396 (13)	0.0341 (12)	−0.0045 (11)	−0.0034 (10)	0.0017 (10)
C10A	0.0368 (13)	0.0439 (12)	0.0314 (11)	−0.0035 (11)	−0.0024 (10)	0.0024 (10)
C11A	0.0351 (12)	0.0376 (11)	0.0297 (11)	−0.0067 (10)	0.0005 (9)	0.0001 (10)
C12A	0.0315 (12)	0.0353 (11)	0.0341 (12)	−0.0043 (10)	0.0012 (10)	−0.0033 (9)
C13A	0.0332 (12)	0.0358 (12)	0.0344 (11)	−0.0043 (10)	−0.0036 (9)	0.0013 (9)
C14A	0.0408 (13)	0.0441 (12)	0.0279 (11)	−0.0001 (11)	−0.0011 (9)	0.0020 (10)
C15A	0.0407 (13)	0.0451 (12)	0.0370 (12)	−0.0020 (11)	−0.0020 (10)	0.0000 (10)
C16A	0.0545 (15)	0.0507 (14)	0.0378 (13)	0.0056 (13)	−0.0074 (12)	0.0036 (11)
C17A	0.0467 (15)	0.0684 (17)	0.0435 (14)	−0.0061 (14)	0.0012 (12)	0.0105 (13)
C18A	0.0688 (19)	0.0559 (15)	0.0358 (13)	0.0049 (14)	−0.0066 (12)	−0.0031 (11)
O1B	0.0369 (9)	0.0433 (8)	0.0274 (7)	0.0066 (7)	0.0002 (6)	−0.0002 (7)
O2B	0.0573 (11)	0.0624 (11)	0.0269 (8)	0.0038 (10)	0.0001 (8)	−0.0015 (8)
O3B	0.0565 (11)	0.0537 (10)	0.0307 (8)	0.0019 (9)	0.0080 (8)	−0.0073 (8)
O4B	0.0482 (10)	0.0480 (9)	0.0339 (8)	0.0089 (8)	0.0033 (8)	−0.0014 (7)
O5B	0.0621 (12)	0.0578 (11)	0.0407 (9)	0.0249 (10)	−0.0030 (9)	0.0005 (8)
O6B	0.152 (3)	0.180 (3)	0.0829 (18)	0.119 (3)	0.0523 (19)	0.066 (2)
C1B	0.0375 (12)	0.0370 (11)	0.0323 (11)	−0.0074 (10)	0.0037 (10)	−0.0076 (9)
C2B	0.0393 (13)	0.0366 (11)	0.0426 (13)	0.0030 (11)	0.0017 (11)	−0.0058 (10)
C3B	0.0419 (13)	0.0361 (11)	0.0379 (12)	0.0043 (10)	−0.0042 (11)	−0.0019 (10)
C4B	0.0383 (12)	0.0343 (11)	0.0333 (12)	−0.0002 (10)	−0.0021 (9)	−0.0045 (9)
C5B	0.0361 (12)	0.0348 (11)	0.0336 (11)	−0.0027 (10)	−0.0001 (10)	−0.0009 (9)
C6B	0.0457 (14)	0.0386 (12)	0.0488 (14)	0.0061 (12)	−0.0013 (12)	−0.0004 (11)
C7B	0.0560 (17)	0.0451 (13)	0.0476 (15)	0.0068 (14)	−0.0128 (13)	0.0047 (12)
C8B	0.0521 (15)	0.0474 (13)	0.0315 (12)	−0.0016 (13)	−0.0073 (11)	0.0034 (11)
C9B	0.0359 (12)	0.0366 (12)	0.0320 (11)	−0.0040 (10)	−0.0048 (9)	0.0002 (9)
C10B	0.0375 (13)	0.0421 (12)	0.0280 (11)	−0.0084 (11)	0.0000 (9)	−0.0020 (10)
C11B	0.0328 (11)	0.0370 (11)	0.0310 (11)	−0.0046 (10)	−0.0012 (9)	−0.0028 (10)
C12B	0.0306 (12)	0.0325 (11)	0.0307 (11)	−0.0011 (9)	0.0015 (9)	−0.0022 (9)

C13B	0.0309 (12)	0.0319 (11)	0.0355 (12)	−0.0020 (10)	−0.0030 (9)	0.0024 (9)
C14B	0.0434 (14)	0.0447 (13)	0.0308 (12)	0.0000 (11)	−0.0027 (10)	0.0013 (10)
C15B	0.0582 (17)	0.0585 (15)	0.0449 (14)	0.0020 (14)	0.0059 (13)	0.0060 (12)
C16B	0.0600 (19)	0.128 (3)	0.0492 (17)	0.033 (2)	−0.0001 (14)	0.0274 (19)
C17B	0.093 (2)	0.0581 (16)	0.0370 (14)	−0.0074 (17)	−0.0121 (15)	−0.0035 (12)
C18B	0.0537 (17)	0.098 (2)	0.0468 (15)	0.0024 (17)	0.0042 (13)	0.0220 (16)
O1WA	0.117 (2)	0.0625 (12)	0.0666 (14)	0.0365 (14)	0.0257 (13)	0.0038 (11)
O1WB	0.0965 (18)	0.0644 (12)	0.0627 (14)	0.0328 (13)	0.0390 (12)	0.0206 (11)

Geometric parameters (Å, °)

O1A—C12A	1.361 (3)	O2B—C10B	1.253 (3)
O1A—C13A	1.370 (3)	O3B—C1B	1.356 (3)
O2A—C10A	1.249 (3)	O3B—H3OB	1.0564
O3A—C1A	1.354 (3)	O4B—C5B	1.360 (3)
O3A—H3OA	0.9841	O4B—H4OB	1.0223
O4A—C5A	1.355 (3)	O5B—C3B	1.363 (3)
O4A—H4OA	0.9200	O5B—C15B	1.494 (3)
O5A—C3A	1.356 (3)	O6B—C16B	1.371 (4)
O5A—C15A	1.466 (3)	O6B—H6OB	0.8200
O6A—C16A	1.431 (3)	C1B—C2B	1.381 (3)
O6A—H6OA	0.8200	C1B—C11B	1.419 (3)
C1A—C2A	1.372 (3)	C2B—C3B	1.380 (3)
C1A—C11A	1.425 (4)	C2B—H2B	0.9300
C2A—C3A	1.387 (3)	C3B—C4B	1.391 (3)
C2A—H2A	0.9300	C4B—C12B	1.380 (3)
C3A—C4A	1.389 (3)	C4B—C14B	1.510 (3)
C4A—C12A	1.377 (3)	C5B—C6B	1.382 (3)
C4A—C14A	1.519 (3)	C5B—C13B	1.397 (3)
C5A—C6A	1.380 (3)	C6B—C7B	1.395 (4)
C5A—C13A	1.399 (3)	C6B—H6B	0.9300
C6A—C7A	1.394 (4)	C7B—C8B	1.353 (4)
C6A—H6A	0.9300	C7B—H7B	0.9300
C7A—C8A	1.370 (4)	C8B—C9B	1.408 (3)
C7A—H7A	0.9300	C8B—H8B	0.9300
C8A—C9A	1.392 (3)	C9B—C13B	1.393 (3)
C8A—H8A	0.9300	C9B—C10B	1.457 (3)
C9A—C13A	1.404 (3)	C10B—C11B	1.433 (3)
C9A—C10A	1.462 (3)	C11B—C12B	1.409 (3)
C10A—C11A	1.441 (3)	C14B—C17B	1.525 (3)
C11A—C12A	1.408 (3)	C14B—C15B	1.534 (4)
C14A—C18A	1.520 (3)	C14B—C18B	1.535 (4)
C14A—C17A	1.539 (4)	C15B—C16B	1.463 (4)
C14A—C15A	1.565 (3)	C15B—H15B	0.9800
C15A—C16A	1.506 (3)	C16B—H16C	0.9633
C15A—H15A	0.9800	C16B—H16D	0.9669
C16A—H16A	0.9700	C17B—H17D	0.9600
C16A—H16B	0.9700	C17B—H17E	0.9600
C17A—H17A	0.9600	C17B—H17F	0.9600

C17A—H17B	0.9600	C18B—H18D	0.9600
C17A—H17C	0.9600	C18B—H18E	0.9600
C18A—H18A	0.9600	C18B—H18F	0.9600
C18A—H18B	0.9600	O1WA—H1WA	0.8300
C18A—H18C	0.9600	O1WA—H2WA	0.9169
O1B—C13B	1.366 (3)	O1WB—H1WB	0.8939
O1B—C12B	1.371 (3)	O1WB—H2WB	0.8249
C12A—O1A—C13A	119.90 (17)	C1B—O3B—H3OB	107.7
C1A—O3A—H3OA	106.0	C5B—O4B—H4OB	110.2
C5A—O4A—H4OA	108.4	C3B—O5B—C15B	106.26 (19)
C3A—O5A—C15A	106.59 (18)	C16B—O6B—H6OB	109.5
C16A—O6A—H6OA	109.5	O3B—C1B—C2B	119.8 (2)
O3A—C1A—C2A	119.9 (2)	O3B—C1B—C11B	118.5 (2)
O3A—C1A—C11A	118.9 (2)	C2B—C1B—C11B	121.7 (2)
C2A—C1A—C11A	121.1 (2)	C3B—C2B—C1B	116.4 (2)
C1A—C2A—C3A	117.0 (2)	C3B—C2B—H2B	121.8
C1A—C2A—H2A	121.5	C1B—C2B—H2B	121.8
C3A—C2A—H2A	121.5	O5B—C3B—C2B	122.4 (2)
O5A—C3A—C2A	122.3 (2)	O5B—C3B—C4B	112.1 (2)
O5A—C3A—C4A	113.02 (19)	C2B—C3B—C4B	125.5 (2)
C2A—C3A—C4A	124.7 (2)	C12B—C4B—C3B	116.5 (2)
C12A—C4A—C3A	117.5 (2)	C12B—C4B—C14B	133.2 (2)
C12A—C4A—C14A	133.1 (2)	C3B—C4B—C14B	110.2 (2)
C3A—C4A—C14A	109.32 (19)	O4B—C5B—C6B	123.3 (2)
O4A—C5A—C6A	123.5 (2)	O4B—C5B—C13B	118.1 (2)
O4A—C5A—C13A	118.3 (2)	C6B—C5B—C13B	118.6 (2)
C6A—C5A—C13A	118.2 (2)	C5B—C6B—C7B	120.4 (2)
C5A—C6A—C7A	121.4 (2)	C5B—C6B—H6B	119.8
C5A—C6A—H6A	119.3	C7B—C6B—H6B	119.8
C7A—C6A—H6A	119.3	C8B—C7B—C6B	121.2 (2)
C8A—C7A—C6A	120.2 (2)	C8B—C7B—H7B	119.4
C8A—C7A—H7A	119.9	C6B—C7B—H7B	119.4
C6A—C7A—H7A	119.9	C7B—C8B—C9B	119.6 (2)
C7A—C8A—C9A	120.1 (2)	C7B—C8B—H8B	120.2
C7A—C8A—H8A	119.9	C9B—C8B—H8B	120.2
C9A—C8A—H8A	119.9	C13B—C9B—C8B	119.3 (2)
C8A—C9A—C13A	119.4 (2)	C13B—C9B—C10B	119.1 (2)
C8A—C9A—C10A	121.7 (2)	C8B—C9B—C10B	121.6 (2)
C13A—C9A—C10A	118.9 (2)	O2B—C10B—C11B	122.1 (2)
O2A—C10A—C11A	122.5 (2)	O2B—C10B—C9B	121.5 (2)
O2A—C10A—C9A	121.1 (2)	C11B—C10B—C9B	116.38 (19)
C11A—C10A—C9A	116.32 (19)	C12B—C11B—C1B	118.2 (2)
C12A—C11A—C1A	118.9 (2)	C12B—C11B—C10B	120.4 (2)
C12A—C11A—C10A	120.6 (2)	C1B—C11B—C10B	121.3 (2)
C1A—C11A—C10A	120.4 (2)	O1B—C12B—C4B	116.81 (19)
O1A—C12A—C4A	117.8 (2)	O1B—C12B—C11B	121.63 (19)
O1A—C12A—C11A	121.5 (2)	C4B—C12B—C11B	121.6 (2)
C4A—C12A—C11A	120.7 (2)	O1B—C13B—C9B	123.3 (2)
O1A—C13A—C5A	116.4 (2)	O1B—C13B—C5B	115.90 (19)

O1A—C13A—C9A	122.8 (2)	C9B—C13B—C5B	120.8 (2)
C5A—C13A—C9A	120.8 (2)	C4B—C14B—C17B	110.42 (19)
C4A—C14A—C18A	114.4 (2)	C4B—C14B—C15B	99.73 (19)
C4A—C14A—C17A	109.87 (19)	C17B—C14B—C15B	115.0 (2)
C18A—C14A—C17A	109.4 (2)	C4B—C14B—C18B	114.0 (2)
C4A—C14A—C15A	98.47 (18)	C17B—C14B—C18B	108.6 (2)
C18A—C14A—C15A	110.2 (2)	C15B—C14B—C18B	109.1 (2)
C17A—C14A—C15A	114.2 (2)	C16B—C15B—O5B	106.2 (2)
O5A—C15A—C16A	107.8 (2)	C16B—C15B—C14B	120.1 (2)
O5A—C15A—C14A	106.66 (19)	O5B—C15B—C14B	106.9 (2)
C16A—C15A—C14A	120.1 (2)	C16B—C15B—H15B	107.7
O5A—C15A—H15A	107.2	O5B—C15B—H15B	107.7
C16A—C15A—H15A	107.2	C14B—C15B—H15B	107.7
C14A—C15A—H15A	107.2	O6B—C16B—C15B	115.9 (3)
O6A—C16A—C15A	113.4 (2)	O6B—C16B—H16C	108.7
O6A—C16A—H16A	108.9	C15B—C16B—H16C	110.2
C15A—C16A—H16A	108.9	O6B—C16B—H16D	102.5
O6A—C16A—H16B	108.9	C15B—C16B—H16D	110.3
C15A—C16A—H16B	108.9	H16C—C16B—H16D	108.9
H16A—C16A—H16B	107.7	C14B—C17B—H17D	109.5
C14A—C17A—H17A	109.5	C14B—C17B—H17E	109.5
C14A—C17A—H17B	109.5	H17D—C17B—H17E	109.5
H17A—C17A—H17B	109.5	C14B—C17B—H17F	109.5
C14A—C17A—H17C	109.5	H17D—C17B—H17F	109.5
H17A—C17A—H17C	109.5	H17E—C17B—H17F	109.5
H17B—C17A—H17C	109.5	C14B—C18B—H18D	109.5
C14A—C18A—H18A	109.5	C14B—C18B—H18E	109.5
C14A—C18A—H18B	109.5	H18D—C18B—H18E	109.5
H18A—C18A—H18B	109.5	C14B—C18B—H18F	109.5
C14A—C18A—H18C	109.5	H18D—C18B—H18F	109.5
H18A—C18A—H18C	109.5	H18E—C18B—H18F	109.5
H18B—C18A—H18C	109.5	H1WA—O1WA—H2WA	109.3
C13B—O1B—C12B	118.98 (17)	H1WB—O1WB—H2WB	100.6
O3A—C1A—C2A—C3A	178.9 (2)	O3B—C1B—C2B—C3B	−177.6 (2)
C11A—C1A—C2A—C3A	−2.1 (3)	C11B—C1B—C2B—C3B	2.6 (4)
C15A—O5A—C3A—C2A	−168.9 (2)	C15B—O5B—C3B—C2B	−166.8 (3)
C15A—O5A—C3A—C4A	11.1 (3)	C15B—O5B—C3B—C4B	11.9 (3)
C1A—C2A—C3A—O5A	179.4 (2)	C1B—C2B—C3B—O5B	176.9 (2)
C1A—C2A—C3A—C4A	−0.6 (4)	C1B—C2B—C3B—C4B	−1.6 (4)
O5A—C3A—C4A—C12A	−177.4 (2)	O5B—C3B—C4B—C12B	179.9 (2)
C2A—C3A—C4A—C12A	2.6 (4)	C2B—C3B—C4B—C12B	−1.5 (4)
O5A—C3A—C4A—C14A	5.2 (3)	O5B—C3B—C4B—C14B	2.1 (3)
C2A—C3A—C4A—C14A	−174.8 (2)	C2B—C3B—C4B—C14B	−179.2 (2)
O4A—C5A—C6A—C7A	180.0 (2)	O4B—C5B—C6B—C7B	179.1 (2)
C13A—C5A—C6A—C7A	0.9 (4)	C13B—C5B—C6B—C7B	−1.1 (4)
C5A—C6A—C7A—C8A	0.4 (4)	C5B—C6B—C7B—C8B	−0.9 (4)
C6A—C7A—C8A—C9A	−1.1 (4)	C6B—C7B—C8B—C9B	1.7 (4)
C7A—C8A—C9A—C13A	0.6 (4)	C7B—C8B—C9B—C13B	−0.4 (4)
C7A—C8A—C9A—C10A	−178.5 (2)	C7B—C8B—C9B—C10B	179.5 (2)

C8A—C9A—C10A—O2A	−0.3 (4)	C13B—C9B—C10B—O2B	−178.8 (2)
C13A—C9A—C10A—O2A	−179.4 (2)	C8B—C9B—C10B—O2B	1.3 (4)
C8A—C9A—C10A—C11A	179.5 (2)	C13B—C9B—C10B—C11B	1.0 (3)
C13A—C9A—C10A—C11A	0.4 (3)	C8B—C9B—C10B—C11B	−179.0 (2)
O3A—C1A—C11A—C12A	−178.2 (2)	O3B—C1B—C11B—C12B	179.7 (2)
C2A—C1A—C11A—C12A	2.8 (3)	C2B—C1B—C11B—C12B	−0.5 (3)
O3A—C1A—C11A—C10A	1.4 (3)	O3B—C1B—C11B—C10B	0.9 (3)
C2A—C1A—C11A—C10A	−177.6 (2)	C2B—C1B—C11B—C10B	−179.3 (2)
O2A—C10A—C11A—C12A	179.4 (2)	O2B—C10B—C11B—C12B	−178.7 (2)
C9A—C10A—C11A—C12A	−0.4 (3)	C9B—C10B—C11B—C12B	1.5 (3)
O2A—C10A—C11A—C1A	−0.2 (4)	O2B—C10B—C11B—C1B	0.1 (4)
C9A—C10A—C11A—C1A	180.0 (2)	C9B—C10B—C11B—C1B	−179.7 (2)
C13A—O1A—C12A—C4A	−179.2 (2)	C13B—O1B—C12B—C4B	−176.88 (19)
C13A—O1A—C12A—C11A	0.4 (3)	C13B—O1B—C12B—C11B	2.7 (3)
C3A—C4A—C12A—O1A	177.84 (19)	C3B—C4B—C12B—O1B	−176.83 (19)
C14A—C4A—C12A—O1A	−5.5 (4)	C14B—C4B—C12B—O1B	0.3 (4)
C3A—C4A—C12A—C11A	−1.8 (3)	C3B—C4B—C12B—C11B	3.6 (3)
C14A—C4A—C12A—C11A	174.9 (2)	C14B—C4B—C12B—C11B	−179.2 (2)
C1A—C11A—C12A—O1A	179.6 (2)	C1B—C11B—C12B—O1B	177.8 (2)
C10A—C11A—C12A—O1A	0.0 (3)	C10B—C11B—C12B—O1B	−3.4 (3)
C1A—C11A—C12A—C4A	−0.8 (3)	C1B—C11B—C12B—C4B	−2.7 (3)
C10A—C11A—C12A—C4A	179.6 (2)	C10B—C11B—C12B—C4B	176.1 (2)
C12A—O1A—C13A—C5A	179.80 (19)	C12B—O1B—C13B—C9B	0.0 (3)
C12A—O1A—C13A—C9A	−0.4 (3)	C12B—O1B—C13B—C5B	179.70 (18)
O4A—C5A—C13A—O1A	−0.7 (3)	C8B—C9B—C13B—O1B	178.1 (2)
C6A—C5A—C13A—O1A	178.5 (2)	C10B—C9B—C13B—O1B	−1.8 (3)
O4A—C5A—C13A—C9A	179.5 (2)	C8B—C9B—C13B—C5B	−1.6 (3)
C6A—C5A—C13A—C9A	−1.4 (3)	C10B—C9B—C13B—C5B	178.5 (2)
C8A—C9A—C13A—O1A	−179.2 (2)	O4B—C5B—C13B—O1B	2.4 (3)
C10A—C9A—C13A—O1A	0.0 (3)	C6B—C5B—C13B—O1B	−177.4 (2)
C8A—C9A—C13A—C5A	0.6 (3)	O4B—C5B—C13B—C9B	−177.9 (2)
C10A—C9A—C13A—C5A	179.8 (2)	C6B—C5B—C13B—C9B	2.3 (3)
C12A—C4A—C14A—C18A	48.8 (4)	C12B—C4B—C14B—C17B	−70.3 (3)
C3A—C4A—C14A—C18A	−134.4 (2)	C3B—C4B—C14B—C17B	106.9 (2)
C12A—C4A—C14A—C17A	−74.8 (3)	C12B—C4B—C14B—C15B	168.3 (3)
C3A—C4A—C14A—C17A	102.1 (2)	C3B—C4B—C14B—C15B	−14.4 (3)
C12A—C4A—C14A—C15A	165.6 (3)	C12B—C4B—C14B—C18B	52.2 (4)
C3A—C4A—C14A—C15A	−17.5 (2)	C3B—C4B—C14B—C18B	−130.5 (3)
C3A—O5A—C15A—C16A	−152.6 (2)	C3B—O5B—C15B—C16B	−150.5 (2)
C3A—O5A—C15A—C14A	−22.4 (2)	C3B—O5B—C15B—C14B	−21.1 (3)
C4A—C14A—C15A—O5A	23.6 (2)	C4B—C14B—C15B—C16B	141.8 (2)
C18A—C14A—C15A—O5A	143.6 (2)	C17B—C14B—C15B—C16B	23.8 (3)
C17A—C14A—C15A—O5A	−92.7 (2)	C18B—C14B—C15B—C16B	−98.5 (3)
C4A—C14A—C15A—C16A	146.4 (2)	C4B—C14B—C15B—O5B	20.9 (3)
C18A—C14A—C15A—C16A	−93.5 (3)	C17B—C14B—C15B—O5B	−97.2 (3)
C17A—C14A—C15A—C16A	30.1 (3)	C18B—C14B—C15B—O5B	140.6 (2)
O5A—C15A—C16A—O6A	48.9 (3)	O5B—C15B—C16B—O6B	−73.8 (3)
C14A—C15A—C16A—O6A	−73.3 (3)	C14B—C15B—C16B—O6B	165.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3A—H3OA···O2A	0.98	1.62	2.530 (3)	152
O4A—H4OA···O1WA	0.92	1.75	2.672 (3)	175
O6A—H6OA···O4B^{i}	0.82	2.12	2.918 (3)	165
O3B—H3OB···O2B	1.06	1.57	2.529 (3)	148
O4B—H4OB···O1WB	1.02	1.62	2.639 (3)	175
O6B—H6OB···O4A^{ii}	0.82	2.25	3.059 (4)	167
O1WA—H1WA···O3A^{iii}	0.83	2.06	2.889 (3)	173
O1WA—H2WA···O6B	0.92	1.84	2.737 (5)	165
O1WB—H1WB···O6A	0.89	1.86	2.691 (3)	153
O1WB—H2WB···O3B^{iv}	0.82	2.06	2.868 (3)	164
C16B—H16C···O2B^{v}	0.96	2.46	3.389 (3)	163

Symmetry codes: (i) *x*−1/2, −*y*+3/2, −*z*+1; (ii) *x*+1/2, −*y*+1/2, −*z*+1; (iii) −*x*+1, *y*−1/2, −*z*+3/2; (iv) −*x*+1, *y*+1/2, −*z*+1/2; (v) −*x*+3/2, −*y*+1, *z*+1/2.

Fig. 1

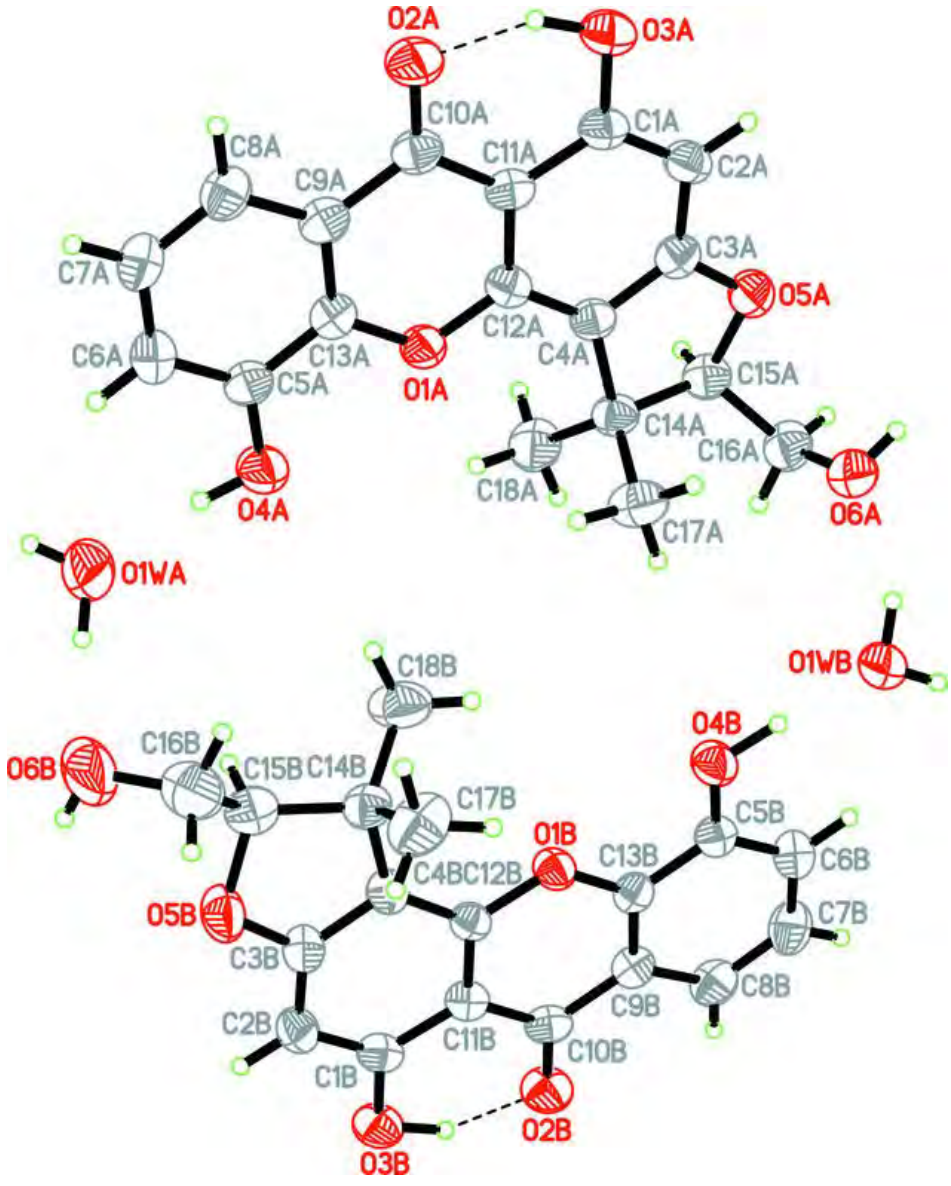
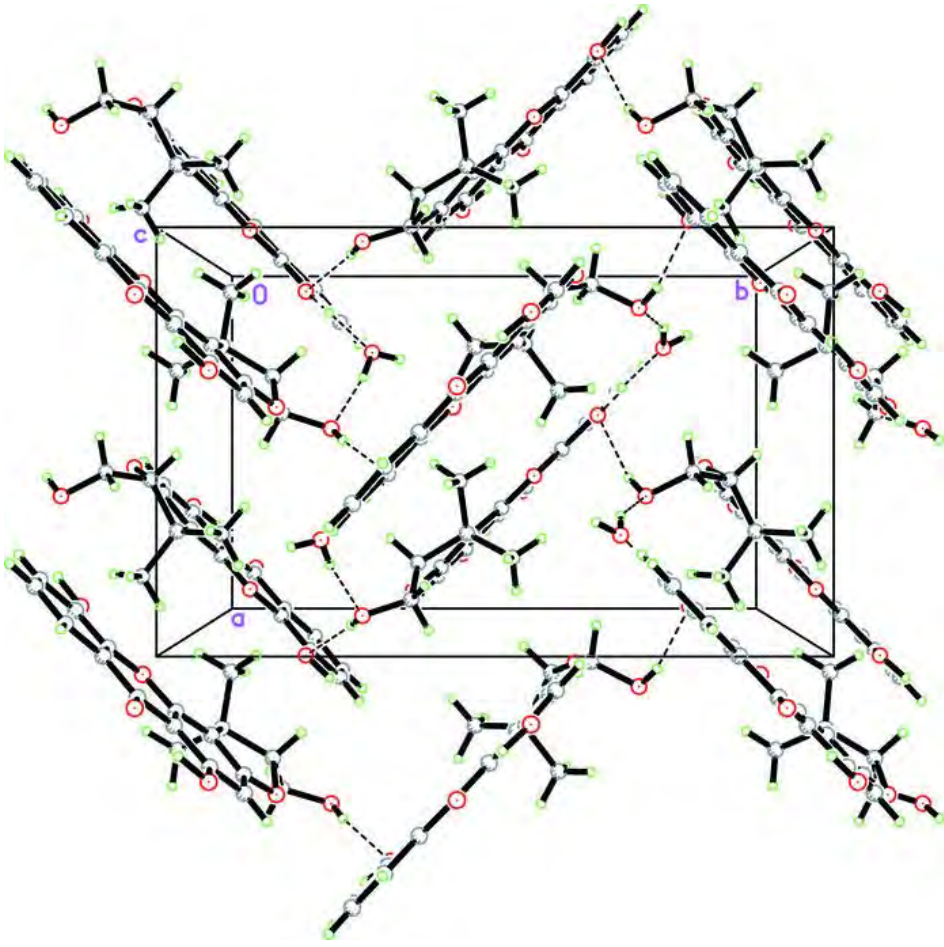


Fig. 2



VERSITA

Chemical Papers 65 (6) 890–897 (2011)
DOI: 10.2478/s11696-011-0084-4

ORIGINAL PAPER

Influence of trimethoxy-substituted positions on fluorescence of heteroaryl chalcone derivatives

[a]Thitipone Suwunwong, [a]Suchada Chantrapromma*, [b]Hoong-Kun Fun

[a]*Department of Chemistry and Centre of Excellence for Innovation in Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand*

[b]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia*

Received 21 April 2011; Revised 6 June 2011; Accepted 15 June 2011

Three series of heteroaryl chalcones, (*E*)-1-(2-pyridyl)-3-(X)prop-2-en-1-one (*Ia–Ic*), (*E*)-1-(2-thienyl)-3-(X)prop-2-en-1-one (*IIa–IIc*), and (*E*)-1-(2-furyl)-3-(X)prop-2-en-1-one (*IIIa–IIIc*), where X = 2,4,5-trimethoxyphenyl (for series *a*), X = 2,4,6-trimethoxyphenyl (for series *b*), and X = 3,4,5-trimethoxyphenyl (for series *c*) were synthesised using basic catalysed aldol condensation and characterised using ^{1}H NMR and FT-IR spectroscopies. Compound *IIa* was also characterised by single crystal X-ray analysis. The absorption and fluorescence emission spectra of these compounds revealed that the absorption and fluorescence depended on the heterocycle rings and trimethoxy-substituted phenyl rings linked to the enone system. The position of methoxy groups substantially affected the fluorescent properties. Compounds *Ia–IIIa* containing the 2,4,5-trimethoxyphenyl moiety exhibited the red-shift phenomenon and strong emission fluorescence.

Keywords: heteroaryl chalcones, fluorescence, aldol condensation, X-ray crystallography, trimethoxyphenyl

Introduction

Chalcones (1,3-diphenyl-2-propen-1-one derivatives) (Schlangen et al., 2009; Sivakumar et al., 2009; Sun & Cui, 2008) were synthesised using the base-catalysed Claisen–Schmidt condensation reaction or the aldol condensation reaction (Navarini et al., 2009). They have many applications, involving biological activities such as antibacterial (Nielsen et al., 2004), antifungal (Batovska et al., 2007; López et al., 2001), anti-inflammatory (Won et al., 2005), and anti-tumour (Romagnoli et al., 2008) properties, as well as non-linear optical (NLO) (Crasta et al., 2004) and electroactive fluorescent properties (Jung et al., 2008). Chalcones with the latter properties were used as fluorescent dyes (Jung et al., 2008), light-emitting diodes (LEDs) (Fayed & Awad, 2004), fluorescent probes (Xu et al., 2005), and fluorescent sensors (Niu et al., 2006). The previous studies revealed that the heterocyclic part in the fluorophore consistently increased the fluorescent property of the compounds and there have been many reports on their fluorescent properties (Knyazhansky et al., 1998; Sun et al., 2005). Our study was focused on the influence of the different positions of substituents on some heteroaryl chalcones, specifically pyridyl, thienyl, and furyl chalcones. In addition, it has been found that electron-donating substituted groups in the π-conjugate system can enhance the fluorescent properties of heteroaryl chalcones (Musil et al., 2007). It is known that the OCH_3 functional group is mostly found in natural products and that these compounds exhibited interesting applications not only as far as their bioactivities (Salem & Werbovetz, 2005; Sivakumar et al., 2010; Zangade et al., 2010) but also where their fluorescent properties (Hirano et al., 2004; Maiti et al., 1999) are concerned. Numerous applications of fluorescent materials and the fluorescence capability of the above compounds led us to synthesise nine small molecules of heteroaryl chalcones (*Ia–Ic*, *IIa–IIc*, and *IIIa–IIIc*) containing trimethoxyphenyl

*Corresponding author, e-mail: suchada.c@psu.ac.th

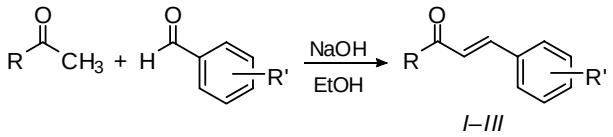


Fig. 1. Synthesis of heteroaryl chalcones *I–III*.

moiety located in various positions of the benzene rings (Fig. 1) with both potential bioactivity and fluorescent properties. Attempts have been made to synthesise and elucidate the effects of structure and the positions substituted on the absorption and fluorescence properties of this class of compounds.

Experimental

Ethanol, acetone, and chloroform were purchased from Merck Ltd. (Thailand) and were used as received. Sodium hydroxide was purchased from Lab-Scan Analytical Sciences, Lab-Scan Asia. Co. (Thailand). 2-Acetylpyridine, 2-acethylthiophene, 2-acetylfuran, and all trimethoxybenzaldehydes were obtained from Fluka (Sigma–Aldrich Chemical Co., Thailand) and were used without purification. IR spectra (KBr pellets) were recorded in the range of 4000–400 cm^{-1} using a Perkin–Elmer FT-IR System Spectrum BX spectrophotometer. ^1H NMR spectra (300 MHz) were recorded on a Bruker Ultra Shield^{TM}, using $CDCl_3$ as solvent and tetramethylsilane as the internal standard. UV-VIS spectra were recorded on a SPECORD S100 (Analytik Jena AG, Germany). Fluorescence emission spectra were recorded on a Perkin–Elmer LS 55 luminescence spectrometer at the ambient temperature.

Synthesis of pyridyl chalcones (Ia–Ic), thienyl chalcones (IIa–IIc), and furyl chalcones (IIIa–IIIc)

A mixture of ethanol (10 mL), 2-acetylpyridine (0.20 mL, 2 mmol), the solution of the respective trimethoxybenzaldehyde (2,4,5-trimethoxyphenyl for *Ia*, 2,4,6-trimethoxyphenyl for *Ib*, and 3,4,5-trimethoxyphenyl for *Ic*) (0.40 g, 2 mmol) in ethanol (20 mL), and 30 % aqueous NaOH (5 mL) was stirred in an ice bath at 5 ℃ for about 4 h. The resulting solid was collected by filtration, washed with distilled water and dried to give an amorphous yellow-coloured solid. This solid was dissolved in 80 mL of hot acetone. The hot solution was filtered and allowed to cool to room temperature. The yellow-coloured microcrystals were collected by filtration. A second recrystallisation from hot acetone gave 0.37 g (1.20 mmol, 61 %) of *Ia*, 0.42 g (1.40 mmol, 70 %) of *Ib*, and 0.40 g (1.32 mmol, 66 %) of *Ic*. Pyridyl chalcones (*Ia–Ic*) were kept under N_2 atmosphere in order to prevent their oxidation.

Thienyl chalcones (*IIa–IIc*) were synthesised in a similar manner, using a mixture of ethanol (10 mL), 2-acetylthiophene (0.35 mL, 2 mmol), a solution of the respective trimethoxybenzaldehyde (2,4,5-trimethoxyphenyl for *IIa*, 2,4,6-trimethoxyphenyl for *IIb*, and 3,4,5-trimethoxyphenyl for *IIc*) (0.40 g, 2 mmol) in ethanol (20 mL), and 30 % aqueous NaOH (5 mL) at 5 ℃ for about 3 h. A second recrystallisation from hot acetone gave 0.45 g (1.48 mmol, 74 %) of *IIa*, 0.54 g (1.77 mmol, 89 %) of *IIb*, and 0.44 g (1.44 mmol, 72 %) of *IIc*.

Analogously, furyl chalcones (*IIIa–IIIc*) were prepared from ethanol (15 mL), 2-acetylfuran (0.22 g, 2 mmol), a solution of the respective trimethoxybenzaldehyde (2,4,5-trimethoxyphenyl for the synthesis of *IIIa*, 2,4,6-trimethoxyphenyl for *IIIb*, and 3,4,5-trimethoxyphenyl for *IIIc*) (0.40 g, 2 mmol) in ethanol (15 mL), and 20 % aqueous NaOH (5 mL). The mixture was allowed to react at 5 ℃ for about 5 h. A second recrystallisation from hot acetone gave 0.37 g (1.27 mmol, 64 %) of *IIIa*, 0.44 g (1.51 mmol, 76 %) of *IIIb*, and 0.44 g (1.51 mmol, 76 %) of *IIIc*.

Results and discussion

The heteroaryl chalcones were synthesised in high yields (more than 60 %) by aldol condensation (Fig. 1). The reaction time and the substituent R (pyridyl, thienyl, and furyl) in the starting methyl ketone play important roles in the product formation. The yields and some physico-chemical properties of all nine heteroaryl chalcones (photochemically stable compounds) are given in Table 1.

The IR spectra of compounds *I–III* revealed the stretching vibrations of aromatic C—H bonds at about 3100–3000 cm^{-1} and C—H bonds of OCH_3 at $\sim$ 2900 cm^{-1}. The strong peak of stretching vibrations of C═O bonds was observed at 1700–1600 cm^{-1} and stretching vibrations of aromatic C═C bonds were observed at 1515–1610 cm^{-1} (Table 2). In the ^1H NMR spectra, two signals of *trans*-arranged hydrogen atoms at δ 7.32–8.42 with coupling constant J = 15.6–15.9 Hz, were observed. The spectra of 2,4,5-trimethoxyphenyl derivatives showed three singlet signals of three OCH_3 in *ortho*, *para*, and *meta* positions at δ 3.95–3.91. As for 2,4,6-trimethoxyphenyl derivatives, the spectra showed singlet peak of two equivalent OCH_3 in *ortho* positions at δ 3.94–3.91 and one peak of OCH_3 in *para* position at δ 3.88–3.86. Finally, the spectra of 3,4,5-trimethoxyphenyl derivatives exhibited singlet peak of two equivalent OCH_3 in *meta* positions at δ 3.92–3.91 and one peak of OCH_3 in *para* position at δ 3.95–3.93 (Table 2).

The UV-VIS spectra of the heteroaryl chalcones exhibit characteristic K and R bands. The absorbance bands in the UV region of 200–300 nm are considered to be the allowed $\pi \rightarrow \pi^*$ transitions of the aromatic system and the forbidden $n \rightarrow \pi^*$ transitions of carbonyl and heterocyclic part (K band) and another band in the region of 300–470 nm is the allowed $\pi \rightarrow \pi^*$

Table 1. Characteristics of the compounds prepared[a]

Compound	R	R′	Yield / %	M.p. / °C	Appearance
Ia	2-Pyridyl	2,4,5-OCH_3	61	155–156	Yellow
Ib		2,4,6-OCH_3	70	119–120	Pale yellow
Ic		3,4,5-OCH_3	66	161–162	Pale yellow
IIa	2-Thienyl	2,4,5-OCH_3	74	128–129	Yellow
IIb		2,4,6-OCH_3	89	108–109	Pale yellow
IIc		3,4,5-OCH_3	72	147–148	Pale yellow
IIIa	2-Furyl	2,4,5-OCH_3	64	83–84	Yellow
IIIb		2,4,6-OCH_3	76	117–118	Pale yellow
IIIc		3,4,5-OCH_3	76	117–118	Pale yellow

a) Apart from *Ia*, all the compounds have already been described in the literature. For *Ib*, see Cordaro et al. (2002), Suzuki et al. (1995a, 1995b); for *Ic*, see Prasad et al. (2007a, 2008); for *IIa*, see Fun et al. (2008); for *IIb*, see Lavrushin et al. (1961, 1962), Tsukerman et al. (1967, 1969); for *IIc*, see Dudeja et al. (1993), Katiyar et al. (1974), Lawrence et al. (2000), Roman (2004), Prasad et al. (2007b), Lin et al. (2007), Lin and Chuang (2007), Suwunwong et al. (2009), Ramesh et al. (2009a, 2009b, 2010), Dulawat et al. (2010), Ramesh and Rao (2010); for *IIIa*, see Nepali et al. (2011); for *IIIb*, see Fun et al. (2010a), Werts et al. (2005); for *IIIc*, see Fun et al. (2010b), Nepali et al. (2011), Singhal and Mishra (1985), Singhal and Misra (1986).

Table 2. Spectral characterisation of heteroaryl chalcones *I–III*

Compound	Spectral data[a]
Ia	IR, $\tilde{\nu}$/cm^{-1}: 3081 (m, CH_{ar}), 2937–2960 (s, CH_3), 1638 (s, C═O), 1576–1591 (s, C═C), 1272–1317 (s, C—N), 1194–1208 (s, C—O) ^{1}H NMR ($CDCl_3$), δ: 8.76 (dd, 1H, J = 0.6 Hz, J = 4.8 Hz, H_{pyr}), 8.26 (d, 1H, J = 15.9 Hz, H_{trans}), 8.14 (dd, 1H, J = 0.9 Hz, J = 6.6 Hz, H_{pyr}), 8.11 (d, 1H, J = 15.9 Hz, H_{trans}), 7.92 (dt, 1H, J = 1.8 Hz, J = 7.8 Hz, H_{pyr}), 7.54 (dt, 2H, J = 0.9 Hz, J = 4.8 Hz, H_{pyr}), 7.28–6.59 (s, 2H, H_{ar}), 3.95–3.91 (s, 9H, OCH_3)
Ib	IR, $\tilde{\nu}$/cm^{-1}: 2972 (m, CH_{ar}), 1669 (s, C═O), 1576 (s, C═C), 1200 (s, C—N), 1123 (s, C—O), 810 (w, C—H) ^{1}H NMR ($CDCl_3$), δ: 8.73 (d, 1H, J = 4.5 Hz, H_{pyr}), 8.42–8.31 (d, 2H, J = 16.2 Hz, H_{trans}), 8.10 (d, 1H, J = 7.8 Hz, H_{pyr}), 7.89–7.49 (dt, 2H, J = 1.5 Hz, J = 7.8 Hz, H_{pyr}), 6.18 (s, 2H, H_{ar}), 3.95–3.88 (9H, s, OCH_3)
Ic	IR, $\tilde{\nu}$/cm^{-1}: 2998–2976 (m, CH_{ar}), 2943 (s, CH_3), 1668 (s, C═O), 1610–1579 (s, C═C), 1322–1290 (s, C—N), 1125 (s, C—O) ^{1}H NMR ($CDCl_3$), δ: 8.76 (dd, 1H, J = 0.9 Hz, J = 4.8 Hz, H_{pyr}), 8.20 (dd, 1H, J = 0.9 Hz, J = 6.9 Hz, H_{pyr}), 8.18 (d, 1H, J = 15.9 Hz, H_{trans}), 7.90 (dt, 1H, J = 1.8 Hz, J = 7.5 Hz, H_{pyr}), 7.88 (d, 1H, J = 15.9 Hz, H_{trans}), 7.50 (dt, 1H, J = 0.9 Hz, J = 4.8 Hz, H_{pyr}), 6.96 (s, 2H, H_{ar}), 3.94–3.91 (9H, s, OCH_3)
IIa	IR, $\tilde{\nu}$/cm^{-1}: 3107 (m, CH_{ar}), 2932 (s, CH_3) 1638–1611 (s, C═O), 1583–1509 (s, C═C), 1206 (s, C—O) ^{1}H NMR ($CDCl_3$), δ: 8.12 (d, 1H, J = 15.6 Hz, H_{trans}), 7.86 (dd, 1H, J = 0.9 Hz, J = 3.9 Hz, H_{th}), 7.65 (dd, 1H, J = 0.9 Hz, J = 4.8 Hz, H_{th}), 7.39 (d, 1H, J = 15.6 Hz, H_{trans}), 7.17 (t, 1H, J = 3.9 Hz, H_{th}), 7.12–6.53 (2H, s, H_{ar}), 3.95–3.91 (9H, s, OCH_3)
IIb	IR, $\tilde{\nu}$/cm^{-1}: 2937 (m, CH_{ar}), 1638 (s, C═O), 1610–1570 (s, C═C), 1204 (s, C—O), 1124 (s, C—S) ^{1}H NMR ($CDCl_3$), δ: 8.28 (d, 1H, J = 15.6, H_{trans}), 7.81 (d, 1H, J = 5.1 Hz, H_{th}), 7.79 (d, 1H, J = 15.6, H_{trans}), 7.60 (d, 1H, J = 5.1 Hz, H_{th}), 7.15 (t, 1H, J = 5.1 Hz, H_{th}), 6.14 (s, 2H, H_{ar}), 3.92–3.86 (9H, s, OCH_3).
IIc	IR, $\tilde{\nu}$/cm^{-1}: 3086–2975 (m, CH_{ar}), 2943 (s, CH_3), 1646 (s, C═O), 1598–1579 (s, C═C), 1125 (s, C—O), 1004–979 (s, C—H) ^{1}H NMR ($CDCl_3$), δ: 7.90 (dd, 1H, J = 0.9 Hz, J = 3.9 Hz, H_{th}), 7.79 (d, 1H, J = 15.6 Hz, H_{trans}), 7.70 (dd, 1H, J = 0.9 Hz, J = 3.9 Hz, H_{th}), 7.32 (d, 1H, J = 15.6 Hz, H_{trans}), 7.21 (t, 1H, J = 3.9 Hz, H_{th}), 6.88 (s, 2H, H_{ar}), 3.95–3.92 (9H, s, OCH_3)
IIIa	IR, $\tilde{\nu}$/cm^{-1}: 3404 (m, CH_{ar}), 2985 (s, CH_3), 1642 (s, C═O), 1585–1515 (s, C═C), 1217 (s, C—O), 1040–1026 (s, C—H) ^{1}H NMR ($CDCl_3$), δ: 8.19 (d, 1H, J = 15.6 Hz, H_{trans}), 7.64 (d, 1H, J = 3.9 Hz, H_{fur}), 7.39 (d, 1H, J = 15.6 Hz, H_{trans}), 7.30 (d, 1H, J = 3.9 Hz, H_{fur}), 7.14–6.53 (s, 2H, H_{ar}), 6.58 (t, 1H, J = 3.9 Hz, H_{fur}), 3.95–3.92 (s, 9H, OCH_3)
IIIb	IR, $\tilde{\nu}$/cm^{-1}: 3001–2977 (m, CH_{ar}), 2946 (s, CH_3), 1654 (s, C═O), 1582 (s, C═C), 1123 (s, C—O), 1064–1011 (s, C—H) ^{1}H NMR ($CDCl_3$), δ: 8.24 (d, 1H, J = 15.6 Hz, H_{trans}), 7.74 (d, 1H, J = 15.9 Hz, H_{trans}), 7.67 (d, 1H, J = 3.9 Hz, H_{fur}), 7.25 (d, 1H, J = 3.9 Hz, H_{fur}), 6.60 (t, 1H, J = 1.8Hz, H_{fur}), 6.17 (s, 2H, H_{ar}), 3.94–3.88 (s, 9H, OCH_3)
IIIc	IR, $\tilde{\nu}$/cm^{-1}: 3001–2977 (m, CH_{ar}), 2946 (s, CH_3), 1654 (s, C═O), 1582 (s, C═C), 1123 (s, C—O), 1064–1011 (s, C—H) ^{1}H NMR ($CDCl_3$), δ: 8.24 (d, 1H, J = 15.6 Hz, H_{trans}), 7.74 (d, 1H, J = 15.9 Hz, H_{trans}), 7.67 (d, 1H, J = 3.9 Hz, H_{fur}), 7.25 (d, 1H, J = 3.9 Hz, H_{fur}), 6.60 (t, 1H, J = 1.8Hz, H_{fur}), 6.17 (s, 2H, H_{ar}), 3.94–3.88 (s, 9H, OCH_3)

a) The IR bands are denoted as m – medium; s – strong; aromatic moieties are denoted as ar – aryl, fur – furyl, th – thienyl.

Table 3. Absorption maxima (λ_{max}) and molar absorptivity (ε) of chalcones *I–III* (5 μM in chloroform)

Compound	K band		R band	
	λ_{max}/nm	ε/(m^2 mol^{-1})	λ_{max}/nm	ε/(m^2 mol^{-1})
Ia	276	5.8508	403	2.4828
Ib	248	1.3540	372	1.2800
	263	1.3520		
Ic	248	1.9600	355	2.3420
IIa	273	3.8800	395	2.3600
IIb	267	1.4320	368	0.6580
IIc	264	1.1240	350	1.5960
IIIa	245	0.6096	396	0.7984
	312	0.5740		
IIIb	260	1.6900	365	1.5343
IIIc	244	1.7000	352	1.3020

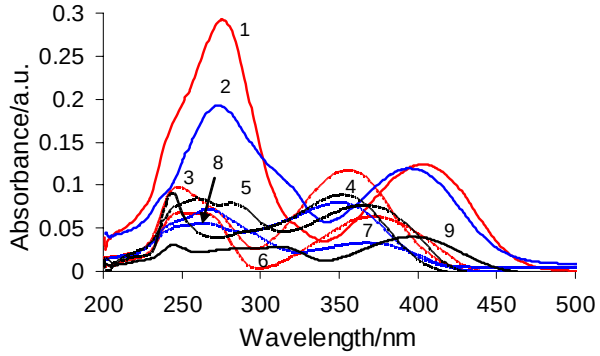


Fig. 2. UV-VIS absorption spectra of heteroaryl chalcones *I–III* (5 μM in chloroform): *Ia* (1), *IIa* (2), *Ic* (3), *IIIc* (4), *IIIb* (5), *Ib* (6), *IIb* (7), *IIc* (8), *IIIa* (9).

Table 4. Fluorescence spectral data (excitation (λ_{ex}), absorption (λ_{abs}), and emission (λ_{em}) maxima, fluorescent quantum yield (Φ_F))^{a,b} and Stokes shift of heteroaryl chalcones *I–III* (5 μM in chloroform)

Compound	λ_{ex}/nm	λ_{abs}/nm	λ_{em}/nm	Φ_F	Stokes shift/nm
Ia	401	403	530	0.18	127
Ib	350	372	453	< 0.01	81
Ic	377	355	502	0.05	147
IIa	394	395	504	0.28	109
IIb	354	368	433	< 0.01	65
IIc	374	350	485	0.06	135
IIIa	394	396	501	0.20	105
IIIb	352	365	431	< 0.01	66
IIIc	372	352	481	0.08	129

a) The emission wavelength was set at 485 nm for excitation spectra study and the excitation wavelength was set at 380 nm for emission spectra study; *b*) coumarin 1 (in ethanol, Φ_F = 0.73) was used as the standard for Φ_F measurements.

transition (R band) with intra-molecular charge transfer (ICT) to the enone system (Zhang et al., 2008; Gaber et al., 2008) as shown in Fig. 2. The molar absorptivity (ε) given in Table 3 was used to compare the spectra of different compounds because it was used to consider an intrinsic property of species (Percino et al., 2011). It was found that the K band of compound *Ia* displayed a hyperchromic shift. Moreover, the strongest absorbance of the ICT band (R band) was also observed in *Ia*. The absorption maxima (λ_{max}) of the ICT band of pyridyl chalcones (*Ia–Ic*) present small red-shift phenomena around 4–10 nm when compared with those of thienyl (*IIa–IIc*) and furyl chalcones (*IIIa–IIIc*) (Table 3, Fig. 2). These are attributed to the capacity of the pyridine moiety to be more aromatic than that of furan and thiophene. Heteroaryl chalcones *Ia–IIIa* with 2,4,5-trimethoxyphenyl chromophore (OCH_3 groups located in *ortho*, *meta*, and *para* positions) exhibit λ_{max} of the ICT transition which is shifted to the longer wavelength (red-shift) by ca 30 nm when compared with those containing 2,4,6-trimethoxyphenyl (OCH_3 groups located in *ortho*, *ortho*, and *para* positions) and by ca 50 nm when compared with those containing 3,4,5-trimethoxyphenyl (OCH_3 groups located in *meta*, *meta*, and *para* positions), as shown in Table 3. These shifts can be interpreted by the capability of electrons from the auxochrome OCH_3 group in the *ortho* and *para* positions of the enone system to yield a more efficient resonance effect than the electrons in the *meta* position. With 2,4,6-trimethoxyphenyl moiety, π-conjugated delocalisation was obstructed by the steric effect of two OCH_3 groups in *ortho* positions.

The absorption spectra of the ICT band (Fig. 2) of all heteroaryl chalcones exhibited λ_{max} in the range of 300–470 nm. The maximum absorbance was observed for compound *Ia*. As a consequence, the excitation wavelengths were selected and set at 380 nm in order to study their emission spectra. Fig. 3 presents the fluorescence emission spectra of heteroaryl chalcones *I–III* in chloroform solution (5 μM). The maximum emission was observed for compound *IIa*. The comparison of the emission of these compounds can be interpreted as that the emission maximum wavelength (λ_{em}) of pyridyl chalcones exhibits red-shift

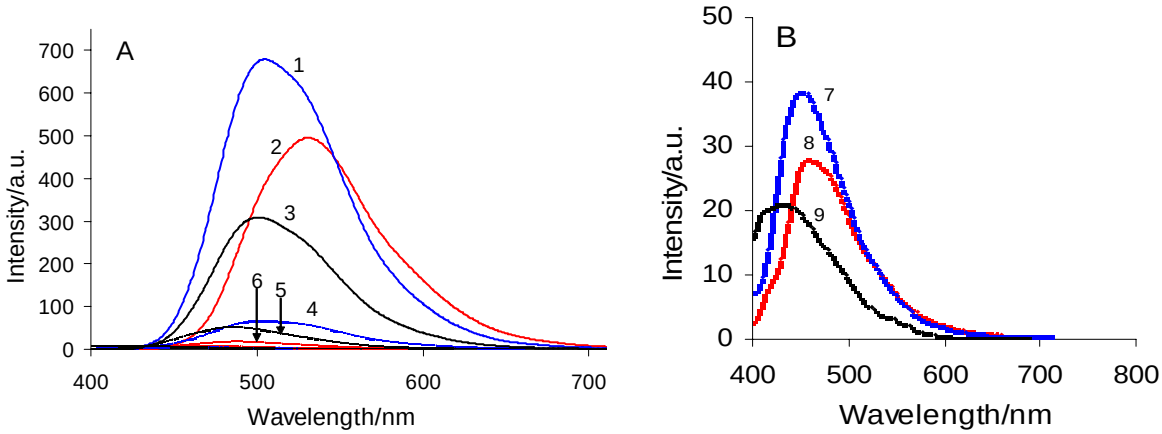


Fig. 3. Fluorescence emission spectra of heteroaryl chalcones (5 μM in chloroform) excited at 380 nm at room temperature: compounds *IIa* (1), *Ia* (2), *IIIa* (3), *IIc* (4), *IIIc* (5), *Ic* (6) using slit width 4.5 (A) and compounds *IIb* (7), *Ib* (8), *IIIb* (9) using slit width 10 (B).

around 15–30 nm when compared with that of thienyl and furyl chalcones (Table 4). This corresponds to the red-shift in absorption spectra caused by the pyridine moiety with the most distinctive aromaticity. However, the λ_{em} of thienyl and furyl chalcones having the same R′ group are almost comparable. The electron-donating methoxy groups in different positions also play an important role on the fluorescent properties of these heteroaryl chalcones. Compounds *Ia*–*IIIa* which contain the 2,4,5-trimethoxyphenyl moiety exhibit strong fluorescence emissions in comparison with other compounds which have methoxy substituents in different positions. It can be seen that the λ_{em} of *Ia*–*IIIa* were red-shifted by 70 nm and 20 nm as compared with the λ_{em} of *Ib*–*IIIb* and *Ic*–*IIIc*, respectively (Fig. 3, Table 4). The shifts were caused by the electron-donating OCH_3 in *ortho* and *meta* positions which increased their fluorescence emission intensity and λ_{em}. By contrast, heteroaryl chalcones containing 2,4,6-trimethoxyphenyl exhibited the lowest emission intensity (in the decreasing order *IIIb*, *Ib*, *IIb*), and λ_{em} which were caused by the steric effect of the two OCH_3 groups in *ortho* position which decreased the electron delocalisation of heteroaryl chalcones. A previous work (Fahrni et al., 2003) concludes that Stokes shift is usually associated with the attached substituent groups and it is enhanced in compounds with a strong electron-donating group, as was also indicated in this work. It was found that the compounds containing 3,4,5-trimethoxyphenyl (*Ic*–*IIIc*) showed the highest Stokes shift values (Table 4). The fluorescent quantum yields of heteroaryl chalcones *I*–*III* in chloroform using coumarin 1 (Φ_F = 0.73 in EtOH) as the standard sample are presented in Table 4. The following equation was used to calculate the fluorescent quantum yield (Φ) of each compound:

$$\Phi_X = \Phi_{ST}\left(\frac{S_X}{S_{ST}}\right)\left(\frac{A_{ST}}{A_X}\right)\left(\frac{n_X^2}{n_{ST}^2}\right) \qquad (1)$$

where subscripts ST and X denote the standard and test respectively, S is the integrated emission band area, A is the absorbance at the excited wavelength and n is the solvent refractive index (Rhys Williams et al., 1983). It was found that thienyl chalcones showed higher values of Φ_F in comparison with those of furyl and pyridyl chalcones (Table 4). However, the Φ_F values of these trimethoxy-substituted heteroaryl chalcones are lower than those of the dimethylamino-substituted heteroaryl chalcones (Gaber et al., 2008). It is important to note that thiophene moiety is a better electron-donor than furan and pyridine and it is also a good fluorophore in the molecule, exhibiting fluorescence even in diluted solutions (Sun & Cui, 2009). Clearly, compounds *Ia*–*IIIa* showed higher values of Φ_F compared with *Ib*–*IIIb* and *Ic*–*IIIc*.

The crystal structures of heteroaryl chalcones *IIc* (Suwunwong et al., 2009), *IIIb* (Fun et al., 2010a), and *IIIc* (Fun et al., 2010b) have already been reported. Crystallographic data of *IIa* were collected using the Oxford Cryosystem Cobra low-temperature attached to a Bruker Apex2 CCD diffractometer with a graphite monochromated Mo K_α radiation at a detector distance of 5 cm using APEX2 software (Bruker AXS, 2005). The data were reduced using the Bruker SAINT program (Siemens AXS, 1998) and the empirical absorption corrections were performed using the SADABS program (Sheldrick, 2003). The structures were solved by direct methods and refined by least-squares using the Bruker SHELXTL software package (Bruker AXS, 2001). All non-hydrogen atoms were refined anisotropically. The hydroxyl H atoms were located from the difference maps and were isotropically refined. The remaining H atoms were placed in calculated positions C—H distances in the range of 0.93–0.98 Å after examining their positions in the difference map. The U_{iso} values were constrained as $1.5U_{eq}$ of the carrier atoms for methyl H atoms and $1.2U_{eq}$ for hydroxyl and the other remaining H atoms. The final

Table 5. Crystallographic data[a] and structure refinement for compound *IIa*

Empirical formula	$C_{20}H_{34}O_2$
Formula mass	306.47
Temperature, T (K)	100(1)
Crystal system, space group	Monoclinic, $P2_1/c$
a (Å)	7.4877(2)
b (Å)	7.9781(2)
c (Å)	24.3557(6)
α (°)	90
β (°)	96.838(2)
γ (°)	90
Unit-cell volume, V (Å^3)	1444.60(6)
Formula per unit cell, Z	4
Density, D_{calcd} (g cm^{-3})	1.399
Absorption coefficient, μ (mm^{-1})	0.237
Crystal size (mm)	0.56 × 0.34 × 0.25
Reflections collected/unique	17551/4502
Final R indices	$R_1 = 0.0677$, w$R_2 = 0.1454$

a) Standard deviations in parentheses.

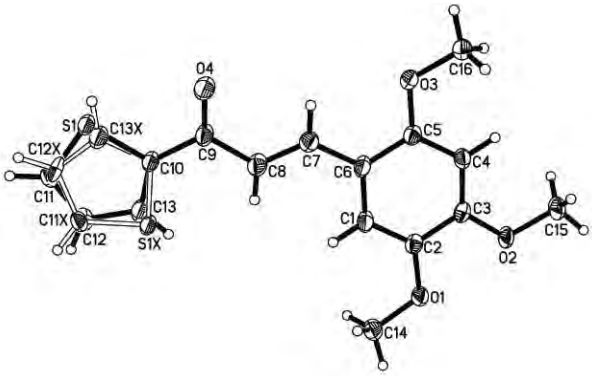


Fig. 4. ORTEP plot of compound *IIa*.

refinement converged well. Materials for publication were prepared using SHELXTL (Bruker AXS, 2001) and PLATON (Spek, 2003) software. The molecular structure of compound *IIa* is shown in Fig. 4. The basic crystallographic data are summarised in Table 5.

The molecule is slightly twisted and the thiophene ring is disordered over two positions. The dihedral angles between the thiophene rings, (S1/C10—C13) and (S1X/C10/C11X—C13X), with the 2,4,5-trimethoxyphenyl ring are 13.7(2)° and 10.0(17)°, respectively. The three methoxy groups have two different conformations, two are co-planar (torsion angles C14—O1—C2—C1 = −1.4(3)° and C15—O2—C3—C4 = 2.8(3)°) and one is slightly twisted (torsion angle C16—O3—C5—C4 = −16.7(3)°) with the attached benzene ring.

Supplementary data

Crystallographic data for the structure reported in this article have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 739791. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: 0044-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

Conclusions

The series of heteroaryl chalcones containing three methoxy groups (2,4,5-, 2,4,6-, and 3,4,5-trimethoxy derivatives) were synthesised by the basic catalysed aldol condensation. Their structures were determined by IR and ^{1}H NMR spectra. By way of representation, the crystal structure of compound *IIa* was determined by X-ray diffraction analysis. The results revealed that heterocyclic groups had a significant effect on the intensities of the emission spectra. In this respect, thienyl is a better fluorophore than the furyl and pyridyl moieties. It was also found that 2,4,5-trimethoxy derivatives exhibited the most intense fluorescence spectra in chloroform solution. Compounds *Ia*, *IIa*, and *IIIa* showed intense fluorescence spectra and emitted green-yellow light at 530 nm, 504 nm, and 501 nm, respectively. Heteroaryl chalcones with trimethoxyphenyl moiety in *ortho*, *meta*, and *para* positions can exhibit strong fluorescent properties as indicated by the fluorescent quantum yield values of *Ia*, *IIa*, and *IIIa* (0.18, 0.28, and 0.20, respectively) which are higher than those of the other compounds in the study.

Acknowledgements. The authors thank the Centre of Excellence for Innovation in Chemistry (PERCH-CIC), Commission on Higher Education, Ministry of Education, Thailand for financial support. The authors also thank the Thailand Research Fund (Grant No. RSA 5280033), Prince of Songkla University and Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160. TS wishes to thank Dr. Nawong Boonnak for his useful discussions.

References

Batovska, D., Parushev, St., Slavova, A., Bankova, V., Tsvetkova, I., Ninova, M., & Najdenski, H. (2007). Study on the substituents' effects of a series of synthetic chalcones against the yeast *Candida albicans*. *European Journal of Medicinal Chemistry*, *42*, 87–92. DOI: 10.1016/j.ejmech.2006.08.012.

Bruker AXS (2005). *APEX2* software. Madison, WI, USA: Bruker AXS Inc.

Bruker AXS (2001). *SHELXTL*, Version 6.10. Madison, WI, USA: Bruker AXS Inc.

Cordaro, J. G., McCusker, J. K., & Bergman, R. G. (2002). Synthesis of mono-substituted 2,2'-bipyridines. *Chemical Communications*, *2002*, 1496–1497. DOI: 10.1039/b203595b.

Crasta, V., Ravindrachary, V., Bhajantri, R. F., & Gonsalves, R. (2004). Growth and characterization of an organic NLO crystal: 1-(4-methylphenyl)-3-(4-methoxyphenyl)-2-propen-1-one. *Journal of Crystal Growth*, *267*, 129–133. DOI: 10.1016/j.jcrysgro.2004.03.037.

Dudeja, M., Malhotra, R., Gupta, M. P., & Dhindsa, K. S. (1993). Synthesis and characterization of cobalt(II),

nickel(II) and copper(II) complexes of 1-acetyl-5-aryl-3-(substituted thienyl)-2-pyrazolines and their microbiocidal activity. *Indian Journal of Chemistry*, *32A*, 975–979.

Dulawat, S. S., Chundawat, J. S., Roy, R. S., Chundawat, S. S., & Verma, B. L. (2010). Microwave assisted improved synthesis of 6-carbethoxy-5-aryl-3-(2-thienyl)-2-cyclohexenones using inorganic solid support and their antibacterial activities. *Journal of the Indian Chemical Society*, *87*, 981–986.

Fahrni, C. J., Yang, L., & VanDerveer, D. G. (2003). Tuning the photoinduced electron-transfer thermodynamics in 1,3,5-triaryl-2-pyrazoline fluorophores: X-ray structures, photophysical characterization, computational analysis, and in vivo evaluation. *Journal of the American Chemical Society*, *125*, 3799–3812. DOI: 10.1021/ja028266o.

Fayed, T. A., & Awad, M. K. (2004). Dual emission of chalcone-analogue dyes emitting in the red region. *Chemical Physics*, *303*, 317–326. DOI: 10.1016/j.chemphys.2004.06.023.

Fun, H.-K., Jebas, S. R., Patil, P. S., & Dharmaprakash, S. M. (2008). (*E*)-1-(2-Thienyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one. *Acta Crystallographica Section E*, *E64*, o1510–o1511. DOI: 10.1107/S1600536808021375.

Fun, H.-K., Suwunwong, T., Chantrapromma, S., & Karalai, C. (2010a). (*E*)-1-(2-Furyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one. *Acta Crystallographica Section E*, *E66*, o2559–o2560. DOI: 10.1107/S1600536810035762.

Fun, H.-K., Suwunwong, T., Chantrapromma, S., & Karalai, C. (2010b). (*E*)-1-(2-Furyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one. *Acta Crystallographica Section E*, *E66*, o3070–o3071. DOI: 10.1107/S160053681004451X.

Gaber, M., El-Daly, S. A., Fayed, T. A., & El-Sayed, Y. S. (2008). Photophysical properties, laser activity and photoreactivity of a heteroaryl chalcone. A model of solvatochromic fluorophore. *Optics & Laser Technology*, *40*, 528–537. DOI: 10.1016/j.optlastec.2007.08.006.

Hirano, J., Hamase, K., Fukuda, H., Tomita, T., & Zaitsu, K. (2004). Novel stable fluorophore, 6-methoxy-4-quinolone, with strong fluorescence in wide pH range of aqueous media, and its application as a fluorescent labeling reagent. *Journal of Chromatography A*, *1059*, 225–231. DOI: 10.1016/j.chroma.2004.10.020.

Jung, Y. J., Son, K.-I., Oh, Y. E., & Noh, D.-Y. (2008). Ferrocenyl chalcones containing anthracenyl group: Synthesis, X-ray crystal structures and electrochemical properties. *Polyhedron*, *27*, 861–867. DOI: 10.1016/j.poly.2007.11.015.

Katiyar, S. S., Lalithambika, M., & Joshi, G. C. (1974). Polarographic investigations on α,β-unsaturated ketones: 1-(2-thienyl)-3-phenyl-2-propenones. *Journal of Electroanalytical Chemistry and Interfacial Electrochemistry*, *53*, 439–447. DOI: 10.1016/S0022-0728(74)80080-X.

Knyazhansky, M. I., Kharlanov, V. A., & Tymiansky, Y. R. (1998). Adiabatic structural relaxation in heterocyclic nitrogen-containing cations. The structure, absorption and fuorescence of the 2,4,6-triarylsubstituted pyridinium cations. *Journal of Photochemistry and Photobiology A: Chemistry*, *118*, 151–156. DOI: 10.1016/S1010-6030(98)00377-3.

Lavrushin, V. F., Tsukerman, S. V., & Nikitchenko, V. M. (1962). Spectra of and halochromism in thiophene analogs of methoxychalcones and their vinylogs. *Zhurnal Obshchei Khimii*, *32*, 3971–3977.

Lavrushin, V. F., Tsukerman, S. V., & Nikitchenko, V. M. (1961). Synthesis of thiophene analogs of di- and trimethoxychalcones and their vinylogs. *Zhurnal Obshchei Khimii*, *31*, 2845–2850.

Lawrence, N. J., McGown, A. T., Ducki, S., & Hadfield, J. A. (2000). The interaction of chalcones with tubulin. *Anti-Cancer Drug Design*, *15*, 135–141.

Lin, R.-H., & Chuang, L.-M. (2007). U.S. Patent No. 2007037 193. Alexandria, VA, USA: U.S. Patent and Trademark Office.

Lin, R.-H., Lin, L., Lin, S.-Y., & Lee, S.-H. (2007). International Patent No. WO 2007082178. Geneva, Switzerland: World Intellectual Property Organization.

López, S. N., Castelli, M. V., Zacchino, S. A., Domínguez, J. N., Lobo, G., Charris-Charris, J., Cortés, J. C. G., Ribas, J. C., Devia, C., Rodríguez, A. M., & Enriz, R. D. (2001). In vitro antifungal evaluation and structure–activity relationships of a new series of chalcone derivatives and synthetic analogues, with inhibitory properties against polymers of the fungal cell wall. *Bioorganic & Medicinal Chemistry*, *9*, 1999–2013. DOI: 10.1016/S0968-0896(01)00116-X.

Maiti, M., Sinha, S., Deb, C., De, A., & Ganguly, T. (1999). Photophysics of 4-methoxy-benzo[b]thiophene in different environments. Its role in non-radiative transitions both as an electron and as an energy donor. *Journal of Luminescence*, *82*, 259–276. DOI: 10.1016/S0022-2313(99)00062-9.

Musil, Z., Zimcik, P., Miletin, M., Kopecky, K., Petrik, P., & Lenco, J. (2007). Influence of electron-withdrawing and electron-donating substituents on photophysical properties of azaphthalocyanines. *Journal of Photochemistry and Photobiology A: Chemistry*, *186*, 316–322. DOI: 10.1016/j.jphotochem.2006.08.024.

Navarini, A. L. F., Chiaradia, L. D., Mascarello, A., Fritzen, M., Nunes, R. J., Yunes, R. A., & Creczynski-Pasa, T. B. (2009). Hydroxychalcones induce apoptosis in B16-F10 melanoma cells via GSH and ATP depletion. *European Journal of Medicinal Chemistry*, *44*, 1630–1637. DOI: 10.1016/j.ejmech.2008.09.009.

Nepali, K., Singh, G., Turan, A., Agarwal, A., Sapra, S., Kumar, R., Banerjee, U. C., Verma, P. K., Satti, N. K., Gupta, M. K., Suri, O. P., & Dhar, K. L. (2011). A rational approach for the design and synthesis of 1-acetyl-3,5-diaryl-4,5-dihydro(1*H*)pyrazoles as a new class of potential non-purine xanthine oxidase inhibitors. *Bioorganic & Medicinal Chemistry*, *19*, 1950–1958. DOI: 10.1016/j.bmc.2011.01.058.

Nielsen, S. F., Boesen, T., Larsen, M., Schønning, K., & Kromann, H. (2004). Antibacterial chalcones—bioisosteric replacement of the 4′-hydroxy group. *Bioorganic & Medicinal Chemistry*, *12*, 3047–3054. DOI: 10.1016/j.bmc.2004.03.071.

Niu, C.-G., Guan, A.-L., Zeng, G.-M., Liu, Y.-G., & Li, Z.-W. (2006). Fluorescence water sensor based on covalent immobilization of chalcone derivative. *Analytica Chimica Acta*, *577*, 264–270. DOI: 10.1016/j.aca.2006.06.046.

Percino, M. J., Chapela, V. M., Pérez-Gutiérrez, E., Cerón, M., & Soriano, G. (2011). Synthesis, optical, and spectroscopic characterisation of substituted 3-phenyl-2-arylacrylonitriles. *Chemical Papers*, *65*, 42–51. DOI: 10.2478/s11696-010-0075-x.

Prasad, Y. R., Kumar P. P., & Kumar, P. R. (2007a). Synthesis and biological evaluation of some new 2,4,6-trisubstituted pyrimidines. *Oriental Journal of Chemistry*, *23*, 1069–1072.

Prasad, Y. R., Kumar, P. P., Kumar, P. R., & Rao, A. S. (2008). Synthesis and antimicrobial activity of some new chalcones of 2-acetyl pyridine. *E-Journal of Chemistry*, *5*, 144–148.

Prasad, Y. R., Kumar, P. R., Sarath, N., & Rao, A. S. (2007b). Synthesis and antimicrobial activity of some new chalcones of 2-acetylthiophene. *International Journal of Chemical Sciences*, *5*, 2372–2378.

Ramesh, B., Kulakarni, S. V., & Ravindra, R. (2010). Synthesis, spectral studies and anti-cancer activities of some new pyrimidine derivatives. *International Journal of Pharmaceutical Sciences*, *2*, 426–428.

Ramesh, B., Prasad, Y. R., & Ahmed, S. M. (2009a). Synthesis and anti-microbial activity of some new pyrimidine derivatives. *Pharmacologyonline*, *2*, 331–335.

Ramesh, B., Prasad, Y. R., & Ahmed, S. M. (2009b). Synthesis and antimicrobial activity of some 2-pyrazoline derivatives. *Pharmacologyonline*, *2*, 327–330.

Ramesh, B., & Rao, B. S. (2010). Synthesis, spectral studies and anti-inflammatory activity of 2-acetyl thiophene. *E-Journal of Chemistry*, *7*, 433–436.

Rhys Williams, A. T., Winfield, S. A., & Miller, J. N. (1983). Relative fluorescence quantum yields using a computer-controlled luminescence spectrometer. *Analyst*, *108*, 1067–1071. DOI: 10.1039/an9830801067.

Romagnoli, R., Baraldi, P. G., Carrion, M. D., Cara, C. L., Cruz-Lopez, O., Preti, D., Tolomeo, M., Grimaudo, S., Di Cristina, A., Zonta, N., Balzarini, J., Brancale, A., Sarkar, T., & Hamel, E. (2008). Design, synthesis, and biological evaluation of thiophene analogues of chalcones. *Bioorganic & Medicinal Chemistry*, *16*, 5367–5376. DOI: 10.1016/j.bmc.2008.04.026.

Roman, G. (2004). Cyclohexenones through addition of ethyl acetoacetate to chalcones derived from 2-acetylthiophene. *Acta Chimica Slovenica*, *51*, 537–544.

Salem, M. M., & Werbovetz, K. A. (2005). Antiprotozoal compounds from *Psorothamnus polydenius*. *Journal of Natural Products*, *68*, 108–111. DOI: 10.1021/np049682k.

Schlangen, K., Miosic, S., Topuz, F., Muster, G., Marosits, T., Seitz, C., & Halbwirth, H. (2009). Chalcone 3-hydroxylation is not a general property of flavonoid 3′-hydroxylase. *Plant Science*, *177*, 97–102. DOI: 10.1016/j.plantsci.2009.04.002.

Sheldrick, G. M. (2008). A short history of *SHELX*. *Acta Crystallographica Section A*, *A64*, 112–122. DOI: 10.1107/S0108767307043930.

Sheldrick, G. M. (2003). *SADABS*, Version 2.10. Göttingen, Germany: University of Göttingen.

Siemens AXS (1998). *SMART* and *SAINT*, Version 5.0. Area detector control and integration software. Madison, WI, USA: Siemens AXS Inc.

Singhal, R. K., & Mishra, N. K. (1985). Studies on cycloimmonium ylides: synthesis of some new 2,4,6-triarylsubstituted pyridines via pyridinium ylides. *Indian Journal of Chemistry Section B*, *24B*, 1079–1080.

Singhal, R. K., & Misra, N. K. (1986). Studies on aroylmethylenesulfonium ylides: synthesis of some new 1,2,3-trisubstituted cyclopropanes via sulfonium ylides. *Current Science*, *55*, 783–784.

Sivakumar, P. M., Prabhawathi, V., & Doble, M. (2010). 2-Methoxy-2′,4′-dichloro chalcone as an antimicrofoulant against marine bacterial biofilm. *Colloids and Surfaces B: Biointerfaces*, *81*, 439–446. DOI: 10.1016/j.colsurfb.2010.07.037.

Sivakumar, P. M., Priya, S., & Doble, M. (2009). Synthesis, biological evaluation, mechanism of action and quantitative structure–activity relationship studies of chalcones as antibacterial agents. *Chemical Biology & Drug Design*, *73*, 403–415. DOI: 10.1111/j.1747-0285.2009.00793.x.

Spek, A. L. (2003). Single-crystal structure validation with the program PLATON. *Journal of Applied Crystallography*, *36*, 7–13. DOI: 10.1107/S0021889802022112.

Sun, X., Zhang, J., & He, B. (2005). The synthesis and photochemical characterization of *meso*-tetra-thienyl porphyrins. *Journal of Photochemistry and Photobiology A: Chemistry*, *172*, 283–288. DOI: 10.1016/j.jphotochem.2004.12.016.

Sun, Y.-F., & Cui, Y.-P. (2009). The synthesis, structure and spectroscopic properties of novel oxazolone-, pyrazolone- and pyrazoline-containing heterocycle chromophores. *Dyes and Pigments*, *81*, 27–34. DOI: 10.1016/j.dyepig.2008.08.010.

Sun, Y.-F., & Cui, Y.-P. (2008). The synthesis, characterization and properties of coumarin-based chromophores containing a chalcone moiety. *Dyes and Pigments*, *78*, 65–76. DOI: 10.1016/j.dyepig.2007.10.014.

Suwunwong, T., Chantrapromma, S., Pakdeevanich, P., & Fun, H.-K. (2009). (*E*)-1-(2-Thienyl)-3-(3,4,5-trimethoxyphenyl) prop-2-en-1-one. *Acta Crystallographica Section E*, *E65*, o1575–o1576. DOI: 10.1107/S1600536809021850.

Suzuki, T., Shinkai, M., & Nanba, N. (1995a). Japan Patent No. 7126543. Tokyo, Japan: Japan Patent Office.

Suzuki, T., Shinkai, M., & Nanba, N. (1995b). Japan Patent No. 7085499. Tokyo, Japan: Japan Patent Office.

Tsukerman, S. V., Nikitchenko, V. M., Orlov, V. D., & Lavrushin, V. F. (1969). Dipole moments of thiophene analogs of chalcones and their vinylogs. *Chemistry of Heterocyclic Compounds*, *3*, 173–177. DOI: 10.1007/BF01172541.

Tsukerman, S. V., Nikitchenko, V. M., Rozum, Y. S., & Lavrushin, V. F. (1967). Infrared spectra of thiophene analogs of chalcones and their vinylogs. *Chemistry of Heterocyclic Compounds*, *3*, 361–366. DOI: 10.1007/BF00945365.

Werts, M. H. V., Nerambourg, N., Pélégry, D., Le Grand, Y., & Blanchard-Desce, M. (2005). Action cross sections of two-photon excited luminescence of some Eu(III) and Tb(III) complexes. *Photochemical & Photobiological Sciences*, *4*, 531–538. DOI: 10.1039/b504495b.

Won, S.-J., Liu, C.-T., Tsao, L.-T., Weng, J.-R., Ko, H.-H., Wang, J.-P., & Lin, C.-N. (2005). Synthetic chalcones as potential anti-inflammatory and cancer chemopreventive agents. *European Journal of Medicinal Chemistry*, *40*, 103–112. DOI: 10.1016/j.ejmech.2004.09.006.

Xu, Z., Bai, G., & Dong, C. (2005). Studies on interaction of an intramolecular charge transfer fluorescence probe: 4′-Dimethylamino-2,5-dihydroxychalcone with DNA. *Bioorganic & Medicinal Chemistry*, *13*, 5694–5699. DOI: 10.1016/j.bmc.2005.06.023.

Zangade, S. B., Jadhav, J. D., Lalpod, Vibhute, Y. B., & Dawane, B. S. (2010). Synthesis and antimicrobial activity of some new chalcones and flavones containing substituted naphthalene moiety. *Journal of Chemical and Pharmaceutical Research*, *2*, 310–314.

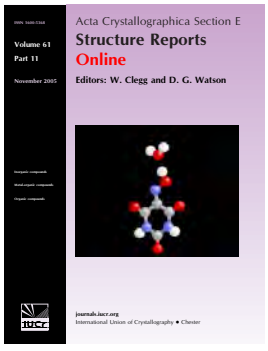
Zhang, J., Xu, Z., Wei, Y., Shuang, S., & Dong, C. (2008). Spectral properties of intramolecular charge transfer fluorescence probe 1-keto-2-(*p*-dimethylaminobenzal)-tetrahydronaphthalene. *Spectrochimica Acta Part A*, *70*, 888–891. DOI: 10.1016/j.saa.2007.10.002.

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

Redetermination of (*E*)-3-(anthracen-9-yl)-1-(2-hydroxyphenyl)prop-2-en-1-one

Suchada Chantrapromma, Thawanrat Kobkeatthawin, Kullapa Chanawanno, Jaruwan Joothamongkhon and Hoong-Kun Fun

Acta Cryst. (2011). E**67**, o2554–o2555



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination of (*E*)-3-(anthracen-9-yl)-1-(2-hydroxyphenyl)prop-2-en-1-one[1]

Suchada Chantrapromma,[a]*§ Thawanrat Kobkeatthawin,[a] Kullapa Chanawanno,[a] Jaruwan Joothamongkhon[a] and Hoong-Kun Fun[b]¶

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

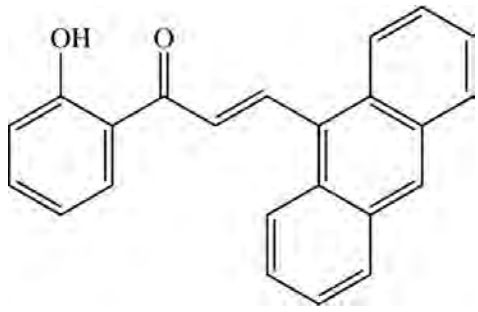
Received 4 July 2011; accepted 26 August 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.132; data-to-parameter ratio = 20.4.

The redetermined structure of title chalcone derivative, $C_{23}H_{16}O_2$, corrects errors in the title, scheme and synthesis in the previous report of the same structure [Jasinski *et al.* (2011). *Acta Cryst.* E**67**, o795]. There are two independent molecules in the asymmetric unit with slight differences in bond lengths and angles. The dihedral angle between the benzene ring and the anthracene ring system is 73.30 (4)° in one molecule and 73.18 (4)° in the other. Both molecules feature an intramolecular O—H···O hydrogen bond, which generates an S(6) ring. In the crystal, molecules are arranged into sheets lying parallel to the *ac* plane and further stacked along the *b* axis by π–π interactions with centroid–centroid distances in the range 3.6421 (6)–3.7607 (6) Å. The crystal structure is further stabilized by C—H···π interactions. There are also C···O [3.2159 (15) Å] short contacts.

Related literature

For the previous structure determination, see: Jasinski *et al.* (2011). For a related structure and background references, see: Joothamongkhon *et al.* (2010). For graph-set motifs, see: Bernstein *et al.* (1995). For the stability of the temperature controller used in the data collection, see Cosier & Glazer (1986).



Experimental

Crystal data

$C_{23}H_{16}O_2$
M_r = 324.36
Monoclinic, $P2_1/c$
a = 14.0843 (2) Å
b = 13.7224 (2) Å
c = 16.9615 (3) Å
β = 101.411 (1)°
V = 3213.36 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.50 × 0.39 × 0.37 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.959, T_{max} = 0.969
40230 measured reflections
9368 independent reflections
7868 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.132
S = 1.02
9368 reflections
459 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1, *Cg*3, *Cg*5, *Cg*6 and *Cg*7 are the centroids of the C1*A*–C6*A*, C8*A*–C13*A*, C1*B*–C6*B*, C1*B*/C6*B*–C8*B*/C13*B*–C14*B* and C8*B*–C13*B* rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
O2*A*—H1*OA*···O1*A*	0.93 (2)	1.69 (2)	2.5459 (12)	152.2 (19)
O2*B*—H1*OB*···O1*B*	0.88 (2)	1.75 (2)	2.5725 (13)	154.2 (19)
C5*A*—H5*AA*···*Cg*5	0.93	2.84	3.6754 (13)	151
C7*A*—H7*AA*···*Cg*6	0.93	2.76	3.6440 (12)	158
C9*A*—H9*AA*···*Cg*7	0.93	2.73	3.6325 (12)	164
C9*B*—H9*BA*···*Cg*1^{i}	0.93	2.76	3.4023 (12)	127
C23*B*—H23*B*···*Cg*3	0.93	2.91	3.7661 (11)	154

Symmetry code: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for a research grant (RSA 5280033) and the Prince of Songkla University for financial support. The authors also thank Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

[1] This paper is dedicated to Her Royal Highness Princess Chulabhorn Walailak of Thailand for her contributions to science on the occasion of her 54th birthday, which fell on July 4th, 2011.
§ Thomson Reuters ResearcherID: A-5085-2009.
¶ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5945).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Jasinski, J. P., Butcher, R. J., Musthafa Khaleel, V., Sarojini, B. K. & Yathirajan, H. S. (2011). *Acta Cryst.* E**67**, o795.

Joothamongkhon, J., Chantrapromma, S., Kobkeatthawin, T. & Fun, H.-K. (2010). *Acta Cryst.* E**66**, o2669–o2670.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2011). E**67**, o2554-o2555 [**doi:10.1107/S1600536811034994**]

Redetermination of (*E*)-3-(anthracen-9-yl)-1-(2-hydroxyphenyl)prop-2-en-1-one

S. Chantrapromma, T. Kobkeatthawin, K. Chanawanno, J. Joothamongkhon and H.-K. Fun

Comment

From our previous work, which revealed that a chalcone derivative containing the anthracene moiety displayed fluorescence (Joothamongkhon *et al.*, 2010), the title compound (I) was synthesized by changing the substituent group on the phenyl ring for comparison of their properties. It was then discovered that a recent study of the same structure (Jasinski *et al.*, 2011) contained errors in the title, scheme and synthesis. It was found that (I) exhibits fluorescence with the maximum emission at 438 nm when excited at 380 nm in chloroform solution. In addition our experiment shows that (I) also exhibits tyrosinase inhibitory activity with % inhibition of 12.882±8.511 at the concentration 0.125 mg ml^{-1} when *L*-tyrosine was used as substrate.

The asymmetric unit of (I) contains two molecules, *A* and *B*, with the same configuration but with slight differences in bond lengths and angles. The molecule of (I) (Fig. 1) exists in an *E* configuration with respect to the C15═C16 double bond [1.3392 (15) Å in molecule *A* and 1.3370 (15) Å in molecule *B*] and the torsion angle C14–C15–C16–C17 = 177.64 (10)° in molecule *A* [-179.49 (10)° in molecule *B*]. The anthracene unit is essentially planar with the *r.m.s*. 0.0270 (1) Å for molecule *A* [0.0236 (1) Å for molecule *B*]. Atom O1 of the prop-2-en-1-one (C15–C17/O1) moiety is deviated from the propene plane with the torsion angle C15–C16–C17–O1 = 18.56 (16)° in molecule *A* [-17.28 (17)° in molecule *B*]. The total molecule is twisted as the dihedral angle between phenyl and anthracene rings is 73.70 (4)° and the mean through the pro-2-en-1-one unit makes the dihedral angles of 14.70 (7) and 61.46 (6)° with the phenyl and anthracene rings, respectively [the corresponding values are 73.18 (4), 11.04 (7) and 62.15 (6)° in molecule *B*]. Intramolecular O2A—H1OA···O1A and O2B—H1OB..O1B hydrogen bonds (Table 1) generate S(6) ring motifs (Bernstein *et al.*, 1995).

The bond distances are comparable with those in the related structure noted above (Joothamongkhon *et al.*, 2010).

In the crystal (Fig. 2), the molecules are arranged into sheets parallel to the *ac* plane and further stacked along the *b* axis by π–π interactions with the centroid···centroid distances: $Cg_1\cdots Cg_2^{ii}$ = 3.6421 (6) Å; $Cg_1\cdots Cg_3^{ii}$ = 3.6800 (7) Å; $Cg_2\cdots Cg_2^{ii}$ = 3.7607 (6) Å; $Cg_4\cdots Cg_8^{iii}$ = 3.6434 (7) Å and $Cg_5\cdots Cg_6^{ii}$ = 3.7084 (6) Å. The crystal structure is further stabilized by C—H···π interactions (Table 1); Cg_1, Cg_2, Cg_3, Cg_4, Cg_5, Cg_6, Cg_7 and Cg_8 are the centroids of C1A–C6A, C1A/C6A–C8A/C13A–C14A, C8A–C13A, C18A–C23A, C1B–C6B, C1B/C6B–C8B/C13B–C14B, C8B–C13B and C18B–C23B rings, respectively. $C\cdots O^{iii}$[3.2159 (15) Å] short contacts were also observed [symmetry code: (iii) -*x*, -1/2 + *y*, 1/2 - *z*].

Experimental

The title compound was synthesized by the condensation of anthracene-9-carbaldehyde (2 mmol, 0.41 g) with 2-hydroxyacetophenone (2 mmol, 0.27 g) in ethanol (40 ml) in the presence of NaOH(aq) (10 ml, 40%). After stirring for 4 hr at room temperature, a yellow solid appeared and was then collected by filtration, washed with distilled water and dried in air. Yellow blocks of (I) were recrystalized from acetone by the slow evaporation of the solvent at room temperature after several days, Mp. 431–432 K.

Refinement

Hydroxy H atoms were located in a difference maps and refined isotropically. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C—H) = 0.93 Å for aromatic and CH. and the U_{iso} values were constrained to be $1.2U_{eq}$ of the carrier atoms. The highest residual electron density peak is located at 0.71 Å from C1A and the deepest hole is located at 0.43 Å from H1OA.

Figures

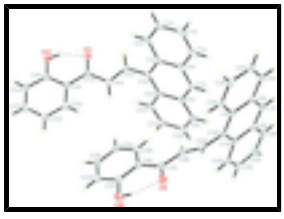


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids. O—H···O hydrogen bonds are shown as dashed lines.

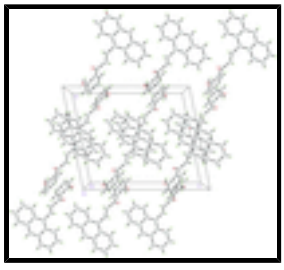


Fig. 2. The crystal packing of the title compound viewed along the *b* axis.

(*E*)-3-(anthracen-9-yl)-1-(2-hydroxyphenyl)prop-2-en-1-one

Crystal data

$C_{23}H_{16}O_2$	$F(000) = 1360$
$M_r = 324.36$	$D_x = 1.341$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point = 431–432 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 14.0843$ (2) Å	Cell parameters from 9368 reflections
$b = 13.7224$ (2) Å	$\theta = 1.9$–$30.0°$
$c = 16.9615$ (3) Å	$\mu = 0.09$ mm^{-1}
$\beta = 101.411$ (1)°	$T = 100$ K
$V = 3213.36$ (9) Å^3	Block, yellow
$Z = 8$	0.50 × 0.39 × 0.37 mm

Data collection

Bruker APEXII CCD diffractometer	9368 independent reflections
Radiation source: sealed tube	7868 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.032$
φ and ω scans	$\theta_{max} = 30.0°$, $\theta_{min} = 1.9°$

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -19 \rightarrow 19$
$T_{min} = 0.959$, $T_{max} = 0.969$	$k = -17 \rightarrow 19$
40230 measured reflections	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.046$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.132$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2 + 1.125P]$ where $P = (F_o^2 + 2F_c^2)/3$
9368 reflections	$(\Delta/\sigma)_{max} = 0.001$
459 parameters	$\Delta\rho_{max} = 0.54$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.23244 (6)	0.20731 (6)	0.25499 (5)	0.02242 (17)
O2A	0.12311 (6)	0.12104 (6)	0.13834 (5)	0.02449 (18)
C1A	0.44611 (8)	0.49417 (8)	0.37297 (6)	0.0174 (2)
C2A	0.50222 (8)	0.44622 (9)	0.32283 (6)	0.0227 (2)
H2AA	0.4773	0.3914	0.2936	0.027*
C3A	0.59212 (9)	0.48005 (11)	0.31737 (7)	0.0271 (3)
H3AA	0.6279	0.4475	0.2850	0.033*
C4A	0.63149 (9)	0.56412 (11)	0.36045 (7)	0.0279 (3)
H4AA	0.6923	0.5867	0.3556	0.033*
C5A	0.58036 (8)	0.61172 (9)	0.40878 (7)	0.0239 (2)
H5AA	0.6069	0.6666	0.4371	0.029*

C6A	0.48658 (8)	0.57869 (8)	0.41671 (6)	0.0185 (2)
C7A	0.43416 (8)	0.62652 (8)	0.46690 (7)	0.0194 (2)
H7AA	0.4608	0.6811	0.4955	0.023*
C8A	0.34265 (8)	0.59435 (8)	0.47522 (6)	0.0173 (2)
C9A	0.29196 (9)	0.64164 (8)	0.52973 (7)	0.0210 (2)
H9AA	0.3197	0.6953	0.5590	0.025*
C10A	0.20378 (9)	0.60920 (9)	0.53942 (7)	0.0231 (2)
H10A	0.1713	0.6412	0.5746	0.028*
C11A	0.16130 (8)	0.52659 (9)	0.49592 (7)	0.0217 (2)
H11A	0.1012	0.5045	0.5032	0.026*
C12A	0.20734 (8)	0.47903 (8)	0.44350 (6)	0.0188 (2)
H12A	0.1782	0.4248	0.4159	0.023*
C13A	0.29970 (8)	0.51123 (8)	0.43031 (6)	0.01584 (19)
C14A	0.35199 (8)	0.46226 (8)	0.37893 (6)	0.01602 (19)
C15A	0.31128 (8)	0.37739 (8)	0.33147 (6)	0.0184 (2)
H15A	0.3487	0.3211	0.3358	0.022*
C16A	0.22424 (8)	0.37492 (8)	0.28239 (6)	0.0199 (2)
H16A	0.1840	0.4292	0.2782	0.024*
C17A	0.19193 (8)	0.28682 (8)	0.23504 (6)	0.0179 (2)
C18A	0.11383 (8)	0.29411 (8)	0.16329 (6)	0.0172 (2)
C19A	0.08536 (8)	0.20943 (9)	0.11699 (6)	0.0193 (2)
C20A	0.01483 (9)	0.21635 (10)	0.04625 (7)	0.0242 (2)
H20A	−0.0024	0.1613	0.0147	0.029*
C21A	−0.02897 (9)	0.30437 (10)	0.02334 (7)	0.0252 (2)
H21A	−0.0761	0.3080	−0.0234	0.030*
C22A	−0.00370 (9)	0.38831 (9)	0.06921 (7)	0.0240 (2)
H22A	−0.0346	0.4472	0.0538	0.029*
C23A	0.06780 (8)	0.38290 (9)	0.13788 (7)	0.0209 (2)
H23A	0.0858	0.4390	0.1679	0.025*
O1B	0.18383 (6)	0.94230 (6)	0.26687 (5)	0.02468 (18)
O2B	0.03389 (7)	0.89265 (7)	0.16165 (5)	0.02348 (18)
C1B	0.50277 (8)	0.87960 (7)	0.47232 (6)	0.01604 (19)
C2B	0.53592 (8)	0.88359 (8)	0.39761 (6)	0.0194 (2)
H2BA	0.4912	0.8925	0.3499	0.023*
C3B	0.63166 (9)	0.87455 (9)	0.39523 (7)	0.0221 (2)
H3BA	0.6512	0.8783	0.3461	0.026*
C4B	0.70218 (9)	0.85953 (9)	0.46673 (7)	0.0223 (2)
H4BA	0.7673	0.8534	0.4641	0.027*
C5B	0.67406 (8)	0.85419 (8)	0.53925 (7)	0.0198 (2)
H5BA	0.7203	0.8442	0.5859	0.024*
C6B	0.57425 (8)	0.86375 (8)	0.54417 (6)	0.0166 (2)
C7B	0.54489 (8)	0.85640 (8)	0.61815 (6)	0.0173 (2)
H7BA	0.5912	0.8461	0.6647	0.021*
C8B	0.44772 (8)	0.86415 (8)	0.62337 (6)	0.0175 (2)
C9B	0.41872 (9)	0.85451 (9)	0.69944 (6)	0.0220 (2)
H9BA	0.4652	0.8414	0.7453	0.026*
C10B	0.32440 (9)	0.86416 (10)	0.70549 (7)	0.0253 (2)
H10B	0.3065	0.8564	0.7550	0.030*
C11B	0.25298 (9)	0.88619 (9)	0.63594 (7)	0.0233 (2)

H11B	0.1888	0.8945	0.6407	0.028*
C12B	0.27755 (8)	0.89535 (8)	0.56208 (7)	0.0196 (2)
H12B	0.2298	0.9104	0.5175	0.024*
C13B	0.37537 (8)	0.88220 (8)	0.55225 (6)	0.0165 (2)
C14B	0.40387 (8)	0.88795 (7)	0.47662 (6)	0.01590 (19)
C15B	0.33125 (8)	0.90127 (8)	0.40140 (6)	0.0180 (2)
H15B	0.3405	0.9525	0.3679	0.022*
C16B	0.25332 (8)	0.84486 (8)	0.37825 (6)	0.0189 (2)
H16B	0.2422	0.7934	0.4110	0.023*
C17B	0.18418 (8)	0.86241 (8)	0.30157 (6)	0.0181 (2)
C18B	0.11590 (8)	0.78474 (8)	0.26706 (6)	0.0167 (2)
C19B	0.04372 (8)	0.80431 (8)	0.19787 (6)	0.0184 (2)
C20B	−0.02191 (8)	0.73166 (9)	0.16524 (6)	0.0215 (2)
H20B	−0.0693	0.7450	0.1200	0.026*
C21B	−0.01681 (8)	0.64008 (9)	0.19984 (7)	0.0229 (2)
H21B	−0.0609	0.5922	0.1778	0.027*
C22B	0.05418 (9)	0.61899 (9)	0.26782 (7)	0.0217 (2)
H22B	0.0578	0.5572	0.2907	0.026*
C23B	0.11898 (8)	0.69067 (8)	0.30083 (6)	0.0190 (2)
H23B	0.1656	0.6765	0.3463	0.023*
H1OA	0.1696 (15)	0.1326 (15)	0.1845 (13)	0.052 (6)*
H1OB	0.0847 (15)	0.9246 (15)	0.1878 (12)	0.052 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0194 (4)	0.0219 (4)	0.0243 (4)	0.0023 (3)	0.0002 (3)	−0.0037 (3)
O2A	0.0203 (4)	0.0243 (4)	0.0272 (4)	0.0028 (3)	0.0006 (3)	−0.0089 (3)
C1A	0.0145 (5)	0.0220 (5)	0.0147 (4)	−0.0008 (4)	0.0008 (3)	0.0034 (4)
C2A	0.0185 (5)	0.0334 (6)	0.0157 (4)	0.0012 (4)	0.0021 (4)	0.0007 (4)
C3A	0.0182 (5)	0.0450 (8)	0.0187 (5)	0.0036 (5)	0.0048 (4)	0.0056 (5)
C4A	0.0159 (5)	0.0417 (7)	0.0254 (5)	−0.0036 (5)	0.0026 (4)	0.0124 (5)
C5A	0.0177 (5)	0.0273 (6)	0.0247 (5)	−0.0061 (4)	−0.0005 (4)	0.0094 (4)
C6A	0.0166 (5)	0.0195 (5)	0.0181 (4)	−0.0027 (4)	0.0004 (4)	0.0062 (4)
C7A	0.0206 (5)	0.0150 (5)	0.0209 (5)	−0.0032 (4)	−0.0001 (4)	0.0028 (4)
C8A	0.0187 (5)	0.0149 (5)	0.0173 (4)	0.0000 (4)	0.0011 (4)	0.0024 (3)
C9A	0.0255 (6)	0.0155 (5)	0.0210 (5)	0.0025 (4)	0.0023 (4)	−0.0006 (4)
C10A	0.0257 (6)	0.0226 (6)	0.0216 (5)	0.0057 (4)	0.0065 (4)	0.0009 (4)
C11A	0.0182 (5)	0.0245 (6)	0.0232 (5)	0.0006 (4)	0.0062 (4)	0.0034 (4)
C12A	0.0171 (5)	0.0193 (5)	0.0199 (5)	−0.0025 (4)	0.0030 (4)	0.0013 (4)
C13A	0.0159 (5)	0.0155 (5)	0.0154 (4)	−0.0003 (4)	0.0015 (3)	0.0019 (3)
C14A	0.0148 (5)	0.0178 (5)	0.0148 (4)	−0.0014 (4)	0.0013 (3)	0.0010 (3)
C15A	0.0176 (5)	0.0193 (5)	0.0182 (5)	−0.0005 (4)	0.0034 (4)	−0.0013 (4)
C16A	0.0194 (5)	0.0200 (5)	0.0192 (5)	0.0001 (4)	0.0016 (4)	−0.0035 (4)
C17A	0.0142 (5)	0.0221 (5)	0.0175 (4)	−0.0013 (4)	0.0037 (4)	−0.0028 (4)
C18A	0.0139 (5)	0.0222 (5)	0.0156 (4)	−0.0021 (4)	0.0036 (3)	−0.0017 (4)
C19A	0.0148 (5)	0.0242 (5)	0.0197 (5)	−0.0007 (4)	0.0055 (4)	−0.0046 (4)
C20A	0.0194 (5)	0.0319 (6)	0.0201 (5)	−0.0018 (5)	0.0012 (4)	−0.0084 (4)

C21A	0.0207 (5)	0.0365 (7)	0.0172 (5)	−0.0010 (5)	0.0010 (4)	−0.0010 (4)
C22A	0.0230 (6)	0.0274 (6)	0.0207 (5)	0.0000 (5)	0.0024 (4)	0.0045 (4)
C23A	0.0216 (5)	0.0221 (5)	0.0188 (5)	−0.0030 (4)	0.0032 (4)	0.0010 (4)
O1B	0.0254 (4)	0.0208 (4)	0.0249 (4)	−0.0021 (3)	−0.0021 (3)	0.0047 (3)
O2B	0.0221 (4)	0.0248 (4)	0.0214 (4)	0.0002 (3)	−0.0009 (3)	0.0023 (3)
C1B	0.0177 (5)	0.0129 (5)	0.0173 (4)	−0.0021 (4)	0.0028 (4)	−0.0009 (3)
C2B	0.0223 (5)	0.0185 (5)	0.0173 (5)	−0.0008 (4)	0.0038 (4)	0.0005 (4)
C3B	0.0242 (6)	0.0221 (5)	0.0217 (5)	−0.0015 (4)	0.0088 (4)	−0.0001 (4)
C4B	0.0179 (5)	0.0230 (6)	0.0273 (5)	−0.0004 (4)	0.0074 (4)	−0.0004 (4)
C5B	0.0165 (5)	0.0194 (5)	0.0228 (5)	−0.0003 (4)	0.0023 (4)	−0.0002 (4)
C6B	0.0173 (5)	0.0136 (5)	0.0186 (5)	−0.0009 (4)	0.0027 (4)	−0.0010 (3)
C7B	0.0179 (5)	0.0162 (5)	0.0169 (4)	−0.0013 (4)	0.0011 (4)	−0.0011 (3)
C8B	0.0195 (5)	0.0159 (5)	0.0169 (4)	−0.0029 (4)	0.0033 (4)	−0.0027 (3)
C9B	0.0234 (6)	0.0260 (6)	0.0163 (5)	−0.0046 (4)	0.0031 (4)	−0.0032 (4)
C10B	0.0254 (6)	0.0324 (6)	0.0193 (5)	−0.0059 (5)	0.0078 (4)	−0.0065 (4)
C11B	0.0183 (5)	0.0278 (6)	0.0249 (5)	−0.0035 (4)	0.0067 (4)	−0.0079 (4)
C12B	0.0177 (5)	0.0189 (5)	0.0219 (5)	−0.0018 (4)	0.0031 (4)	−0.0042 (4)
C13B	0.0172 (5)	0.0139 (5)	0.0180 (4)	−0.0020 (4)	0.0026 (4)	−0.0028 (3)
C14B	0.0170 (5)	0.0129 (5)	0.0171 (4)	−0.0015 (4)	0.0018 (4)	−0.0006 (3)
C15B	0.0193 (5)	0.0171 (5)	0.0172 (4)	0.0005 (4)	0.0023 (4)	0.0003 (4)
C16B	0.0188 (5)	0.0186 (5)	0.0182 (5)	−0.0012 (4)	0.0010 (4)	0.0013 (4)
C17B	0.0162 (5)	0.0194 (5)	0.0183 (4)	−0.0002 (4)	0.0026 (4)	−0.0006 (4)
C18B	0.0144 (5)	0.0203 (5)	0.0155 (4)	0.0000 (4)	0.0035 (3)	−0.0020 (4)
C19B	0.0164 (5)	0.0231 (5)	0.0164 (4)	0.0019 (4)	0.0046 (4)	−0.0015 (4)
C20B	0.0163 (5)	0.0305 (6)	0.0173 (5)	−0.0011 (4)	0.0028 (4)	−0.0052 (4)
C21B	0.0191 (5)	0.0264 (6)	0.0237 (5)	−0.0049 (4)	0.0057 (4)	−0.0081 (4)
C22B	0.0212 (5)	0.0208 (5)	0.0241 (5)	−0.0019 (4)	0.0069 (4)	−0.0028 (4)
C23B	0.0172 (5)	0.0216 (5)	0.0184 (5)	0.0002 (4)	0.0042 (4)	−0.0014 (4)

Geometric parameters (Å, °)

O1A—C17A	1.2466 (14)	O1B—C17B	1.2438 (14)
O2A—C19A	1.3449 (14)	O2B—C19B	1.3537 (14)
O2A—H1OA	0.93 (2)	O2B—H1OB	0.88 (2)
C1A—C14A	1.4186 (14)	C1B—C14B	1.4138 (15)
C1A—C2A	1.4306 (15)	C1B—C6B	1.4354 (14)
C1A—C6A	1.4329 (16)	C1B—C2B	1.4356 (14)
C2A—C3A	1.3688 (17)	C2B—C3B	1.3627 (16)
C2A—H2AA	0.9300	C2B—H2BA	0.9300
C3A—C4A	1.418 (2)	C3B—C4B	1.4224 (17)
C3A—H3AA	0.9300	C3B—H3BA	0.9300
C4A—C5A	1.3606 (19)	C4B—C5B	1.3668 (15)
C4A—H4AA	0.9300	C4B—H4BA	0.9300
C5A—C6A	1.4277 (15)	C5B—C6B	1.4307 (15)
C5A—H5AA	0.9300	C5B—H5BA	0.9300
C6A—C7A	1.3962 (16)	C6B—C7B	1.4003 (14)
C7A—C8A	1.3957 (15)	C7B—C8B	1.3930 (15)
C7A—H7AA	0.9300	C7B—H7BA	0.9300
C8A—C9A	1.4306 (15)	C8B—C9B	1.4344 (14)

C8A—C13A	1.4371 (15)	C8B—C13B	1.4383 (15)
C9A—C10A	1.3595 (17)	C9B—C10B	1.3590 (17)
C9A—H9AA	0.9300	C9B—H9BA	0.9300
C10A—C11A	1.4187 (17)	C10B—C11B	1.4231 (17)
C10A—H10A	0.9300	C10B—H10B	0.9300
C11A—C12A	1.3657 (15)	C11B—C12B	1.3699 (15)
C11A—H11A	0.9300	C11B—H11B	0.9300
C12A—C13A	1.4327 (15)	C12B—C13B	1.4315 (15)
C12A—H12A	0.9300	C12B—H12B	0.9300
C13A—C14A	1.4167 (14)	C13B—C14B	1.4200 (14)
C14A—C15A	1.4664 (15)	C14B—C15B	1.4802 (14)
C15A—C16A	1.3392 (15)	C15B—C16B	1.3370 (15)
C15A—H15A	0.9300	C15B—H15B	0.9300
C16A—C17A	1.4728 (15)	C16B—C17B	1.4821 (14)
C16A—H16A	0.9300	C16B—H16B	0.9300
C17A—C18A	1.4736 (14)	C17B—C18B	1.4767 (15)
C18A—C23A	1.4072 (16)	C18B—C23B	1.4095 (15)
C18A—C19A	1.4153 (15)	C18B—C19B	1.4179 (14)
C19A—C20A	1.4014 (15)	C19B—C20B	1.3972 (16)
C20A—C21A	1.3768 (18)	C20B—C21B	1.3828 (18)
C20A—H20A	0.9300	C20B—H20B	0.9300
C21A—C22A	1.3962 (17)	C21B—C22B	1.3989 (17)
C21A—H21A	0.9300	C21B—H21B	0.9300
C22A—C23A	1.3828 (16)	C22B—C23B	1.3827 (16)
C22A—H22A	0.9300	C22B—H22B	0.9300
C23A—H23A	0.9300	C23B—H23B	0.9300
C19A—O2A—H1OA	104.3 (13)	C19B—O2B—H1OB	102.6 (13)
C14A—C1A—C2A	122.30 (10)	C14B—C1B—C6B	120.07 (9)
C14A—C1A—C6A	119.63 (10)	C14B—C1B—C2B	122.56 (10)
C2A—C1A—C6A	118.04 (10)	C6B—C1B—C2B	117.34 (10)
C3A—C2A—C1A	120.75 (12)	C3B—C2B—C1B	121.28 (10)
C3A—C2A—H2AA	119.6	C3B—C2B—H2BA	119.4
C1A—C2A—H2AA	119.6	C1B—C2B—H2BA	119.4
C2A—C3A—C4A	120.91 (11)	C2B—C3B—C4B	121.07 (10)
C2A—C3A—H3AA	119.5	C2B—C3B—H3BA	119.5
C4A—C3A—H3AA	119.5	C4B—C3B—H3BA	119.5
C5A—C4A—C3A	120.09 (11)	C5B—C4B—C3B	119.82 (11)
C5A—C4A—H4AA	120.0	C5B—C4B—H4BA	120.1
C3A—C4A—H4AA	120.0	C3B—C4B—H4BA	120.1
C4A—C5A—C6A	120.94 (12)	C4B—C5B—C6B	120.71 (10)
C4A—C5A—H5AA	119.5	C4B—C5B—H5BA	119.6
C6A—C5A—H5AA	119.5	C6B—C5B—H5BA	119.6
C7A—C6A—C5A	121.26 (11)	C7B—C6B—C5B	120.89 (10)
C7A—C6A—C1A	119.48 (10)	C7B—C6B—C1B	119.33 (10)
C5A—C6A—C1A	119.26 (10)	C5B—C6B—C1B	119.77 (9)
C8A—C7A—C6A	121.55 (10)	C8B—C7B—C6B	121.30 (10)
C8A—C7A—H7AA	119.2	C8B—C7B—H7BA	119.4
C6A—C7A—H7AA	119.2	C6B—C7B—H7BA	119.4
C7A—C8A—C9A	120.72 (10)	C7B—C8B—C9B	120.55 (10)

C7A—C8A—C13A	119.85 (10)	C7B—C8B—C13B	120.12 (9)
C9A—C8A—C13A	119.41 (10)	C9B—C8B—C13B	119.33 (10)
C10A—C9A—C8A	120.95 (11)	C10B—C9B—C8B	121.02 (11)
C10A—C9A—H9AA	119.5	C10B—C9B—H9BA	119.5
C8A—C9A—H9AA	119.5	C8B—C9B—H9BA	119.5
C9A—C10A—C11A	120.00 (10)	C9B—C10B—C11B	119.96 (10)
C9A—C10A—H10A	120.0	C9B—C10B—H10B	120.0
C11A—C10A—H10A	120.0	C11B—C10B—H10B	120.0
C12A—C11A—C10A	120.98 (11)	C12B—C11B—C10B	120.83 (11)
C12A—C11A—H11A	119.5	C12B—C11B—H11B	119.6
C10A—C11A—H11A	119.5	C10B—C11B—H11B	119.6
C11A—C12A—C13A	121.14 (10)	C11B—C12B—C13B	121.21 (11)
C11A—C12A—H12A	119.4	C11B—C12B—H12B	119.4
C13A—C12A—H12A	119.4	C13B—C12B—H12B	119.4
C14A—C13A—C12A	123.25 (10)	C14B—C13B—C12B	123.29 (10)
C14A—C13A—C8A	119.15 (9)	C14B—C13B—C8B	119.16 (10)
C12A—C13A—C8A	117.51 (9)	C12B—C13B—C8B	117.54 (9)
C13A—C14A—C1A	120.29 (10)	C1B—C14B—C13B	119.97 (9)
C13A—C14A—C15A	121.33 (9)	C1B—C14B—C15B	119.04 (9)
C1A—C14A—C15A	118.38 (9)	C13B—C14B—C15B	120.98 (10)
C16A—C15A—C14A	124.87 (10)	C16B—C15B—C14B	124.67 (10)
C16A—C15A—H15A	117.6	C16B—C15B—H15B	117.7
C14A—C15A—H15A	117.6	C14B—C15B—H15B	117.7
C15A—C16A—C17A	120.39 (10)	C15B—C16B—C17B	121.49 (10)
C15A—C16A—H16A	119.8	C15B—C16B—H16B	119.3
C17A—C16A—H16A	119.8	C17B—C16B—H16B	119.3
O1A—C17A—C16A	119.67 (10)	O1B—C17B—C18B	120.49 (10)
O1A—C17A—C18A	120.69 (10)	O1B—C17B—C16B	119.92 (10)
C16A—C17A—C18A	119.62 (10)	C18B—C17B—C16B	119.59 (10)
C23A—C18A—C19A	118.57 (10)	C23B—C18B—C19B	117.98 (10)
C23A—C18A—C17A	122.35 (10)	C23B—C18B—C17B	122.32 (10)
C19A—C18A—C17A	119.07 (10)	C19B—C18B—C17B	119.70 (10)
O2A—C19A—C20A	117.85 (10)	O2B—C19B—C20B	117.32 (10)
O2A—C19A—C18A	122.52 (10)	O2B—C19B—C18B	122.50 (10)
C20A—C19A—C18A	119.63 (11)	C20B—C19B—C18B	120.17 (10)
C21A—C20A—C19A	120.22 (11)	C21B—C20B—C19B	120.36 (10)
C21A—C20A—H20A	119.9	C21B—C20B—H20B	119.8
C19A—C20A—H20A	119.9	C19B—C20B—H20B	119.8
C20A—C21A—C22A	121.06 (11)	C20B—C21B—C22B	120.44 (11)
C20A—C21A—H21A	119.5	C20B—C21B—H21B	119.8
C22A—C21A—H21A	119.5	C22B—C21B—H21B	119.8
C23A—C22A—C21A	119.21 (11)	C23B—C22B—C21B	119.57 (11)
C23A—C22A—H22A	120.4	C23B—C22B—H22B	120.2
C21A—C22A—H22A	120.4	C21B—C22B—H22B	120.2
C22A—C23A—C18A	121.27 (11)	C22B—C23B—C18B	121.48 (10)
C22A—C23A—H23A	119.4	C22B—C23B—H23B	119.3
C18A—C23A—H23A	119.4	C18B—C23B—H23B	119.3
C14A—C1A—C2A—C3A	178.79 (11)	C14B—C1B—C2B—C3B	−179.47 (10)
C6A—C1A—C2A—C3A	0.37 (16)	C6B—C1B—C2B—C3B	−1.22 (16)

C1A—C2A—C3A—C4A	−0.77 (18)	C1B—C2B—C3B—C4B	0.83 (18)
C2A—C3A—C4A—C5A	0.78 (18)	C2B—C3B—C4B—C5B	−0.13 (18)
C3A—C4A—C5A—C6A	−0.38 (17)	C3B—C4B—C5B—C6B	−0.13 (18)
C4A—C5A—C6A—C7A	179.27 (11)	C4B—C5B—C6B—C7B	178.72 (11)
C4A—C5A—C6A—C1A	−0.01 (16)	C4B—C5B—C6B—C1B	−0.31 (16)
C14A—C1A—C6A—C7A	2.27 (15)	C14B—C1B—C6B—C7B	0.20 (15)
C2A—C1A—C6A—C7A	−179.27 (10)	C2B—C1B—C6B—C7B	−178.10 (10)
C14A—C1A—C6A—C5A	−178.44 (10)	C14B—C1B—C6B—C5B	179.24 (10)
C2A—C1A—C6A—C5A	0.03 (15)	C2B—C1B—C6B—C5B	0.95 (15)
C5A—C6A—C7A—C8A	−179.69 (10)	C5B—C6B—C7B—C8B	−179.03 (10)
C1A—C6A—C7A—C8A	−0.41 (16)	C1B—C6B—C7B—C8B	0.01 (16)
C6A—C7A—C8A—C9A	177.12 (10)	C6B—C7B—C8B—C9B	178.75 (10)
C6A—C7A—C8A—C13A	−1.18 (16)	C6B—C7B—C8B—C13B	−1.40 (16)
C7A—C8A—C9A—C10A	−178.48 (10)	C7B—C8B—C9B—C10B	178.48 (11)
C13A—C8A—C9A—C10A	−0.17 (16)	C13B—C8B—C9B—C10B	−1.37 (17)
C8A—C9A—C10A—C11A	0.81 (17)	C8B—C9B—C10B—C11B	−1.32 (19)
C9A—C10A—C11A—C12A	−0.55 (17)	C9B—C10B—C11B—C12B	1.74 (19)
C10A—C11A—C12A—C13A	−0.37 (17)	C10B—C11B—C12B—C13B	0.62 (18)
C11A—C12A—C13A—C14A	177.54 (10)	C11B—C12B—C13B—C14B	177.98 (10)
C11A—C12A—C13A—C8A	0.98 (16)	C11B—C12B—C13B—C8B	−3.23 (16)
C7A—C8A—C13A—C14A	0.91 (15)	C7B—C8B—C13B—C14B	2.56 (15)
C9A—C8A—C13A—C14A	−177.41 (10)	C9B—C8B—C13B—C14B	−177.58 (10)
C7A—C8A—C13A—C12A	177.61 (10)	C7B—C8B—C13B—C12B	−176.28 (10)
C9A—C8A—C13A—C12A	−0.71 (15)	C9B—C8B—C13B—C12B	3.57 (15)
C12A—C13A—C14A—C1A	−175.56 (10)	C6B—C1B—C14B—C13B	1.00 (15)
C8A—C13A—C14A—C1A	0.95 (15)	C2B—C1B—C14B—C13B	179.20 (10)
C12A—C13A—C14A—C15A	3.81 (16)	C6B—C1B—C14B—C15B	−178.15 (9)
C8A—C13A—C14A—C15A	−179.68 (9)	C2B—C1B—C14B—C15B	0.05 (16)
C2A—C1A—C14A—C13A	179.07 (10)	C12B—C13B—C14B—C1B	176.41 (10)
C6A—C1A—C14A—C13A	−2.53 (15)	C8B—C13B—C14B—C1B	−2.36 (15)
C2A—C1A—C14A—C15A	−0.31 (15)	C12B—C13B—C14B—C15B	−4.45 (16)
C6A—C1A—C14A—C15A	178.08 (9)	C8B—C13B—C14B—C15B	176.78 (10)
C13A—C14A—C15A—C16A	52.54 (15)	C1B—C14B—C15B—C16B	125.91 (12)
C1A—C14A—C15A—C16A	−128.08 (12)	C13B—C14B—C15B—C16B	−53.23 (16)
C14A—C15A—C16A—C17A	177.64 (10)	C14B—C15B—C16B—C17B	−179.49 (10)
C15A—C16A—C17A—O1A	18.56 (16)	C15B—C16B—C17B—O1B	−17.28 (17)
C15A—C16A—C17A—C18A	−159.78 (10)	C15B—C16B—C17B—C18B	162.91 (10)
O1A—C17A—C18A—C23A	−178.62 (10)	O1B—C17B—C18B—C23B	174.78 (10)
C16A—C17A—C18A—C23A	−0.30 (15)	C16B—C17B—C18B—C23B	−5.41 (15)
O1A—C17A—C18A—C19A	−0.13 (15)	O1B—C17B—C18B—C19B	−5.71 (16)
C16A—C17A—C18A—C19A	178.20 (9)	C16B—C17B—C18B—C19B	174.10 (9)
C23A—C18A—C19A—O2A	−177.65 (10)	C23B—C18B—C19B—O2B	179.06 (9)
C17A—C18A—C19A—O2A	3.80 (15)	C17B—C18B—C19B—O2B	−0.47 (15)
C23A—C18A—C19A—C20A	1.96 (15)	C23B—C18B—C19B—C20B	0.22 (15)
C17A—C18A—C19A—C20A	−176.59 (10)	C17B—C18B—C19B—C20B	−179.31 (10)
O2A—C19A—C20A—C21A	177.39 (11)	O2B—C19B—C20B—C21B	−179.19 (10)
C18A—C19A—C20A—C21A	−2.24 (17)	C18B—C19B—C20B—C21B	−0.28 (16)
C19A—C20A—C21A—C22A	0.64 (18)	C19B—C20B—C21B—C22B	−0.11 (17)
C20A—C21A—C22A—C23A	1.23 (18)	C20B—C21B—C22B—C23B	0.56 (17)

C21A—C22A—C23A—C18A	−1.48 (17)	C21B—C22B—C23B—C18B	−0.63 (16)
C19A—C18A—C23A—C22A	−0.11 (16)	C19B—C18B—C23B—C22B	0.24 (15)
C17A—C18A—C23A—C22A	178.39 (10)	C17B—C18B—C23B—C22B	179.76 (10)

Hydrogen-bond geometry (Å, °)

*Cg*1, *Cg*3, *Cg*5, *Cg*6 and *Cg*7 are the centroids of the C1A–C6A, C8A–C13A, C1B–C6B, C1B/C6B–C8B/C13B–C14B and C8B–C13B rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2A—H1OA···O1A	0.93 (2)	1.69 (2)	2.5459 (12)	152.2 (19)
O2B—H1OB···O1B	0.88 (2)	1.75 (2)	2.5725 (13)	154.2 (19)
C5A—H5AA···Cg5	0.93	2.84	3.6754 (13)	151
C7A—H7AA···Cg6	0.93	2.76	3.6440 (12)	158
C9A—H9AA···Cg7	0.93	2.73	3.6325 (12)	164
C9B—H9BA···Cg1^{i}	0.93	2.76	3.4023 (12)	127
C23B—H23B···Cg3	0.93	2.91	3.7661 (11)	154

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2.

Fig. 1

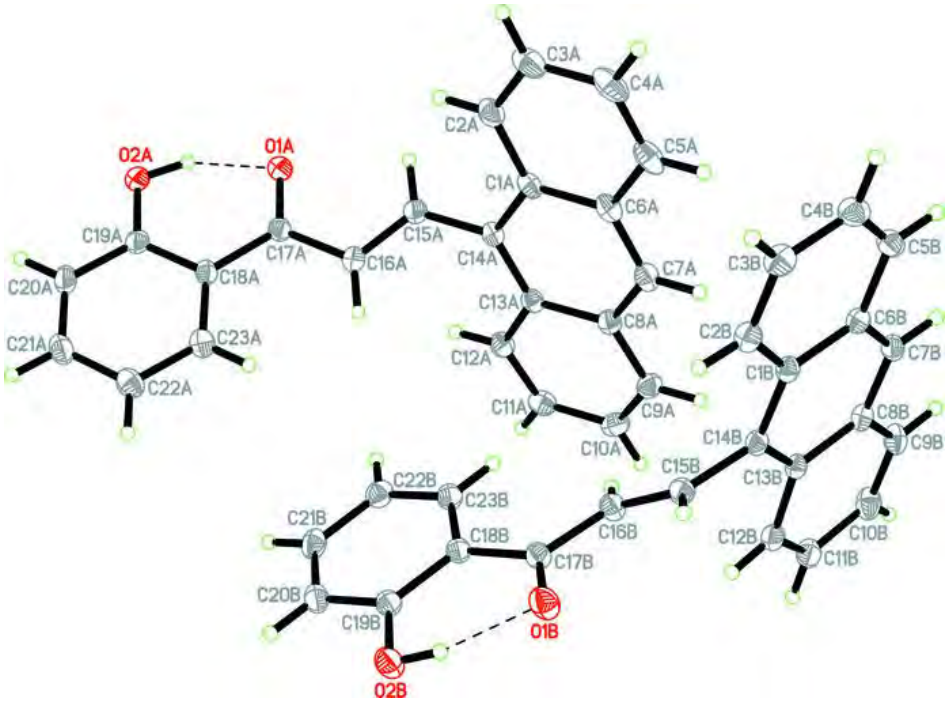
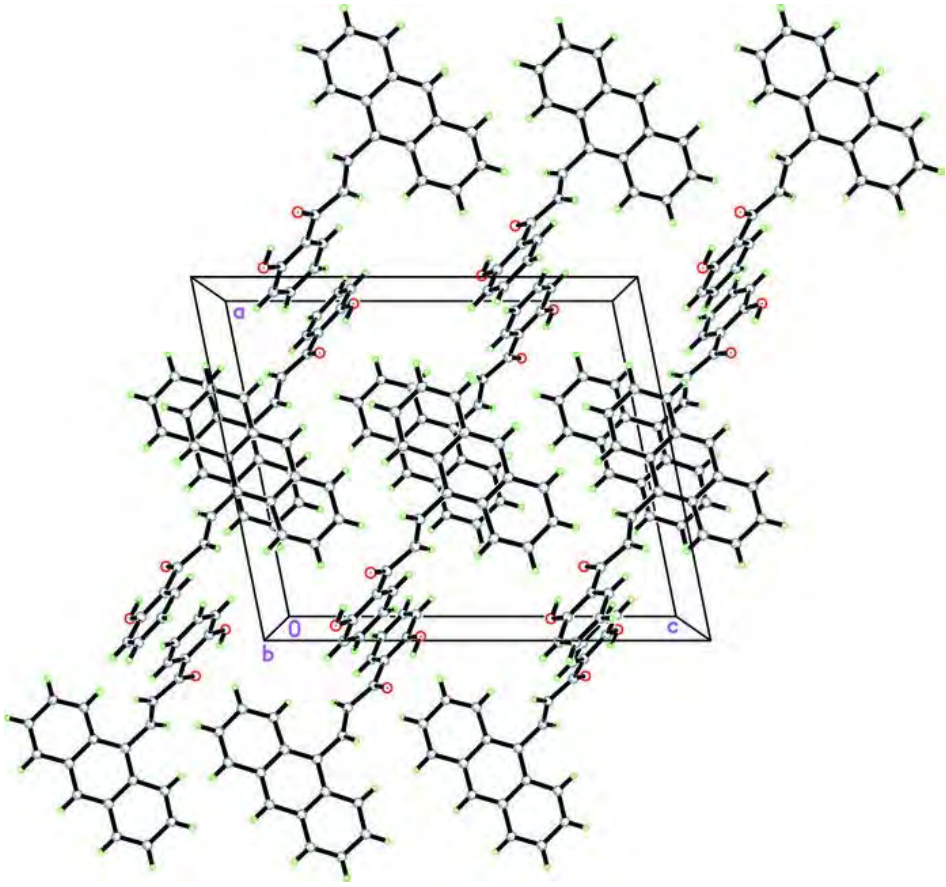


Fig. 2

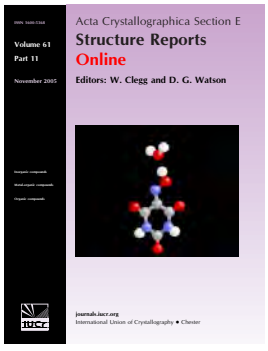


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-1-(2-Hydroxyphenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun, Thitipone Suwunwong, Kullapa Chanawanno, Pitikan Wisitsak and Suchada Chantrapromma

Acta Cryst. (2011). E**67**, o2287–o2288



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(2-Hydroxyphenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun,[a]*‡ Thitipone Suwunwong,[b] Kullapa Chanawanno,[b] Pitikan Wisitsak[c] and Suchada Chantrapromma[b]§

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [c]Excellence Center, Mae Fah Luang University, Thasud, Muang, Chaing Rai 57100, Thailand
Correspondence e-mail: hkfun@usm.my

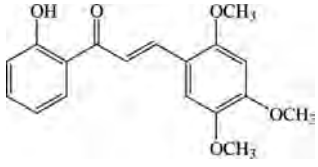
Received 26 July 2011; accepted 3 August 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.095; data-to-parameter ratio = 8.5.

In the title chalcone derivative, $C_{18}H_{18}O_5$, the dihedral angle between the hydroxy-substituted benzene ring and the trimethoxy-substituted benzene ring is 16.3 (1)°. The three methoxy groups are essentially coplanar with the benzene ring to which they are attached, with an r.m.s. deviation of 0.0208 Å. An intramolecular O—H···O hydrogen bond generates an S(6) ring motif. In the crystal, weak C—H···O interactions link molecules into helical chains along the b axis. These chains are connected into sheets parallel to the bc plane by further weak C—H···O interactions.

Related literature

For background to and applications of chalcones, see: Boeck *et al.* (2006); Cheng *et al.* (2008); Hatayama *et al.* (2010); Jung *et al.* (2008); Lee *et al.* (2006); Liu *et al.* (2011); Nerya *et al.* (2004); Patil & Dharmaprakash (2008); Saydam *et al.* (2003); Tewtrakul *et al.* (2003). For related structures, see: Suwunwong *et al.* (2009); Fun *et al.* (2010). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986). For standard bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{18}H_{18}O_5$
M_r = 314.32
Orthorhombic, $P2_12_12_1$
a = 4.2891 (2) Å
b = 17.3341 (9) Å
c = 20.5732 (10) Å
V = 1529.57 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.56 × 0.16 × 0.14 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.946, T_{max} = 0.986
16077 measured reflections
2392 independent reflections
1946 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.095
S = 1.08
2392 reflections
280 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*1···O2	0.89 (3)	1.73 (3)	2.541 (2)	152 (2)
C5—H5*A*···O5^{i}	0.96 (2)	2.57 (2)	3.254 (3)	129.0 (18)
C16—H16*C*···O1^{ii}	1.04 (3)	2.42 (3)	3.446 (3)	167.5 (18)

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, -z$; (ii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (grant No. RSA5280033) and the Prince of Songkla University for financial support. The authors also thank Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160. KC thanks the Crystal Materials Research Unit, Prince of Songkla University, for a Research Assistance fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5295).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Boeck, P., Falcão, C. A. B., Leal, P. C., Yunes, R. A., Filho, V. C., Torres-Santos, E. C. & Rossi-Bergmann, B. (2006). *Bioorg. Med. Chem.* **14**, 1538–1545.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cheng, J.-H., Hung, C.-F., Yang, S.-C., Wang, J.-P., Won, S.-J. & Lin, C.-N. (2008). *Bioorg. Med. Chem.* **16**, 7270–7276.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Fun, H.-K., Kobkeatthawin, T., Ruanwas, P. & Chantrapromma, S. (2010). *Acta Cryst.* E**66**, o1973–o1974.

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Hatayama, M., Unno, H., Kusunoki, M., Takahashi, S., Nishino, T. & Nakayama, T. (2010). *J. Biosci. Bioeng.* **110**, 158–164.
Jung, Y. J., Son, K. I., Oh, Y. E. & Noh, D. Y. (2008). *Polyhedron*, **27**, 861–867.
Lee, S. H., Seo, G. S., Kim, J. Y., Jin, X. Y., Kim, H.-D. & Sohn, D. H. (2006). *Eur. J. Pharmacol.* **532**, 178–186.
Liu, X.-F., Zheng, C.-J., Sun, L.-P., Liu, X.-K. & Piao, H.-R. (2011). *Eur. J. Med. Chem.* **46**, 3469–3473.
Nerya, O., Musa, R., Khatib, S., Tamir, S. & Vaya, J. (2004). *Phytochemistry*, **65**, 1389–1395.
Patil, P. S. & Dharmaprakash, S. M. (2008). *Mater. Lett.* **62**, 451–453.
Saydam, G., Aydin, H. H., Sahin, F., Kucukoglu, O., Erciyas, E., Terzioglu, E., Buyukkececi, F. & Omaya, S. B. (2003). *Leuk. Res.* **27**, 57–64.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suwunwong, T., Chantrapromma, S. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o120.
Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol.* **25**, 503–508.

supplementary materials

Acta Cryst. (2011). E**67**, o2287-o2288 [**doi:10.1107/S1600536811031382**]

(*E*)-1-(2-Hydroxyphenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

H.-K. Fun, T. Suwunwong, K. Chanawanno, P. Wisitsak and S. Chantrapromma

Comment

Chalcones or 1,3-diphenyl-2-propen-1-ones are commonly found in the natural products (Hatayama *et al.*, 2010). They have a wide range of applications including non-linear optical effects (Patil & Dharmaprakash, 2008) in fluorescent materials (Jung *et al.*, 2008) and have biological activities such as antibacterial (Liu *et al.*, 2011), anti-inflammatory (Lee *et al.*, 2006), antileishmanial (Boeck *et al.*, 2006), cytotoxic (Saydam *et al.*, 2003), anti-oxidant (Cheng *et al.*, 2008), HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003) as well as anti-tyrosinase activities (Nerya *et al.*, 2004). The various interesting applications of chalcones lead us to synthesize the title chalcone derivative in order to study its tyrosinase inhibitory activity and also to compare its properies with the previously published related compounds (Suwunwong *et al.*, 2009; Fun *et al.*, 2010). Our experiment shows that (I) exhibits tyrosinase inhibitory activity with the IC_{50} value of 0.075 ± 0.000 mg ml^{-1}. Its tyrosinase inhibitory activity is therefore about 0.08 times that of the standard anti-tyrosinase kojic acid. Herein the crystal structure of (I) is reported.

The molecule of (I) in Fig. 1 exists in an *E* configuration with respect to the C8═C9 double bond [1.343 (3) Å]. The molecule is twisted with the dihedral angle between the 2-hydroxyphenyl and the 2,4,5-trimethoxyphenyl benzene rings being 16.3 (1)°. The middle prop-2-en-1-one unit (O2/C7–C9) is slightly twisted with the torsion angle O2–C7–C8–C9 = -8.8 (3)°. The mean plane through this unit makes dihedral angles of 13.48 (14)° and 2.85 (14)° with the 2-hydroxyphenyl and the 2,4,5-trimethoxyphenyl benzene rings, respectively. The three methoxy groups of 2,4,5-trimethoxyphenyl unit are essentially co-planar with the attached benzene ring with torsion angles C16–O3–C11–C12 = -1.0 (3)°, C17–O4–C13–C12 = -2.5 (3)° and C18–O5–C14–C15 = 3.1 (3)°. An O1—H1O1···O2 intramolecular hydrogen bond generates an S(6) ring motif (Bernstein *et al.*, 1995) (Table 1). The bond distances have of normal values (Allen *et al.*, 1987) and are comparable with closely related structures (Suwunwong *et al.*, 2009; Fun *et al.*, 2010).

In the crystal packing (Fig. 2), weak C16—H16C···$O1^{ii}$ interactions (Table 1) link the molecules into helical chains along the *b* axis. These chains are further connected by weak C5—H5A···$O5^{i}$ interactions (Table 1) into supramolecular sheets parallel to the *bc* plane and stacked along the *a* axis.

Experimental

The title compound was prepared by stirring the mixed solution of 2-hydroxyacetophenone (0.24 ml, 2 mmol) and 2,4,5-trimethoxybenzaldehyde (0.40 g, 2 mmol) in ethanol (30 ml) in the presence of 10% NaOH(aq) (5 ml). After 4 h of stirring at room temperature, the orange solid was obtained and was then collected by filtration, washed with distilled water, dried and purified by recrystallization from hot acetone. Yellow block-shaped single crystals suitable for *x*-ray structure determination were grown over a period of several days by slow evaporation of the acetone/ethanol (1:1 *v*/*v*) solvent at room temperature, Mp. 404–405 K.

Refinement

All H atoms were located in difference Fourier maps and refined isotropically. The highest residual electron density peak is located at 0.78 Å from C14 and the deepest hole is located at 0.71 Å from C12. A total of 1662 Friedel pairs were merged before final refinement as there are no significant anomalous dispersion effects to determine the absolute configuration.

Figures

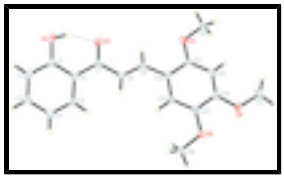


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids. An intramolecular O—H···O hydrogen bonds is shown as dashed line.

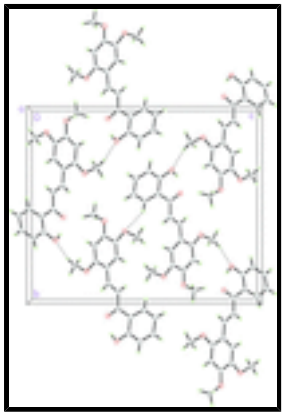


Fig. 2. The crystal packing of the title compound, showing supramolecular sheets. Hydrogen bonds are shown as dashed lines.

(*E*)-1-(2-Hydroxyphenyl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{18}H_{18}O_5$	D_x = 1.365 Mg m^{-3}
M_r = 314.32	Melting point = 404–405 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 2392 reflections
a = 4.2891 (2) Å	θ = 2.0–29.0°
b = 17.3341 (9) Å	μ = 0.10 mm^{-1}
c = 20.5732 (10) Å	T = 100 K
V = 1529.57 (13) Å^3	Block, yellow
Z = 4	0.56 × 0.16 × 0.14 mm
F(000) = 664	

Data collection

Bruker APEXII CCD area-detector diffractometer	2392 independent reflections
Radiation source: sealed tube	1946 reflections with $I > 2\sigma(I)$

graphite

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

T_{min} = 0.946, T_{max} = 0.986

16077 measured reflections

R_{int} = 0.045

θ_{max} = 29.0°, θ_{min} = 2.0°

h = −5→5

k = −23→17

l = −24→27

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.041

$wR(F^2)$ = 0.095

S = 1.08

2392 reflections

280 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.2277P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.25 e Å^{-3}

$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.2483 (4)	0.67956 (9)	0.11729 (7)	0.0294 (4)
O2	0.0456 (4)	0.56059 (8)	0.15537 (6)	0.0277 (4)
O3	0.5147 (4)	0.34185 (8)	0.25185 (6)	0.0239 (4)
O4	0.3357 (4)	0.08107 (8)	0.17331 (7)	0.0241 (4)
O5	−0.0186 (4)	0.13159 (7)	0.08358 (6)	0.0228 (4)
C1	−0.3401 (5)	0.63667 (11)	0.06566 (9)	0.0205 (5)
C2	−0.5266 (6)	0.67134 (12)	0.01880 (10)	0.0245 (5)
C3	−0.6134 (6)	0.63167 (13)	−0.03616 (11)	0.0267 (5)
C4	−0.5151 (6)	0.55587 (12)	−0.04557 (10)	0.0254 (5)
C5	−0.3396 (5)	0.52027 (12)	0.00162 (10)	0.0214 (5)

C6	−0.2498 (5)	0.55849 (11)	0.05878 (9)	0.0181 (4)
C7	−0.0708 (5)	0.52102 (11)	0.11099 (9)	0.0194 (4)
C8	−0.0393 (6)	0.43673 (11)	0.11163 (9)	0.0189 (4)
C9	0.1472 (5)	0.40085 (12)	0.15445 (10)	0.0197 (5)
C10	0.1985 (5)	0.31843 (11)	0.16063 (9)	0.0178 (4)
C11	0.3882 (5)	0.28887 (11)	0.21024 (9)	0.0188 (4)
C12	0.4385 (5)	0.20945 (12)	0.21658 (9)	0.0197 (5)
C13	0.3018 (5)	0.15894 (11)	0.17306 (9)	0.0183 (4)
C14	0.1069 (5)	0.18730 (11)	0.12307 (9)	0.0178 (4)
C15	0.0587 (5)	0.26522 (11)	0.11777 (9)	0.0174 (4)
C16	0.7030 (6)	0.31494 (14)	0.30485 (10)	0.0256 (5)
C17	0.5405 (6)	0.04847 (13)	0.22131 (12)	0.0267 (5)
C18	−0.2027 (6)	0.15760 (14)	0.03089 (11)	0.0236 (5)
H2A	−0.586 (6)	0.7230 (14)	0.0249 (10)	0.033 (7)*
H3A	−0.739 (7)	0.6549 (13)	−0.0687 (10)	0.029 (6)*
H4A	−0.557 (6)	0.5303 (12)	−0.0823 (10)	0.021 (6)*
H5A	−0.280 (6)	0.4677 (13)	−0.0057 (10)	0.029 (6)*
H8A	−0.163 (6)	0.4083 (11)	0.0806 (9)	0.015 (5)*
H9A	0.258 (6)	0.4335 (12)	0.1827 (9)	0.022 (6)*
H12A	0.568 (6)	0.1911 (12)	0.2488 (9)	0.019 (6)*
H15A	−0.079 (6)	0.2835 (12)	0.0829 (9)	0.020 (6)*
H16A	0.766 (7)	0.3640 (14)	0.3280 (10)	0.036 (7)*
H16B	0.890 (6)	0.2889 (13)	0.2889 (10)	0.028 (6)*
H16C	0.574 (7)	0.2787 (14)	0.3348 (10)	0.033 (6)*
H17A	0.467 (7)	0.0620 (13)	0.2662 (11)	0.040 (7)*
H17B	0.757 (7)	0.0676 (13)	0.2147 (11)	0.031 (7)*
H17C	0.539 (6)	−0.0083 (13)	0.2145 (9)	0.027 (6)*
H18A	−0.274 (6)	0.1128 (13)	0.0074 (9)	0.023 (6)*
H18B	−0.088 (6)	0.1914 (12)	0.0019 (10)	0.021 (6)*
H18C	−0.394 (7)	0.1848 (14)	0.0468 (10)	0.032 (7)*
H1O1	−0.123 (7)	0.6493 (15)	0.1401 (11)	0.042 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0418 (10)	0.0179 (8)	0.0286 (8)	0.0059 (8)	−0.0036 (9)	−0.0062 (7)
O2	0.0388 (10)	0.0186 (7)	0.0257 (7)	0.0033 (8)	−0.0077 (8)	−0.0047 (6)
O3	0.0308 (9)	0.0202 (7)	0.0207 (7)	−0.0013 (8)	−0.0069 (7)	−0.0022 (6)
O4	0.0311 (9)	0.0154 (7)	0.0256 (7)	0.0029 (7)	−0.0058 (7)	0.0032 (6)
O5	0.0307 (9)	0.0168 (7)	0.0208 (7)	0.0004 (7)	−0.0068 (7)	−0.0008 (5)
C1	0.0220 (11)	0.0172 (10)	0.0222 (10)	−0.0009 (9)	0.0066 (9)	0.0001 (8)
C2	0.0265 (12)	0.0155 (10)	0.0316 (11)	0.0015 (10)	0.0039 (10)	0.0023 (9)
C3	0.0269 (13)	0.0248 (12)	0.0283 (12)	0.0010 (10)	−0.0028 (11)	0.0085 (9)
C4	0.0311 (13)	0.0229 (11)	0.0221 (10)	−0.0027 (11)	−0.0029 (11)	0.0003 (9)
C5	0.0263 (12)	0.0151 (10)	0.0226 (10)	0.0002 (10)	0.0029 (10)	0.0003 (8)
C6	0.0188 (11)	0.0149 (10)	0.0206 (9)	−0.0012 (9)	0.0045 (9)	0.0019 (8)
C7	0.0211 (11)	0.0182 (10)	0.0189 (9)	−0.0001 (9)	0.0050 (9)	−0.0012 (8)
C8	0.0225 (11)	0.0167 (10)	0.0176 (9)	−0.0014 (9)	0.0023 (9)	−0.0011 (8)

C9	0.0223 (11)	0.0189 (10)	0.0178 (9)	−0.0022 (9)	0.0045 (9)	−0.0028 (8)
C10	0.0207 (10)	0.0181 (10)	0.0146 (9)	0.0022 (9)	0.0026 (9)	0.0001 (8)
C11	0.0204 (11)	0.0195 (10)	0.0164 (9)	−0.0009 (9)	0.0036 (9)	−0.0020 (8)
C12	0.0206 (11)	0.0228 (11)	0.0156 (9)	0.0023 (9)	−0.0003 (9)	0.0030 (8)
C13	0.0208 (11)	0.0153 (10)	0.0188 (9)	0.0013 (9)	0.0037 (9)	0.0034 (8)
C14	0.0205 (11)	0.0168 (10)	0.0162 (9)	−0.0006 (9)	0.0027 (9)	−0.0014 (8)
C15	0.0190 (10)	0.0193 (10)	0.0139 (9)	0.0015 (9)	0.0008 (9)	0.0021 (8)
C16	0.0287 (13)	0.0278 (13)	0.0202 (11)	−0.0017 (11)	−0.0052 (10)	0.0018 (9)
C17	0.0314 (14)	0.0196 (12)	0.0291 (12)	0.0059 (11)	−0.0038 (12)	0.0066 (9)
C18	0.0266 (13)	0.0217 (12)	0.0226 (11)	0.0003 (11)	−0.0066 (11)	−0.0003 (9)

Geometric parameters (Å, °)

O1—C1	1.355 (2)	C8—C9	1.343 (3)
O1—H1O1	0.89 (3)	C8—H8A	0.96 (2)
O2—C7	1.246 (2)	C9—C10	1.451 (3)
O3—C11	1.368 (2)	C9—H9A	0.94 (2)
O3—C16	1.435 (3)	C10—C11	1.402 (3)
O4—C13	1.358 (2)	C10—C15	1.410 (3)
O4—C17	1.437 (3)	C11—C12	1.400 (3)
O5—C14	1.372 (2)	C12—C13	1.383 (3)
O5—C18	1.415 (3)	C12—H12A	0.92 (2)
C1—C2	1.389 (3)	C13—C14	1.413 (3)
C1—C6	1.417 (3)	C14—C15	1.371 (3)
C2—C3	1.375 (3)	C15—H15A	0.98 (2)
C2—H2A	0.94 (2)	C16—H16A	1.01 (2)
C3—C4	1.394 (3)	C16—H16B	0.98 (3)
C3—H3A	0.95 (2)	C16—H16C	1.04 (2)
C4—C5	1.375 (3)	C17—H17A	1.00 (2)
C4—H4A	0.89 (2)	C17—H17B	1.00 (3)
C5—C6	1.404 (3)	C17—H17C	0.99 (2)
C5—H5A	0.96 (2)	C18—H18A	0.96 (2)
C6—C7	1.471 (3)	C18—H18B	0.97 (2)
C7—C8	1.467 (3)	C18—H18C	1.00 (3)
C1—O1—H1O1	105.5 (16)	O3—C11—C12	122.76 (18)
C11—O3—C16	118.72 (16)	O3—C11—C10	116.12 (17)
C13—O4—C17	117.32 (16)	C12—C11—C10	121.11 (19)
C14—O5—C18	116.65 (16)	C13—C12—C11	119.82 (19)
O1—C1—C2	118.27 (18)	C13—C12—H12A	120.2 (13)
O1—C1—C6	121.59 (19)	C11—C12—H12A	119.9 (13)
C2—C1—C6	120.13 (19)	O4—C13—C12	125.57 (18)
C3—C2—C1	120.7 (2)	O4—C13—C14	114.32 (17)
C3—C2—H2A	120.9 (14)	C12—C13—C14	120.11 (17)
C1—C2—H2A	118.4 (14)	C15—C14—O5	125.95 (18)
C2—C3—C4	120.3 (2)	C15—C14—C13	119.32 (18)
C2—C3—H3A	121.4 (14)	O5—C14—C13	114.72 (17)
C4—C3—H3A	118.3 (14)	C14—C15—C10	122.06 (19)
C5—C4—C3	119.4 (2)	C14—C15—H15A	117.9 (12)
C5—C4—H4A	118.9 (15)	C10—C15—H15A	120.1 (12)

C3—C4—H4A	121.6 (14)	O3—C16—H16A	103.7 (14)
C4—C5—C6	122.0 (2)	O3—C16—H16B	111.0 (13)
C4—C5—H5A	117.5 (13)	H16A—C16—H16B	109 (2)
C6—C5—H5A	120.5 (13)	O3—C16—H16C	110.3 (14)
C5—C6—C1	117.40 (18)	H16A—C16—H16C	111.9 (18)
C5—C6—C7	123.12 (18)	H16B—C16—H16C	111 (2)
C1—C6—C7	119.48 (17)	O4—C17—H17A	110.4 (16)
O2—C7—C8	120.31 (18)	O4—C17—H17B	110.2 (14)
O2—C7—C6	120.05 (17)	H17A—C17—H17B	110 (2)
C8—C7—C6	119.61 (18)	O4—C17—H17C	106.8 (14)
C9—C8—C7	121.5 (2)	H17A—C17—H17C	111.1 (18)
C9—C8—H8A	121.7 (12)	H17B—C17—H17C	108 (2)
C7—C8—H8A	116.9 (12)	O5—C18—H18A	107.7 (13)
C8—C9—C10	127.1 (2)	O5—C18—H18B	112.3 (14)
C8—C9—H9A	115.3 (13)	H18A—C18—H18B	109.8 (17)
C10—C9—H9A	117.5 (13)	O5—C18—H18C	110.9 (13)
C11—C10—C15	117.57 (18)	H18A—C18—H18C	107 (2)
C11—C10—C9	120.77 (18)	H18B—C18—H18C	109.4 (19)
C15—C10—C9	121.66 (18)		
O1—C1—C2—C3	176.7 (2)	C16—O3—C11—C10	178.07 (19)
C6—C1—C2—C3	−3.1 (3)	C15—C10—C11—O3	−178.60 (18)
C1—C2—C3—C4	0.0 (3)	C9—C10—C11—O3	0.9 (3)
C2—C3—C4—C5	2.2 (3)	C15—C10—C11—C12	0.5 (3)
C3—C4—C5—C6	−1.4 (3)	C9—C10—C11—C12	179.9 (2)
C4—C5—C6—C1	−1.6 (3)	O3—C11—C12—C13	179.56 (19)
C4—C5—C6—C7	177.9 (2)	C10—C11—C12—C13	0.6 (3)
O1—C1—C6—C5	−176.0 (2)	C17—O4—C13—C12	−2.5 (3)
C2—C1—C6—C5	3.9 (3)	C17—O4—C13—C14	177.65 (18)
O1—C1—C6—C7	4.4 (3)	C11—C12—C13—O4	179.0 (2)
C2—C1—C6—C7	−175.7 (2)	C11—C12—C13—C14	−1.2 (3)
C5—C6—C7—O2	168.3 (2)	C18—O5—C14—C15	3.1 (3)
C1—C6—C7—O2	−12.2 (3)	C18—O5—C14—C13	−176.94 (19)
C5—C6—C7—C8	−13.8 (3)	O4—C13—C14—C15	−179.32 (19)
C1—C6—C7—C8	165.8 (2)	C12—C13—C14—C15	0.9 (3)
O2—C7—C8—C9	−8.8 (3)	O4—C13—C14—O5	0.7 (3)
C6—C7—C8—C9	173.2 (2)	C12—C13—C14—O5	−179.08 (19)
C7—C8—C9—C10	179.7 (2)	O5—C14—C15—C10	−179.87 (19)
C8—C9—C10—C11	−176.5 (2)	C13—C14—C15—C10	0.2 (3)
C8—C9—C10—C15	2.9 (3)	C11—C10—C15—C14	−0.8 (3)
C16—O3—C11—C12	−1.0 (3)	C9—C10—C15—C14	179.7 (2)

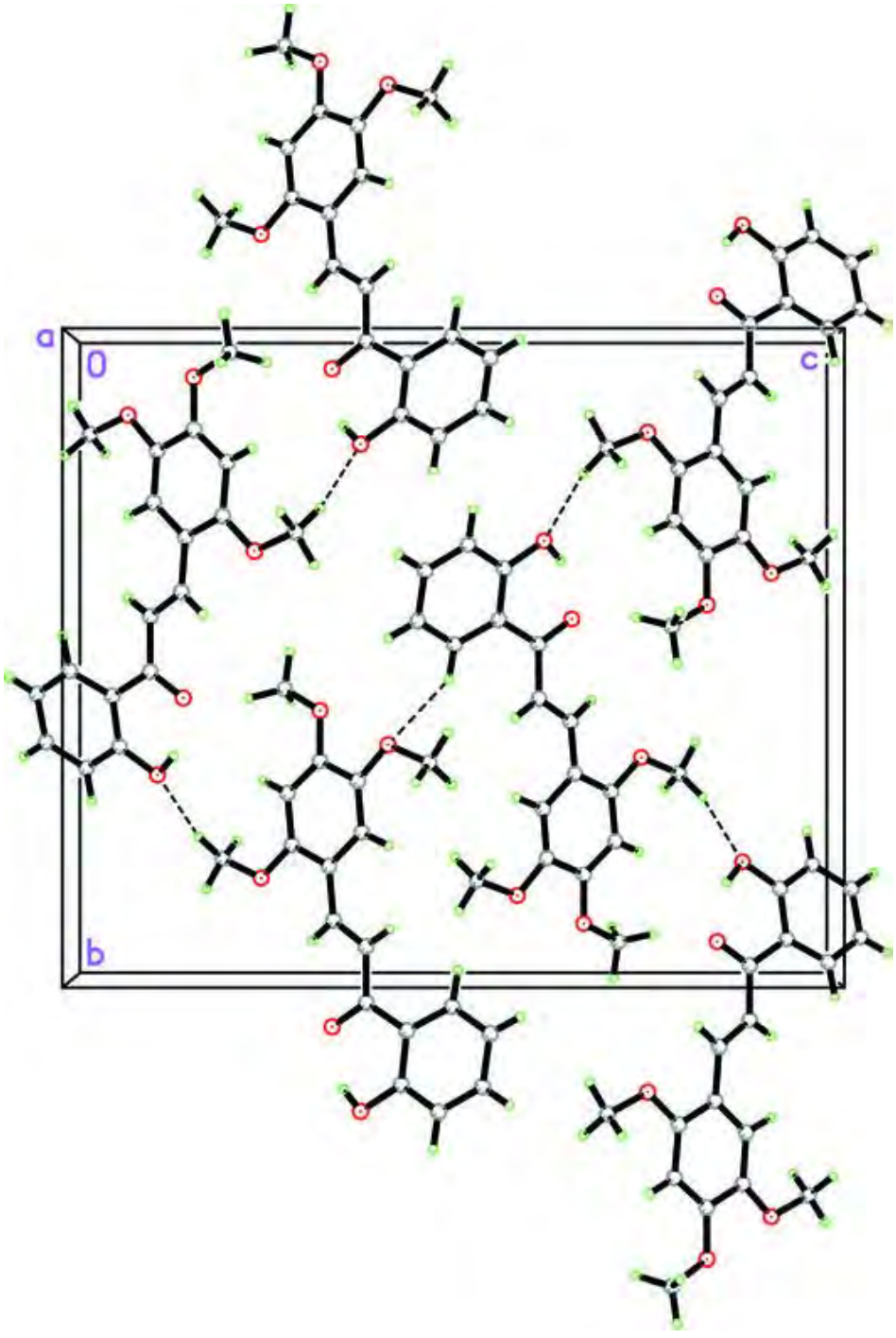
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O1···O2	0.89 (3)	1.73 (3)	2.541 (2)	152 (2)
C5—H5A···O5^{i}	0.96 (2)	2.57 (2)	3.254 (3)	129.0 (18)
C16—H16C···O1^{ii}	1.04 (3)	2.42 (3)	3.446 (3)	167.5 (18)

Symmetry codes: (i) *x*−1/2, −*y*+1/2, −*z*; (ii) −*x*, *y*−1/2, −*z*+1/2.

Fig. 1

Fig. 2

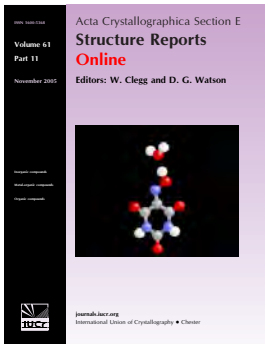


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-1-(Pyridin-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun, Thitipone Suwunwong and Suchada Chantrapromma

Acta Cryst. (2011). E**67**, o2406–o2407



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(Pyridin-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun,[a]*‡ Thitipone Suwunwong[b] and Suchada Chantrapromma[b]§

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

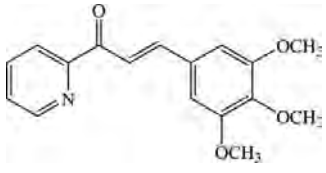
Received 11 August 2011; accepted 16 August 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.091; data-to-parameter ratio = 16.1.

In the title heteroaryl chalcone derivative, $C_{17}H_{17}NO_4$, the dihedral angle between the pyridine and benzene rings is 10.82 (5)°. The two methoxy groups at the *meta* positions are essentially coplanar with the attached benzene rings [C—O—C—C torsion angles = −0.97 (14) and 179.47 (9)°], whereas the methoxy group at the *para* position is twisted from the attached ring with a C—O—C—C torsion angle of −104.48 (11)°. A C—H···O close contact involving two of the methoxy groups generates an S(6) ring motif. In the crystal, molecules are linked by weak C—H···O interactions into columns along the b axis.

Related literature

For background and applications of chalcones, see: Gacche *et al.* (2008); Isomoto *et al.* (2005); Jung *et al.* (2008); Nowakowska *et al.* (2001); Patil & Dharmaprakash (2008); Shibata (1994); Tewtrakul *et al.* (2003). For standard bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Fun *et al.* (2010); Suwunwong, Chantrapromma & Fun (2009); Suwunwong, Chantrapromma, Pakdeevanich & Fun (2009). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{17}H_{17}NO_4$
M_r = 299.32
Orthorhombic, $Pna2_1$
a = 25.0498 (5) Å
b = 3.9799 (1) Å
c = 14.3267 (3) Å
V = 1428.31 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.58 × 0.41 × 0.32 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.945, T_{max} = 0.969
26700 measured reflections
3245 independent reflections
3130 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.091
S = 1.08
3245 reflections
202 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16B···O3^{i}	0.96	2.49	3.3358 (14)	147
C16—H16C···O4	0.96	2.57	3.0817 (15)	113

Symmetry code: (i) $x, y-1, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (grant No. RSA5280033) and Prince of Songkla University for the financial support. The authors also thank the Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5312).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Fun, H.-K., Suwunwong, T., Chantrapromma, S. & Karalai, C. (2010). *Acta Cryst.* E**66**, o3070–o3071.
Gacche, R. N., Dhole, N. A., Kamble, S. G. & Bandgar, B. P. (2008). *J. Enzyme Inhib. Med. Chem.* **23**, 28–31.
Isomoto, H., Furusu, H., Ohnita, K., Wen, C. Y., Inoue, K. & Kohno, S. (2005). *World J. Gastroenterol.* **11**, 1629–1633.
Jung, Y. J., Son, K. I., Oh, Y. E. & Noh, D. Y. (2008). *Polyhedron*, **27**, 861–867.
Nowakowska, Z., Wyrzykiewicz, E. & Kedzia, B. (2001). *Farmaco*, **56**, 325–329.

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Patil, P. S. & Dharmaprakash, S. M. (2008). *Mater. Lett.* **62**, 451–453.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shibata, S. (1994). *Stem Cells*, **12**, 44–52.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suwunwong, T., Chantrapromma, S. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o120.
Suwunwong, T., Chantrapromma, S., Pakdeevanich, P. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o1575–o1576.
Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol.* **25**, 503–508.

supplementary materials

Acta Cryst. (2011). E**67**, o2406-o2407 [**doi:10.1107/S1600536811033198**]

(*E*)-1-(Pyridin-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

H.-K. Fun, T. Suwunwong and S. Chantrapromma

Comment

Chalcones have a wide range of applications such as in non-linear optical devices (Patil & Dharmaprakash, 2008), electro-active fluorescent materials (Jung *et al.*, 2008), HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003) as well as various biological properties including antioxidant (Gacche *et al.*, 2008), antibacterial (Nowakowska *et al.*, 2001; Isomoto *et al.*, 2005), and anticancer activities (Shibata, 1994). The title heteroaryl chalcone derivative (I) was synthesized during the course of our study on biological and pharmacological properties of synthetic chalcones and heteroaryl chalcones. Our result shows that (I) possesses moderate analgesic activity. It was also tested for antibacterial activity and found to be inactive.

The molecule of the title heteroaryl chalcone derivative (Fig. 1) exists in an *E* configuration with respect to the C7═C8 double bond [1.3456 (14) Å] and the torsion angle C6–C7–C8–C9 = -177.30 (10)°. The molecule is twisted as the dihedral angle between pyridine and 3,4,5-trimethoxyphenyl rings is 10.82 (5)°. The propenone bridge (C6-C8/O1) is nearly planar with the torsion angle O1–C6–C7–C8 = -7.16 (18)°. The mean plane through this bridge makes dihedral angles of 10.37 (8)° and 3.63 (8)° with the planes of pyridine and benzene rings, respectively. The three methoxy substituents of 3,4,5-trimethoxyphenyl unit have two different orientations in which the two methoxy groups at the *meta*-positions are co-planar with torsion angles C15–O2–C11–C10 = -0.97 (14)° and C17–O4–C13–C12 = 179.47 (9)°, whereas the third group at the *para*-position is twisted out of plane with a torsion angle of C16–O3–C12–C11 = -104.48 (11)°. A weak C16—H16C···O4 intramolecular interaction generates S(6) ring motif (Bernstein *et al.*, 1995) (Table 1). The bond angles are of normal values (Allen *et al.*, 1987) and are comparable with the related structures (Fun *et al.*, 2010; Suwunwong, Chantrapromma & Fun, 2009; Suwunwong, Chantrapromma, Pakdeevanich & Fun, 2009).

In the crystal (Fig. 2), the molecules are linked by weak intemolecular C16—H16B···O3^{i} interactions (Table 1) into columns along the *b* axis.

Experimental

The title compound was synthesized by the condensation of 3,4,5-trimethoxybenzaldehyde (0.40 g, 2 mmol) with 2-acetylpyridine (0.20 g, 2 mmol) in ethanol (30 ml) in the presence of 30% NaOH(aq) (5 ml). After stirring in ice bath at 278 K for 3 h, the resulting pale yellow solid appeared and was then collected by filtration, washed with distilled water, dried and purified by repeated recrystallization from acetone. Pale yellow block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystalized from acetone/ethanol (1:1 v/v) by the slow evaporation of the solvent at room temperature after four days, Mp. 434-435 K.

Refinement

All H atoms were placed in calculated positions, with C-H = 0.93 Å, U_{iso} = 1.2U_{eq}(C) for aromatic and CH and C-H = 0.96 Å, U_{iso} = 1.5U_{eq}(C) for CH_3 atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.12 Å from C4 and the deepest hole is located at 0.28 Å from N1. A total of 2730

Friedel pairs were merged before final refinement as there is no significant anomalous dispersion for the determination of the absolute structure.

Figures

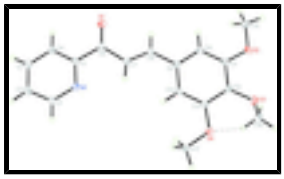


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. The dashed line indicates a weak intramolecular hydrogen bond.

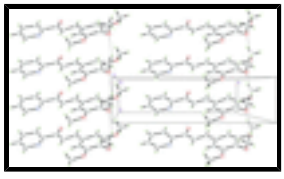


Fig. 2. The crystal packing of the title compound, showing columns along the *b* axis. Hydrogen bonds are shown as dashed lines.

(*E*)-1-(Pyridin-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{17}H_{17}NO_4$	D_x = 1.392 Mg m^{-3}
M_r = 299.32	Melting point = 434–435 K
Orthorhombic, *Pna*2$_1$	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 3245 reflections
a = 25.0498 (5) Å	θ = 2.2–35.0°
b = 3.9799 (1) Å	μ = 0.10 mm^{-1}
c = 14.3267 (3) Å	*T* = 100 K
V = 1428.31 (5) Å^3	Block, pale yellow
Z = 4	0.58 × 0.41 × 0.32 mm
F(000) = 632	

Data collection

Bruker APEXII CCD area-detector diffractometer	3245 independent reflections
Radiation source: sealed tube	3130 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.026
φ and ω scans	θ_{max} = 35.0°, θ_{min} = 2.2°
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	*h* = −34→40
T_{min} = 0.945, T_{max} = 0.969	*k* = −6→6
26700 measured reflections	*l* = −23→20

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods

Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.033$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.091$	H-atom parameters constrained
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 0.087P]$ where $P = (F_o^2 + 2F_c^2)/3$
3245 reflections	$(\Delta/\sigma)_{max} = 0.001$
202 parameters	$\Delta\rho_{max} = 0.37$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.24601 (4)	−0.1060 (3)	0.05761 (7)	0.02596 (19)
O2	0.08957 (3)	0.0024 (2)	0.50009 (5)	0.01754 (15)
O3	0.00155 (3)	0.3315 (2)	0.44825 (6)	0.01804 (15)
O4	−0.01556 (3)	0.5094 (2)	0.26892 (6)	0.01841 (15)
C4	0.23610 (4)	−0.0027 (3)	−0.13617 (8)	0.01727 (17)
H4A	0.2657	−0.1252	−0.1167	0.021*
C3	0.22725 (5)	0.0591 (3)	−0.23046 (8)	0.01872 (18)
H3A	0.2504	−0.0255	−0.2754	0.022*
C2	0.18322 (5)	0.2495 (3)	−0.25613 (7)	0.01913 (18)
H2A	0.1766	0.2978	−0.3186	0.023*
C1	0.14921 (4)	0.3668 (3)	−0.18655 (8)	0.01865 (19)
H1A	0.1198	0.4947	−0.2042	0.022*
N1	0.15656 (4)	0.3053 (2)	−0.09560 (6)	0.01632 (16)
C5	0.19958 (4)	0.1232 (2)	−0.07164 (7)	0.01393 (16)
C6	0.20623 (4)	0.0461 (3)	0.03063 (7)	0.01630 (17)
C7	0.16244 (4)	0.1520 (3)	0.09225 (7)	0.01565 (17)
H7A	0.1354	0.2880	0.0687	0.019*
C8	0.16076 (4)	0.0553 (3)	0.18220 (7)	0.01548 (16)
H8A	0.1893	−0.0724	0.2037	0.019*
C9	0.11859 (4)	0.1310 (3)	0.24945 (7)	0.01371 (16)

C10	0.12577 (4)	0.0284 (3)	0.34211 (7)	0.01412 (16)
H10A	0.1570	−0.0816	0.3596	0.017*
C11	0.08615 (4)	0.0913 (2)	0.40822 (6)	0.01337 (16)
C12	0.03898 (4)	0.2565 (3)	0.38196 (7)	0.01385 (15)
C13	0.03186 (4)	0.3557 (2)	0.28851 (7)	0.01376 (16)
C14	0.07153 (4)	0.2953 (3)	0.22275 (7)	0.01422 (16)
H14A	0.0669	0.3638	0.1612	0.017*
C15	0.13684 (5)	−0.1702 (3)	0.52820 (8)	0.01924 (18)
H15A	0.1339	−0.2324	0.5927	0.029*
H15B	0.1672	−0.0258	0.5199	0.029*
H15C	0.1412	−0.3686	0.4909	0.029*
C16	−0.04469 (5)	0.1175 (3)	0.44731 (9)	0.02059 (19)
H16A	−0.0727	0.2196	0.4831	0.031*
H16B	−0.0357	−0.0964	0.4740	0.031*
H16C	−0.0565	0.0864	0.3841	0.031*
C17	−0.02386 (5)	0.6161 (3)	0.17457 (8)	0.01905 (19)
H17A	−0.0580	0.7245	0.1695	0.029*
H17B	−0.0229	0.4244	0.1339	0.029*
H17C	0.0038	0.7709	0.1570	0.029*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0183 (4)	0.0404 (5)	0.0192 (4)	0.0113 (3)	0.0019 (3)	0.0060 (4)
O2	0.0191 (4)	0.0228 (4)	0.0107 (3)	0.0004 (3)	−0.0005 (2)	0.0022 (2)
O3	0.0168 (3)	0.0225 (3)	0.0148 (3)	−0.0011 (3)	0.0040 (3)	−0.0044 (3)
O4	0.0152 (3)	0.0256 (4)	0.0144 (3)	0.0049 (3)	0.0000 (3)	0.0018 (3)
C4	0.0158 (4)	0.0190 (4)	0.0171 (4)	0.0019 (3)	0.0036 (3)	0.0001 (3)
C3	0.0215 (5)	0.0190 (4)	0.0157 (4)	−0.0015 (3)	0.0065 (4)	−0.0015 (3)
C2	0.0220 (5)	0.0218 (4)	0.0136 (4)	−0.0027 (4)	0.0009 (3)	0.0001 (3)
C1	0.0178 (4)	0.0238 (5)	0.0144 (4)	0.0007 (3)	−0.0011 (3)	0.0019 (3)
N1	0.0134 (3)	0.0218 (4)	0.0137 (3)	0.0011 (3)	0.0000 (3)	0.0012 (3)
C5	0.0126 (4)	0.0168 (4)	0.0124 (3)	−0.0001 (3)	0.0017 (3)	0.0006 (3)
C6	0.0136 (4)	0.0210 (4)	0.0143 (4)	0.0013 (3)	0.0012 (3)	0.0012 (3)
C7	0.0140 (4)	0.0203 (4)	0.0126 (3)	0.0018 (3)	0.0013 (3)	0.0003 (3)
C8	0.0146 (4)	0.0188 (4)	0.0131 (4)	0.0012 (3)	0.0006 (3)	0.0003 (3)
C9	0.0132 (4)	0.0162 (4)	0.0117 (3)	−0.0009 (3)	0.0002 (3)	0.0000 (3)
C10	0.0133 (4)	0.0178 (4)	0.0112 (3)	0.0003 (3)	−0.0006 (3)	0.0003 (3)
C11	0.0149 (4)	0.0149 (4)	0.0103 (3)	−0.0014 (3)	−0.0004 (3)	−0.0001 (3)
C12	0.0144 (4)	0.0157 (4)	0.0114 (3)	−0.0005 (3)	0.0003 (3)	−0.0014 (3)
C13	0.0129 (4)	0.0157 (4)	0.0127 (3)	0.0003 (3)	−0.0005 (3)	−0.0011 (3)
C14	0.0130 (4)	0.0179 (4)	0.0118 (3)	−0.0001 (3)	−0.0001 (3)	0.0003 (3)
C15	0.0227 (5)	0.0193 (4)	0.0157 (4)	−0.0006 (3)	−0.0038 (4)	0.0031 (3)
C16	0.0180 (4)	0.0202 (4)	0.0235 (5)	−0.0009 (3)	0.0060 (4)	0.0008 (4)
C17	0.0177 (4)	0.0226 (5)	0.0168 (4)	0.0018 (3)	−0.0027 (3)	0.0038 (3)

Geometric parameters (Å, °)

O1—C6	1.2285 (13)	C7—H7A	0.9300

O2—C11	1.3656 (12)	C8—C9	1.4611 (14)
O2—C15	1.4269 (14)	C8—H8A	0.9300
O3—C12	1.3676 (12)	C9—C10	1.4005 (14)
O3—C16	1.4376 (14)	C9—C14	1.4013 (14)
O4—C13	1.3654 (13)	C10—C11	1.3946 (14)
O4—C17	1.4320 (14)	C10—H10A	0.9300
C4—C3	1.3909 (16)	C11—C12	1.4034 (14)
C4—C5	1.3938 (14)	C12—C13	1.4072 (13)
C4—H4A	0.9300	C13—C14	1.3904 (14)
C3—C2	1.3877 (17)	C14—H14A	0.9300
C3—H3A	0.9300	C15—H15A	0.9600
C2—C1	1.3918 (16)	C15—H15B	0.9600
C2—H2A	0.9300	C15—H15C	0.9600
C1—N1	1.3386 (14)	C16—H16A	0.9600
C1—H1A	0.9300	C16—H16B	0.9600
N1—C5	1.3434 (13)	C16—H16C	0.9600
C5—C6	1.5061 (14)	C17—H17A	0.9600
C6—C7	1.4697 (14)	C17—H17B	0.9600
C7—C8	1.3456 (14)	C17—H17C	0.9600
C11—O2—C15	116.68 (9)	C11—C10—H10A	120.0
C12—O3—C16	114.63 (8)	C9—C10—H10A	120.0
C13—O4—C17	116.95 (8)	O2—C11—C10	124.28 (9)
C3—C4—C5	118.42 (10)	O2—C11—C12	115.64 (9)
C3—C4—H4A	120.8	C10—C11—C12	120.08 (9)
C5—C4—H4A	120.8	O3—C12—C11	119.55 (9)
C2—C3—C4	118.73 (9)	O3—C12—C13	120.83 (9)
C2—C3—H3A	120.6	C11—C12—C13	119.56 (9)
C4—C3—H3A	120.6	O4—C13—C14	124.05 (9)
C3—C2—C1	118.68 (10)	O4—C13—C12	115.58 (8)
C3—C2—H2A	120.7	C14—C13—C12	120.37 (9)
C1—C2—H2A	120.7	C13—C14—C9	119.80 (9)
N1—C1—C2	123.48 (10)	C13—C14—H14A	120.1
N1—C1—H1A	118.3	C9—C14—H14A	120.1
C2—C1—H1A	118.3	O2—C15—H15A	109.5
C1—N1—C5	117.24 (9)	O2—C15—H15B	109.5
N1—C5—C4	123.44 (9)	H15A—C15—H15B	109.5
N1—C5—C6	116.56 (8)	O2—C15—H15C	109.5
C4—C5—C6	119.96 (9)	H15A—C15—H15C	109.5
O1—C6—C7	123.89 (10)	H15B—C15—H15C	109.5
O1—C6—C5	119.75 (10)	O3—C16—H16A	109.5
C7—C6—C5	116.32 (9)	O3—C16—H16B	109.5
C8—C7—C6	121.12 (9)	H16A—C16—H16B	109.5
C8—C7—H7A	119.4	O3—C16—H16C	109.5
C6—C7—H7A	119.4	H16A—C16—H16C	109.5
C7—C8—C9	126.53 (9)	H16B—C16—H16C	109.5
C7—C8—H8A	116.7	O4—C17—H17A	109.5
C9—C8—H8A	116.7	O4—C17—H17B	109.5
C10—C9—C14	120.19 (9)	H17A—C17—H17B	109.5
C10—C9—C8	118.17 (9)	O4—C17—H17C	109.5

C14—C9—C8	121.63 (9)	H17A—C17—H17C	109.5
C11—C10—C9	120.00 (9)	H17B—C17—H17C	109.5
C5—C4—C3—C2	1.43 (16)	C15—O2—C11—C12	179.12 (9)
C4—C3—C2—C1	−0.96 (17)	C9—C10—C11—O2	−179.72 (9)
C3—C2—C1—N1	−0.13 (18)	C9—C10—C11—C12	0.18 (15)
C2—C1—N1—C5	0.71 (17)	C16—O3—C12—C11	−104.48 (11)
C1—N1—C5—C4	−0.19 (15)	C16—O3—C12—C13	78.28 (12)
C1—N1—C5—C6	−177.94 (10)	O2—C11—C12—O3	3.10 (14)
C3—C4—C5—N1	−0.88 (16)	C10—C11—C12—O3	−176.82 (9)
C3—C4—C5—C6	176.80 (10)	O2—C11—C12—C13	−179.63 (8)
N1—C5—C6—O1	−176.67 (11)	C10—C11—C12—C13	0.45 (14)
C4—C5—C6—O1	5.50 (16)	C17—O4—C13—C14	−0.67 (15)
N1—C5—C6—C7	5.36 (14)	C17—O4—C13—C12	179.47 (9)
C4—C5—C6—C7	−172.47 (10)	O3—C12—C13—O4	−3.86 (14)
O1—C6—C7—C8	−7.16 (18)	C11—C12—C13—O4	178.91 (9)
C5—C6—C7—C8	170.72 (10)	O3—C12—C13—C14	176.28 (9)
C6—C7—C8—C9	−177.30 (10)	C11—C12—C13—C14	−0.96 (14)
C7—C8—C9—C10	−175.49 (10)	O4—C13—C14—C9	−179.04 (9)
C7—C8—C9—C14	5.80 (17)	C12—C13—C14—C9	0.81 (14)
C14—C9—C10—C11	−0.33 (15)	C10—C9—C14—C13	−0.16 (15)
C8—C9—C10—C11	−179.06 (9)	C8—C9—C14—C13	178.52 (9)
C15—O2—C11—C10	−0.97 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16B···O3^{i}	0.96	2.49	3.3358 (14)	147
C16—H16C···O4	0.96	2.57	3.0817 (15)	113

Symmetry codes: (i) *x*, *y*−1, *z*.

Fig. 1

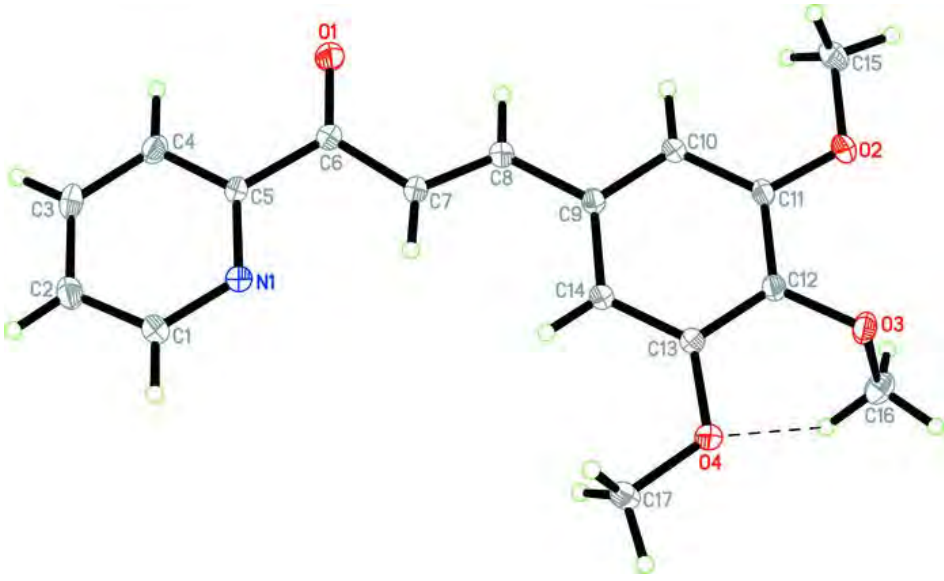
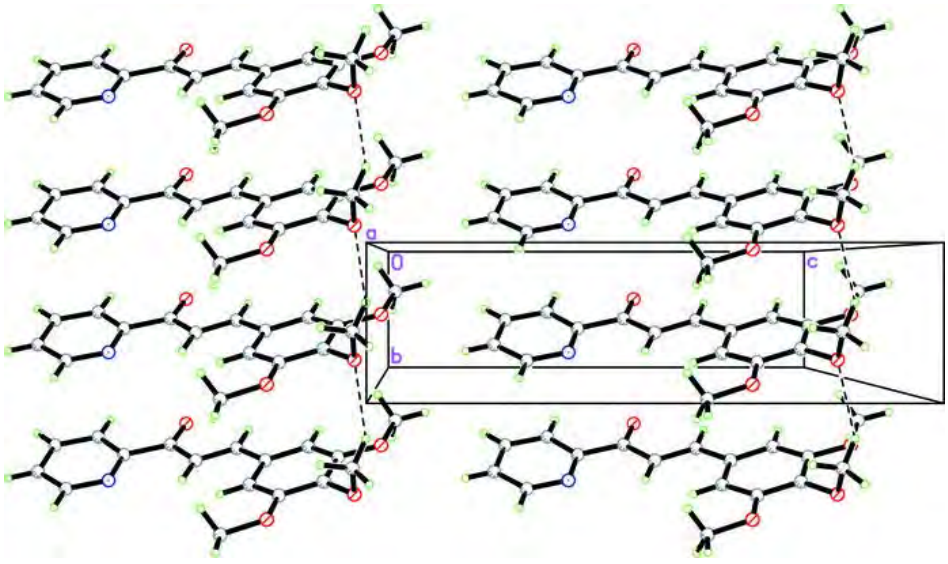


Fig. 2

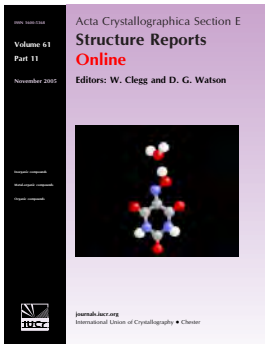


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, H. Stoeckli-Evans, E. R. T. Tiekink and M. Weil**

(2*E*)-1-(Pyridin-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun, Suchada Chantrapromma and Thitipone Suwunwong

Acta Cryst. (2011). E**67**, o2789–o2790



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*E*)-1-(Pyridin-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun,[a]*‡ Suchada Chantrapromma[b]§ and Thitipone Suwunwong[b]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

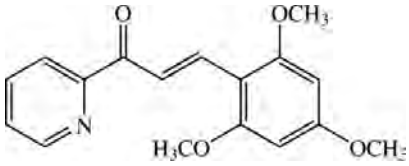
Received 19 September 2011; accepted 23 September 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.105; data-to-parameter ratio = 8.6.

The title heteroaryl chalcone derivative, $C_{17}H_{17}NO_4$, is a condensation product of 2-acetylpyridine and 2,4,6-trimethoxybenzaldehyde. The molecule is roughly planar, the dihedral angle between the pyridine and benzene rings being 5.51 (10)°. All the three methoxy groups are almost co-planar with the bound benzene ring [r.m.s. deviation of 0.0306 (2) Å]. A weak C—H···O intramolecular interaction involving one of the *ortho*-methoxy groups generates an S(6) ring motif. In the crystal, the molecules are linked by weak C—H···O interactions into anti-parallel face-to-face pairs. Adjacent pairs are further connected into sheets parallel to the *ab* plane.

Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Chantrapromma *et al.* (2009); Fun *et al.* (2010, 2011). For background to and applications of chalcones and heteroaryl chalcones, see: Bandgar *et al.* (2010); Gacche *et al.* (2008); Go *et al.* (2005); Isomoto *et al.* (2005); Jung *et al.* (2008); Suwunwong *et al.* (2011); Tewtrakul *et al.* (2003). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{17}H_{17}NO_4$
M_r = 299.32
Orthorhombic, *Fdd*2
a = 31.563 (2) Å
b = 44.508 (3) Å
c = 3.9504 (3) Å
V = 5549.6 (7) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.58 × 0.14 × 0.04 mm

Data collection

Bruker APEXII CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.943, T_{max} = 0.996
31465 measured reflections
2309 independent reflections
1908 reflections with $I > 2\sigma(I)$
R_{int} = 0.100

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.105
S = 1.09
2309 reflections
267 parameters
1 restraint
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3*A*···O2^{i}	1.00 (2)	2.45 (3)	3.369 (2)	157.8 (17)
C7—H11*B*···O4	0.97 (3)	2.31 (2)	2.835 (2)	113.1 (18)
C17—H17*B*···O4^{ii}	0.99 (2)	2.46 (2)	3.337 (3)	148 (2)

Symmetry codes: (i) $x+\frac{9}{4}, -y+\frac{9}{4}, z+\frac{1}{4}$; (ii) $-x+\frac{1}{2}, -y, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (grant No. RSA5280033) and Prince of Songkla University for financial support. They also thank Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160. Mr Teerasak Anantapong, Department of Biotechnology, Faculty of Agro-Industry, Prince of Songkla University, is acknowledged for the bacterial assay.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2642).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bandgar, B. P., Gawande, S. S., Bodade, R. G., Totre, J. V. & Khobragade, N. (2010). *Bioorg. Med. Chem.* **18**, 1364–1370.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chantrapromma, S., Suwunwong, T., Karalai, C. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o893–o894.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Fun, H.-K., Suwunwong, T. & Chantrapromma, S. (2011). *Acta Cryst.* E**67**, o2406–o2407.
Fun, H.-K., Suwunwong, T., Chantrapromma, S. & Karalai, C. (2010). *Acta Cryst.* E**66**, o2559–o2560.
Gacche, R. N., Dhole, N. A., Kamble, S. G. & Bandgar, B. P. (2008). *J. Enzyme Inhib. Med. Chem.* **23**, 28–31.
Go, M.-L., Wu, X. & Liu, X.-L. (2005). *Curr. Med. Chem.* **12**, 483–499.
Isomoto, H., Furusu, H., Ohnita, K., Wen, C. Y., Inoue, K. & Kohno, S. (2005). *World J. Gastroenterol.* **11**, 1629–1633.
Jung, Y. J., Son, K. I., Oh, Y. E. & Noh, D. Y. (2008). *Polyhedron* **27**, 861–867.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suwunwong, T., Chantrapromma, S. & Fun, H.-K. (2011). *Chem. Pap.*, doi: 10.2478/s11696-011-0084-4.
Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol.* **25**, 503–508.

supplementary materials

Acta Cryst. (2011). E**67**, o2789-o2790 [**doi:10.1107/S1600536811039110**]

(2*E*)-1-(Pyridin-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

H.-K. Fun, S. Chantrapromma and T. Suwunwong

Comment

Chalcones and heteroaryl chalcones have drawn a lot of interests due to their wide range of biological properties including antioxidant (Gacche *et al.*, 2008), antibacterial (Go *et al.*, 2005; Isomoto *et al.*, 2005), anti-inflammatory and anticancer (Bandgar *et al.*, 2010) as well as HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003) activities. Furthermore they also exhibit fluorescent property (Jung *et al.*, 2008; Suwunwong *et al.*, 2011). In our on-going research on the biological and fluorescent properties of chalcones and heteroaryl chalcones (Chantrapromma *et al.*, 2009; Fun *et al.*, 2010, 2011; Suwunwong *et al.*, 2011), the title heteroaryl chalcone derivative (I) was synthesized in order to study the effects of substituted positions on the fluorescent property in comparision with the closely related compounds (Fun *et al.*, 2011; Suwunwong *et al.*, 2011). In addition (I) was also tested for analgesic and antibacterial activities. Our results showed that (I) exhibits a moderate analgesic activity but is inactive for antibacterial activity. Herein we report the crystal structure of (I).

The molecule of the title heteroaryl chalcone derivative (Fig. 1) exists in an *E* configuration with respect to the C7═C8 double bond [1.341 (3) Å]. The torsion angle C6–C7–C8–C9 is 179.0 (2)°. The molecule is almost planar with a dihedral angle between the pyridine and 2,4,6-trimethoxyphenyl rings of 5.51 (10)°. Atoms of the propenone bridge (C6, C7, C8 and O1) lie on the same plane [*r.m.s*. deviation of 0.017 (2)] and the torsion angle O1–C6–C7–C8 is -5.8 (4)°. The mean plane through this bridge makes dihedral angles of 6.96 (16) and 11.72 (16)° with the planes of pyridine and benzene rings, respectively. All the three substituted methoxy groups of the 2,4,6-trimethoxyphenyl unit are co-planar with the phenyl ring as indicated by the torsion angles C15–O2–C10–C11 = -0.4 (3)°, C16–O3–C12–C13 = 0.9 (3)° and C17–O4–C14–C13 = -4.7 (3)°. In the molecule, a weak intramolecular C7—H7A···O4 interaction (Table 1) generates an S(6) ring motif (Bernstein *et al.*, 1995). The bond distances are of normal values (Allen *et al.*, 1987) and comparable with related structures (Chantrapromma *et al.*, 2009; Fun *et al.*, 2010; 2011).

In the crystal packing (Fig. 2), only the two *ortho*-methoxy groups are involved in weak C—H···O interactions (Table 1). The adjacent molecules are linked by weak C17—H17B···O4 interaction (Table 1) into anti-parallel face-to-face pairs. The adjacent pairs were further connected by weak C3—H3A···O2 interactions (Table 1) into sheets parallel to the *ab* plane which are stacked down the *c* axis. The crystal may be further stabilized by C···O [3.203 (2) Å] short contacts.

Experimental

The title compound was synthesized by the condensation reaction of 2,4,6-trimethoxybenzaldehyde (0.40 g, 2 mmol) with 2-acetylpyridine (0.20 g, 2 mmol) in ethanol (30 ml) in the presence of 30% NaOH(aq) (5 ml). After stirring in ice bath at 278 K for 4 h, the resulting pale yellow solid appeared and was then collected by filtration, washed with distilled water, dried and purified by repeated recrystallization from acetone. Pale yellow plate-shaped single crystals of the title compound suitable for *X*-ray structure determination were recrystalized from acetone/ethanol (1:1 *v*/*v*) by the evaporation of the solvent at room temperature after several days, M.p. 392-393 K.

Refinement

All H atoms were located in difference maps and refined isotropically. A total of 1754 Friedel pairs were merged before final refinement as there is no large anomalous dispersion for the determination of the absolute structure.

Figures

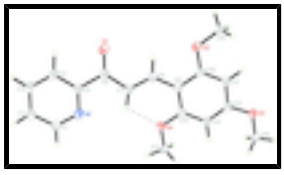


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids. A weak intramolecular C—H···O interaction is shown as a dashed line.

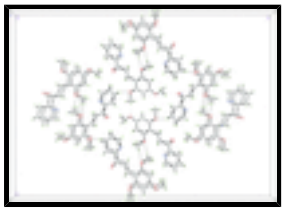


Fig. 2. The crystal packing of the title compound viewed along the *c* axis, showing molecular sheets parallel to the *ab* plane. Hydrogen bonds are shown as dashed lines.

(2*E*)-1-(Pyridin-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{17}H_{17}NO_4$	D_x = 1.433 Mg m^{-3}
M_r = 299.32	Melting point = 392–393 K
Orthorhombic, *Fdd*2	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: F 2 -2d	Cell parameters from 2309 reflections
a = 31.563 (2) Å	θ = 1.6–30.0°
b = 44.508 (3) Å	μ = 0.10 mm^{-1}
c = 3.9504 (3) Å	*T* = 100 K
V = 5549.6 (7) Å^3	Plate, pale yellow
Z = 16	0.58 × 0.14 × 0.04 mm
F(000) = 2528	

Data collection

Bruker APEXII CCD area detector diffractometer	2309 independent reflections
Radiation source: sealed tube	1908 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.100
φ and ω scans	θ_{max} = 30.0°, θ_{min} = 1.6°
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	*h* = −44→44
T_{min} = 0.943, T_{max} = 0.996	*k* = −62→62
31465 measured reflections	*l* = −5→5

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.105$	All H-atom parameters refined
$S = 1.09$	$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 4.8268P]$ where $P = (F_o^2 + 2F_c^2)/3$
2309 reflections	$(\Delta/\sigma)_{max} = 0.001$
267 parameters	$\Delta\rho_{max} = 0.23$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.18610 (5)	0.11217 (3)	1.2030 (5)	0.0298 (4)
O2	0.07115 (4)	0.04799 (3)	1.1758 (5)	0.0239 (3)
O3	0.05980 (4)	−0.04831 (3)	0.6752 (5)	0.0248 (4)
O4	0.19307 (4)	0.00496 (3)	0.6918 (5)	0.0242 (4)
N1	0.27419 (5)	0.08486 (4)	0.7393 (6)	0.0245 (4)
C1	0.31419 (7)	0.09271 (5)	0.6662 (7)	0.0263 (5)
C2	0.33299 (7)	0.11932 (5)	0.7701 (7)	0.0266 (5)
C3	0.30912 (7)	0.13912 (5)	0.9612 (7)	0.0270 (5)
C4	0.26779 (7)	0.13153 (5)	1.0406 (7)	0.0254 (5)
C5	0.25139 (6)	0.10419 (4)	0.9256 (7)	0.0225 (4)
C6	0.20685 (6)	0.09511 (4)	1.0251 (7)	0.0226 (4)
C7	0.19132 (7)	0.06578 (4)	0.9048 (7)	0.0228 (4)
C8	0.15391 (7)	0.05521 (4)	1.0129 (7)	0.0228 (4)
C9	0.13222 (6)	0.02737 (4)	0.9251 (6)	0.0213 (4)
C10	0.08883 (6)	0.02411 (4)	1.0100 (6)	0.0217 (4)
C11	0.06569 (6)	−0.00125 (4)	0.9267 (7)	0.0225 (4)

C12	0.08555 (6)	−0.02465 (4)	0.7554 (6)	0.0216 (4)
C13	0.12828 (6)	−0.02332 (4)	0.6760 (7)	0.0220 (4)
C14	0.15094 (6)	0.00253 (4)	0.7601 (6)	0.0217 (4)
C15	0.02726 (7)	0.04603 (5)	1.2600 (7)	0.0252 (5)
C16	0.07863 (7)	−0.07331 (5)	0.5027 (7)	0.0259 (5)
C17	0.21410 (7)	−0.02095 (5)	0.5537 (7)	0.0261 (5)
H1A	0.3305 (7)	0.0785 (5)	0.524 (8)	0.023 (6)*
H2A	0.3622 (7)	0.1239 (5)	0.708 (9)	0.027 (6)*
H3A	0.3216 (7)	0.1581 (5)	1.051 (8)	0.025 (6)*
H4A	0.2489 (7)	0.1445 (5)	1.187 (9)	0.030 (7)*
H8A	0.1375 (8)	0.0676 (6)	1.166 (9)	0.039 (8)*
H11A	0.0350 (7)	−0.0029 (5)	0.980 (8)	0.023 (6)*
H11B	0.2095 (7)	0.0554 (5)	0.747 (9)	0.025 (6)*
H13A	0.1415 (7)	−0.0402 (5)	0.545 (8)	0.025 (6)*
H15A	0.0101 (8)	0.0457 (6)	1.054 (9)	0.033 (8)*
H15B	0.0223 (8)	0.0281 (6)	1.428 (10)	0.038 (7)*
H15C	0.0194 (7)	0.0634 (5)	1.389 (8)	0.024 (7)*
H16A	0.0549 (7)	−0.0874 (5)	0.453 (8)	0.023 (6)*
H16B	0.0916 (7)	−0.0668 (5)	0.282 (9)	0.023 (6)*
H16C	0.1003 (8)	−0.0827 (5)	0.644 (9)	0.031 (7)*
H17A	0.2010 (8)	−0.0257 (6)	0.328 (9)	0.030 (8)*
H17B	0.2445 (7)	−0.0160 (5)	0.542 (8)	0.025 (6)*
H17C	0.2105 (7)	−0.0383 (5)	0.714 (9)	0.028 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0320 (8)	0.0216 (7)	0.0360 (11)	−0.0005 (6)	0.0043 (8)	−0.0058 (8)
O2	0.0246 (7)	0.0189 (6)	0.0283 (9)	0.0001 (5)	0.0026 (7)	−0.0024 (7)
O3	0.0251 (7)	0.0175 (6)	0.0317 (10)	−0.0026 (5)	0.0018 (7)	−0.0032 (7)
O4	0.0236 (7)	0.0194 (6)	0.0295 (10)	−0.0009 (5)	0.0026 (7)	−0.0034 (7)
N1	0.0284 (9)	0.0189 (8)	0.0264 (11)	0.0003 (6)	−0.0011 (8)	−0.0007 (8)
C1	0.0279 (10)	0.0233 (9)	0.0276 (13)	0.0008 (8)	0.0002 (10)	0.0019 (10)
C2	0.0290 (11)	0.0239 (9)	0.0270 (13)	−0.0012 (8)	−0.0014 (10)	0.0061 (10)
C3	0.0320 (11)	0.0203 (9)	0.0288 (14)	−0.0037 (8)	−0.0050 (11)	0.0023 (10)
C4	0.0313 (11)	0.0197 (9)	0.0251 (12)	−0.0004 (8)	−0.0021 (10)	0.0009 (9)
C5	0.0274 (10)	0.0188 (9)	0.0214 (11)	−0.0013 (7)	−0.0012 (9)	0.0033 (9)
C6	0.0267 (10)	0.0185 (9)	0.0224 (11)	0.0004 (7)	−0.0010 (9)	0.0029 (9)
C7	0.0278 (10)	0.0162 (9)	0.0242 (12)	0.0008 (7)	−0.0007 (9)	0.0012 (9)
C8	0.0266 (10)	0.0173 (8)	0.0245 (12)	0.0013 (7)	−0.0014 (9)	0.0017 (9)
C9	0.0258 (10)	0.0181 (9)	0.0200 (12)	0.0013 (7)	−0.0009 (9)	0.0019 (9)
C10	0.0287 (10)	0.0162 (8)	0.0201 (11)	0.0015 (7)	−0.0017 (9)	0.0011 (9)
C11	0.0233 (9)	0.0195 (9)	0.0248 (12)	0.0001 (7)	0.0002 (9)	0.0027 (9)
C12	0.0287 (10)	0.0156 (8)	0.0206 (12)	−0.0024 (7)	−0.0039 (9)	0.0021 (8)
C13	0.0263 (10)	0.0176 (8)	0.0220 (12)	−0.0009 (7)	−0.0012 (9)	−0.0009 (9)
C14	0.0245 (10)	0.0191 (9)	0.0214 (12)	0.0007 (7)	−0.0014 (9)	0.0028 (9)
C15	0.0247 (10)	0.0231 (9)	0.0279 (13)	0.0018 (8)	0.0003 (10)	−0.0022 (10)
C16	0.0305 (11)	0.0179 (9)	0.0292 (13)	−0.0016 (8)	−0.0011 (10)	−0.0026 (10)

C17	0.0264 (11)	0.0194 (9)	0.0325 (14)	0.0009 (8)	0.0045 (10)	−0.0029 (10)

Geometric parameters (Å, °)

O1—C6	1.224 (3)	C7—H11B	0.96 (3)
O2—C10	1.368 (2)	C8—C9	1.458 (3)
O2—C15	1.427 (3)	C8—H8A	0.97 (3)
O3—C12	1.367 (2)	C9—C14	1.413 (3)
O3—C16	1.434 (3)	C9—C10	1.417 (3)
O4—C14	1.361 (2)	C10—C11	1.384 (3)
O4—C17	1.438 (3)	C11—C12	1.392 (3)
N1—C5	1.341 (3)	C11—H11A	0.99 (2)
N1—C1	1.342 (3)	C12—C13	1.386 (3)
C1—C2	1.387 (3)	C13—C14	1.395 (3)
C1—H1A	0.99 (3)	C13—H13A	1.00 (3)
C2—C3	1.383 (3)	C15—H15A	0.98 (3)
C2—H2A	0.97 (2)	C15—H15B	1.05 (3)
C3—C4	1.384 (3)	C15—H15C	0.96 (3)
C3—H3A	1.00 (2)	C16—H16A	1.00 (2)
C4—C5	1.398 (3)	C16—H16B	1.01 (3)
C4—H4A	1.01 (3)	C16—H16C	0.98 (3)
C5—C6	1.515 (3)	C17—H17A	1.01 (3)
C6—C7	1.474 (3)	C17—H17B	0.99 (2)
C7—C8	1.341 (3)	C17—H17C	1.00 (3)
C10—O2—C15	117.39 (16)	O2—C10—C9	115.35 (17)
C12—O3—C16	117.48 (16)	C11—C10—C9	122.49 (19)
C14—O4—C17	117.55 (16)	C10—C11—C12	119.22 (19)
C5—N1—C1	117.13 (18)	C10—C11—H11A	121.7 (14)
N1—C1—C2	124.1 (2)	C12—C11—H11A	119.1 (14)
N1—C1—H1A	116.3 (13)	O3—C12—C13	123.98 (18)
C2—C1—H1A	119.5 (13)	O3—C12—C11	114.91 (18)
C3—C2—C1	118.2 (2)	C13—C12—C11	121.11 (18)
C3—C2—H2A	121.2 (15)	C12—C13—C14	118.69 (19)
C1—C2—H2A	120.6 (15)	C12—C13—H13A	119.3 (13)
C2—C3—C4	118.8 (2)	C14—C13—H13A	121.8 (13)
C2—C3—H3A	121.3 (14)	O4—C14—C13	121.27 (19)
C4—C3—H3A	119.8 (14)	O4—C14—C9	115.97 (17)
C3—C4—C5	119.2 (2)	C13—C14—C9	122.75 (18)
C3—C4—H4A	123.1 (13)	O2—C15—H15A	110.2 (17)
C5—C4—H4A	117.7 (13)	O2—C15—H15B	109.8 (14)
N1—C5—C4	122.54 (19)	H15A—C15—H15B	115 (2)
N1—C5—C6	117.99 (17)	O2—C15—H15C	109.0 (14)
C4—C5—C6	119.4 (2)	H15A—C15—H15C	108 (2)
O1—C6—C7	123.82 (19)	H15B—C15—H15C	104 (2)
O1—C6—C5	118.70 (18)	O3—C16—H16A	105.8 (14)
C7—C6—C5	117.47 (19)	O3—C16—H16B	111.0 (14)
C8—C7—C6	120.1 (2)	H16A—C16—H16B	108 (2)
C8—C7—H11B	124.1 (14)	O3—C16—H16C	110.5 (17)
C6—C7—H11B	115.8 (14)	H16A—C16—H16C	112 (2)

C7—C8—C9	129.5 (2)	H16B—C16—H16C	109 (2)
C7—C8—H8A	118.2 (15)	O4—C17—H17A	108.4 (15)
C9—C8—H8A	112.3 (15)	O4—C17—H17B	106.7 (14)
C14—C9—C10	115.68 (17)	H17A—C17—H17B	114 (2)
C14—C9—C8	125.35 (18)	O4—C17—H17C	109.0 (16)
C10—C9—C8	118.97 (18)	H17A—C17—H17C	111 (2)
O2—C10—C11	122.16 (18)	H17B—C17—H17C	108 (2)
C5—N1—C1—C2	−0.2 (4)	C8—C9—C10—O2	−1.0 (3)
N1—C1—C2—C3	0.0 (4)	C14—C9—C10—C11	−2.2 (3)
C1—C2—C3—C4	0.1 (4)	C8—C9—C10—C11	178.1 (2)
C2—C3—C4—C5	−0.1 (4)	O2—C10—C11—C12	179.5 (2)
C1—N1—C5—C4	0.3 (3)	C9—C10—C11—C12	0.5 (4)
C1—N1—C5—C6	−177.1 (2)	C16—O3—C12—C13	0.9 (3)
C3—C4—C5—N1	−0.1 (4)	C16—O3—C12—C11	−179.1 (2)
C3—C4—C5—C6	177.2 (2)	C10—C11—C12—O3	−178.1 (2)
N1—C5—C6—O1	178.0 (2)	C10—C11—C12—C13	1.9 (4)
C4—C5—C6—O1	0.5 (3)	O3—C12—C13—C14	177.6 (2)
N1—C5—C6—C7	−1.1 (3)	C11—C12—C13—C14	−2.3 (3)
C4—C5—C6—C7	−178.5 (2)	C17—O4—C14—C13	−4.7 (3)
O1—C6—C7—C8	−5.8 (4)	C17—O4—C14—C9	174.2 (2)
C5—C6—C7—C8	173.2 (2)	C12—C13—C14—O4	179.2 (2)
C6—C7—C8—C9	179.0 (2)	C12—C13—C14—C9	0.4 (3)
C7—C8—C9—C14	14.7 (4)	C10—C9—C14—O4	−177.1 (2)
C7—C8—C9—C10	−165.6 (2)	C8—C9—C14—O4	2.6 (3)
C15—O2—C10—C11	−0.4 (3)	C10—C9—C14—C13	1.8 (3)
C15—O2—C10—C9	178.7 (2)	C8—C9—C14—C13	−178.6 (2)
C14—C9—C10—O2	178.7 (2)		

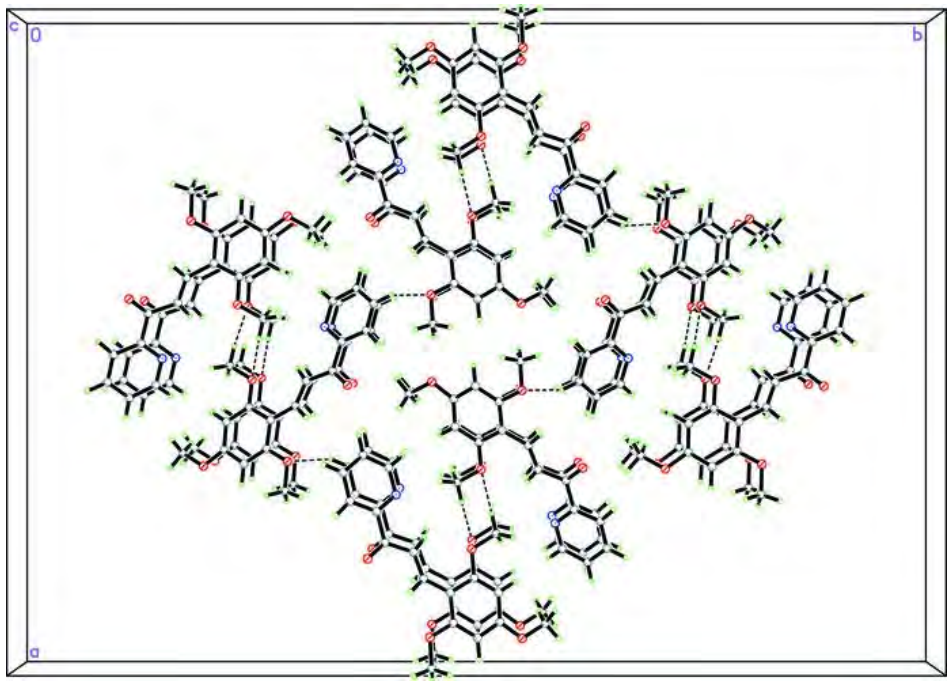
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3A···O2^{i}	1.00 (2)	2.45 (3)	3.369 (2)	157.8 (17)
C7—H11B···O4	0.97 (3)	2.31 (2)	2.835 (2)	113.1 (18)
C17—H17B···O4^{ii}	0.99 (2)	2.46 (2)	3.337 (3)	148 (2)

Symmetry codes: (i) *x*+9/4, −*y*+9/4, *z*+1/4; (ii) −*x*+1/2, −*y*, *z*−1/2.

Fig. 1

Fig. 2

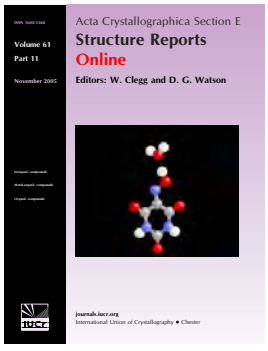


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, H. Stoeckli-Evans, E. R. T. Tiekink and M. Weil**

(*E*)-1-(Thiophen-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun, Thitipone Suwunwong, Teerasak Anantapong, Chatchanok Karalai and Suchada Chantrapromma

Acta Cryst. (2011). E**67**, o3074–o3075



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(Thiophen-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun,[a]*‡ Thitipone Suwunwong,[b] Teerasak Anantapong,[c] Chatchanok Karalai[b] and Suchada Chantrapromma[b]§

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [c]Department of Biotechnology, Faculty of Agro-Industry, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

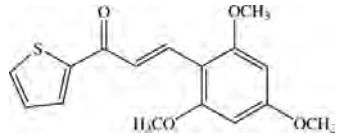
Received 6 October 2011; accepted 17 October 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.066; wR factor = 0.178; data-to-parameter ratio = 20.1.

There are two crystallograpically independent molecules in the asymmetric unit of the title heteroaryl chalcone derivative, $C_{16}H_{16}O_4S$, with slightly different conformations. The thienyl ring of one molecule is disordered over two positions, with a refined site-occupancy ratio of 0.713 (5):0.287 (5). The molecules are twisted: the dihedral angle between the thienyl and benzene rings is 9.72 (19)° in the ordered molecule, and 3.8 (4) and 2.1 (8)° for the major and minor components, respectively, in the disordered molecule. In both molecules, all three substituted methoxy groups are coplanar with the benzene ring to which they are attached. In each molecule, a weak intramolecular C—H···O interaction generates an S(6) ring motif. In the crystal structure, adjacent molecules are linked into a three-dimensional network by weak C—H···O interactions.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related literature on hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Chantrapromma *et al.* (2009); Fun *et al.* (2010, 2011); Suwunwong *et al.* (2009). For background to and applications of chalcones, see: Go *et al.* (2005); Liu *et al.* (2008); Ng *et al.* (2009); Ni *et al.* (2004); Suwunwong *et al.* (2011); Tewtrakul *et al.* (2003). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{16}H_{16}O_4S$
M_r = 304.36
Orthorhombic, $Pna2_1$
a = 22.8482 (10) Å
b = 31.2117 (13) Å
c = 3.9876 (2) Å
V = 2843.7 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 100 K
0.60 × 0.06 × 0.05 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.869, T_{max} = 0.988
20029 measured reflections
8085 independent reflections
5348 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.178
S = 1.01
8085 reflections
402 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.08 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Absolute structure: Flack (1983), with 3389 Friedel pairs
Flack parameter: 0.09 (11)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2B—H2B···O3A	0.93	2.56	3.482 (10)	171
C6A—H6A···O4A	0.93	2.22	2.815 (4)	121
C6B—H6B···O4B	0.93	2.24	2.824 (4)	120
C15A—H15C···O1B^{i}	0.96	2.51	3.451 (5)	166
C15B—H15F···O1A^{ii}	0.96	2.55	3.355 (5)	141
C16B—H16E···O3B^{iii}	0.96	2.59	3.401 (4)	142

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z-1$; (ii) $-x+1, -y+1, z-\frac{1}{2}$; (iii) $-x+1, -y+1, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for a research grant (No. RSA 5280033) and Prince of Songkla University for financial support. The authors also thank Universiti Sains Malaysia for a Research University grant (No. 1001/PFIZIK/811160).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2650).

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, email: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chantrapromma, S., Suwunwong, T., Karalai, C. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o893–o894.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Fun, H.-K., Chantrapromma, S. & Suwunwong, T. (2011). *Acta Cryst.* E**67**, o2789–o2790.

Fun, H.-K., Suwunwong, T., Chantrapromma, S. & Karalai, C. (2010). *Acta Cryst.* E**66**, o2559–o2560.

Go, M.-L., Wu, X. & Liu, X.-L. (2005). *Curr. Med. Chem.* **12**, 483–499.

Liu, X. L., Xu, Y. J. & Go, M. L. (2008). *Eur. J. Med. Chem.* **43**, 1681–1687.

Ng, L.-T., Ko, H.-H. & Lu, T.-M. (2009). *Bioorg. Med. Chem.* **17**, 4360–4366.

Ni, L., Meng, C. Q. & Sikorski, J. A. (2004). *Expert Opin. Ther. Pat.* **14**, 1669–1691.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Suwunwong, T., Chantrapromma, S. & Fun, H.-K. (2011). *Chem. Pap.* **65**, 890–897.

Suwunwong, T., Chantrapromma, S., Pakdeevanich, P. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o1575–o1576.

Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol.* **25**, 503–508.

supplementary materials

Acta Cryst. (2011). E**67**, o3074-o3075 [**doi:10.1107/S1600536811042930**]

(*E*)-1-(Thiophen-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

H.-K. Fun, T. Suwunwong, T. Anantapong, C. Karalai and S. Chantrapromma

Comment

Chalcones have been reported to be responsible for a variety of biological activities such as analgesic, anti-inflammatory, antibacterial and antimycotic (Go *et al.*, 2005; Liu *et al.*, 2008; Ni *et al.*, 2004) as well as HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003) and tyrosinase inhibitory (Ng *et al.*, 2009) properties. Our research on the fluorescent and biological studies of chalcones and heteroaryl chalcone derivatives (Chantrapromma *et al.*, 2009; Suwunwong *et al.*, 2009, 2011) led us to synthesize the title heteroaryl chalcone (I). (I) exhibits fluorescent property (Suwunwong *et al.*, 2011) and possess moderate analgesic property. It was also tested for antibacterial activities but found to be inactive. Herein we report the crystal structure of (I).

There are two crystallographic independent molecules *A* and *B* in the asymmetric unit of (I) with different conformations of the methoxy group at *para* position or at atom C11 and also in bond angles (Fig. 1). The thienyl ring of molecule *B* is disordered over two orientations with the refined site-occupancy ratio of 0.713 (5):0.287 (5). The thienyl rings in the major and minor components are related by 180° rotation. The molecule of (I) is slightly twisted. The dihedral angle between the thienyl and benzene rings is 9.72 (19)° in molecule *A* whereas these values are 3.8 (4) and 2.1 (8)° for the major and minor components in the disordered molecule *B*. The central prop-2-en-1-one bridge (C5–C7/O1) in both molecules is slightly twisted as indicated by the torsion angle O1—C5—C6—C7 = 5.8 (6) and 6.6 (6)° in molecules *A* and *B*, respectively. The mean plane through this bridge makes dihedral angles of 8.9 (3) and 2.3 (2)° with the thienyl and benzene rings, respectively, in molecule *A* whereas the corresponding values are 4.2 (4) and 8.0 (3)° in molecule *B* for the major component, and 8.2 (8) and 8.0 (3)° for the minor component. In both molecules, all the three substituted methoxy groups are co-planar with the attached benzene with torsion angles C14—O2—C9—C10 = -5.3 (5)°; C15—O3—C11—C12 = 3.7 (5)° and C16—O4—C13—C12 = 0.3 (5)° in molecule *A*. The corresponding values are 0.6 (5), 178.0 (3) and 0.6 (5)° in molecule *B*. This also indicates that the methoxy group at the *para* position (or at atom C11) has different conformations as it points toward the methoxy group at the *ortho* position at atom C13 (in molecule *A*) whereas it points toward the *ortho* methoxy at atom C9 (in molecule *B*). In each molecule, intramolecular C—H···O weak interaction (Table 1) generates S(6) ring motif (Bernstein *et al.*, 1995). The bond distances agree with the literature values (Allen *et al.*, 1987) and are comparable with those observed in related structures (Chantrapromma *et al.*, 2009; Fun *et al.*, 2010, 2011; Suwunwong *et al.*, 2009).

In the crystal packing (Fig. 2), adjacent molecules are linked into a three-dimensional network by weak C—H···O interactions (Table 1).

Experimental

The title compound was synthesized by the condensation of 2,4,6-trimethoxybenzaldehyde (0.40 g, 2 mmol) with 2-acethylthiophene (0.35 ml, 2 mmol) in ethanol (30 ml) in the presence of 30% NaOH (aq) (5 ml). After stirring for 3 h in ice bath at 278 K, the resulting pale-yellow solid was collected by filtration, washed with distilled water, dried in air and purified by recrystallization from acetone. Pale-yellow needle-shaped single crystals of the title compound suitable for X-ray structure

determination were recrystallized from acetone–ethanol (1:1 *v*/*v*) by slow evaporation of the solvent at room temperature after several days; m.p. 381–382 K.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93 Å, U_{iso} = 1.2U_{eq}(C) for aromatic and methyne C atoms and C—H = 0.96 Å, U_{iso} = 1.5U_{eq}(C) for methyl groups. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 1.65 Å from C3 and the deepest hole is located at 0.29 Å from S1A. The thienyl ring of molecule B is disordered over two sites with refined site occupancies of 0.713 (5) and 0.287 (5). Initially SAME, DELU and SIMU restraints were used. In the final refinement, these restraints were removed. A total of 3389 Friedel pairs were used to determine the absolute structure.

Figures

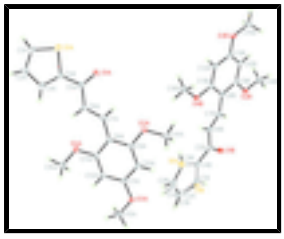


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids. Open bonds show the minor component of the disordered thienyl ring.

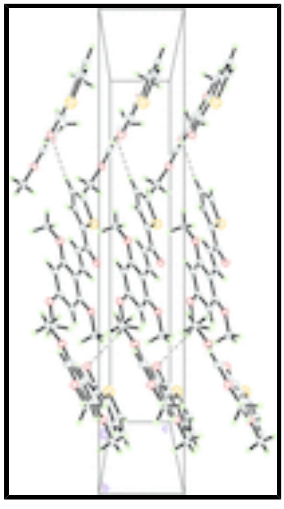


Fig. 2. The crystal packing of the title compound viewed along the *b* axis. Only the major component of disorder is shown. Weak C—H···O interactions are shown as dashed lines.

(*E*)-1-(Thiophen-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{16}H_{16}O_4S$	D_x = 1.422 Mg m^{-3}
M_r = 304.36	Melting point = 381–382 K
Orthorhombic, *Pna*2$_1$	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 8085 reflections
a = 22.8482 (10) Å	θ = 1.3–30.0°
b = 31.2117 (13) Å	μ = 0.24 mm^{-1}

c = 3.9876 (2) Å	T = 100 K
V = 2843.7 (2) Å^3	Needle, pale yellow
Z = 8	0.60 × 0.06 × 0.05 mm
F(000) = 1280	

Data collection

Bruker APEXII CCD area-detector diffractometer	8085 independent reflections
Radiation source: sealed tube	5348 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.065
φ and ω scans	θ_{max} = 30.0°, θ_{min} = 1.3°
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	h = −27→32
T_{min} = 0.869, T_{max} = 0.988	k = −43→40
20029 measured reflections	l = −5→5

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.066	H-atom parameters constrained
$wR(F^2)$ = 0.178	$w = 1/[\sigma^2(F_o^2) + (0.0951P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
8085 reflections	$\Delta\rho_{max}$ = 1.08 e Å^{-3}
402 parameters	$\Delta\rho_{min}$ = −0.56 e Å^{-3}
1 restraint	Absolute structure: Flack (1983), with 3389 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.09 (11)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1A	0.13970 (4)	0.41398 (3)	1.0329 (3)	0.0270 (2)	
O1A	0.21057 (10)	0.48128 (7)	0.7262 (8)	0.0228 (6)	
O2A	0.27887 (11)	0.61480 (7)	0.4202 (7)	0.0213 (6)	
O3A	0.19621 (10)	0.75129 (7)	0.6341 (7)	0.0206 (6)	
O4A	0.09780 (10)	0.62191 (7)	0.9862 (7)	0.0211 (6)	
C1A	0.07345 (15)	0.40255 (11)	1.2033 (10)	0.0217 (8)	
H1A	0.0622	0.3752	1.2684	0.026*	
C2A	0.03841 (16)	0.43786 (10)	1.2349 (11)	0.0241 (8)	
H2A	0.0005	0.4367	1.3198	0.029*	
C3A	0.06544 (15)	0.47698 (12)	1.1249 (10)	0.0236 (8)	
H3A	0.0483	0.5040	1.1312	0.028*	
C4A	0.12232 (15)	0.46779 (10)	1.0057 (10)	0.0192 (7)	
C5A	0.16654 (15)	0.49669 (10)	0.8497 (10)	0.0198 (7)	
C6A	0.15415 (15)	0.54312 (10)	0.8687 (10)	0.0209 (8)	
H6A	0.1214	0.5529	0.9841	0.025*	
C7A	0.19013 (15)	0.57103 (10)	0.7198 (10)	0.0187 (7)	
H7A	0.2213	0.5587	0.6051	0.022*	
C8A	0.18848 (15)	0.61784 (10)	0.7077 (10)	0.0166 (7)	
C9A	0.23464 (15)	0.64009 (10)	0.5441 (10)	0.0175 (7)	
C10A	0.23602 (15)	0.68428 (10)	0.5233 (10)	0.0177 (7)	
H10A	0.2669	0.6980	0.4162	0.021*	
C11A	0.19052 (15)	0.70813 (10)	0.6647 (10)	0.0170 (7)	
C12A	0.14394 (15)	0.68798 (10)	0.8199 (10)	0.0190 (7)	
H12A	0.1135	0.7040	0.9108	0.023*	
C13A	0.14324 (14)	0.64351 (10)	0.8385 (10)	0.0185 (7)	
C14A	0.32476 (14)	0.63633 (10)	0.2398 (10)	0.0190 (7)	
H14A	0.3511	0.6155	0.1470	0.029*	
H14B	0.3458	0.6548	0.3900	0.029*	
H14C	0.3080	0.6531	0.0619	0.029*	
C15A	0.15164 (15)	0.77720 (10)	0.7938 (11)	0.0208 (8)	
H15A	0.1602	0.8070	0.7584	0.031*	
H15B	0.1511	0.7713	1.0300	0.031*	
H15C	0.1141	0.7705	0.6989	0.031*	
C16A	0.05059 (16)	0.64661 (11)	1.1205 (11)	0.0232 (8)	
H16A	0.0221	0.6278	1.2182	0.035*	
H16B	0.0327	0.6629	0.9441	0.035*	
H16C	0.0653	0.6657	1.2892	0.035*	
S1B	0.42377 (10)	0.79935 (8)	1.2577 (7)	0.0198 (5)	0.713 (5)
O1B	0.52996 (11)	0.74766 (7)	1.3193 (8)	0.0283 (7)	
O2B	0.65708 (9)	0.63419 (7)	1.2581 (7)	0.0199 (6)	
O3B	0.62447 (10)	0.49871 (7)	0.7051 (7)	0.0192 (5)	
O4B	0.47249 (10)	0.60034 (7)	0.8092 (7)	0.0194 (5)	
C1B	0.3535 (4)	0.8012 (3)	1.105 (2)	0.0191 (18)	0.713 (5)
H1B	0.3290	0.8248	1.1252	0.023*	0.713 (5)
C2B	0.3376 (4)	0.7629 (3)	0.947 (3)	0.027 (2)	0.713 (5)

H2B	0.3018	0.7578	0.8433	0.032*	0.713 (5)
C3B	0.3838 (5)	0.7331 (4)	0.968 (3)	0.032 (3)	0.713 (5)
H3B	0.3804	0.7050	0.8923	0.038*	0.713 (5)
S1X	0.3779 (3)	0.7288 (2)	0.9029 (17)	0.0168 (13)*	0.287 (5)
C1X	0.3356 (11)	0.7722 (7)	0.978 (7)	0.013 (5)*	0.287 (5)
H1BX	0.2977	0.7753	0.8964	0.015*	0.287 (5)
C2X	0.3631 (11)	0.8023 (9)	1.170 (6)	0.018 (6)*	0.287 (5)
H2BX	0.3451	0.8268	1.2552	0.022*	0.287 (5)
C3X	0.4244 (12)	0.7909 (7)	1.225 (7)	0.014 (6)*	0.287 (5)
H3BX	0.4524	0.8086	1.3225	0.017*	0.287 (5)
C4B	0.43447 (15)	0.74896 (10)	1.1089 (10)	0.0189 (7)	
C5B	0.49125 (15)	0.72746 (10)	1.1768 (10)	0.0195 (8)	
C6B	0.49764 (15)	0.68308 (10)	1.0567 (11)	0.0213 (8)	
H6B	0.4682	0.6704	0.9301	0.026*	
C7B	0.54678 (15)	0.66094 (10)	1.1323 (10)	0.0200 (8)	
H7B	0.5731	0.6754	1.2698	0.024*	
C8B	0.56499 (15)	0.61804 (9)	1.0324 (10)	0.0175 (7)	
C9B	0.62300 (16)	0.60488 (10)	1.0981 (10)	0.0188 (7)	
C10B	0.64515 (15)	0.56527 (10)	0.9991 (11)	0.0192 (7)	
H10B	0.6836	0.5575	1.0465	0.023*	
C11B	0.60766 (15)	0.53768 (10)	0.8260 (10)	0.0182 (7)	
C12B	0.54957 (14)	0.54812 (10)	0.7577 (10)	0.0176 (7)	
H12B	0.5253	0.5291	0.6446	0.021*	
C13B	0.52869 (15)	0.58828 (11)	0.8645 (10)	0.0184 (7)	
C14B	0.71693 (15)	0.62351 (11)	1.3201 (11)	0.0239 (8)	
H14D	0.7362	0.6474	1.4246	0.036*	
H14E	0.7360	0.6169	1.1117	0.036*	
H14F	0.7188	0.5991	1.4657	0.036*	
C15B	0.68437 (15)	0.48626 (11)	0.7563 (12)	0.0270 (9)	
H15D	0.6916	0.4596	0.6443	0.040*	
H15E	0.6917	0.4830	0.9920	0.040*	
H15F	0.7098	0.5079	0.6669	0.040*	
C16B	0.43513 (15)	0.57035 (11)	0.6405 (10)	0.0209 (8)	
H16D	0.3961	0.5817	0.6297	0.031*	
H16E	0.4348	0.5438	0.7615	0.031*	
H16F	0.4495	0.5655	0.4174	0.031*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0279 (5)	0.0170 (4)	0.0359 (6)	−0.0005 (4)	0.0025 (5)	0.0010 (4)
O1A	0.0172 (11)	0.0159 (11)	0.0354 (17)	0.0015 (9)	0.0028 (12)	−0.0011 (11)
O2A	0.0188 (12)	0.0160 (11)	0.0290 (15)	0.0019 (9)	0.0044 (11)	0.0041 (10)
O3A	0.0194 (12)	0.0099 (10)	0.0326 (16)	0.0006 (9)	0.0018 (11)	0.0029 (10)
O4A	0.0174 (12)	0.0135 (11)	0.0324 (16)	−0.0022 (9)	0.0064 (12)	−0.0005 (11)
C1A	0.0233 (17)	0.0186 (16)	0.023 (2)	−0.0046 (14)	0.0012 (16)	0.0030 (14)
C2A	0.0229 (17)	0.0171 (16)	0.032 (2)	−0.0014 (13)	0.0067 (18)	0.0011 (16)
C3A	0.0213 (18)	0.0237 (17)	0.026 (2)	−0.0096 (14)	0.0046 (16)	0.0041 (15)

C4A	0.0231 (17)	0.0125 (14)	0.0219 (19)	−0.0005 (12)	−0.0008 (16)	−0.0015 (14)
C5A	0.0195 (17)	0.0135 (15)	0.026 (2)	0.0001 (12)	−0.0029 (16)	−0.0021 (14)
C6A	0.0208 (17)	0.0131 (15)	0.029 (2)	0.0009 (12)	0.0005 (16)	−0.0023 (14)
C7A	0.0206 (16)	0.0141 (14)	0.0214 (19)	0.0022 (12)	0.0010 (15)	0.0019 (15)
C8A	0.0159 (15)	0.0132 (14)	0.0207 (19)	−0.0015 (12)	−0.0011 (14)	−0.0012 (13)
C9A	0.0201 (16)	0.0141 (14)	0.0181 (18)	0.0018 (12)	−0.0025 (15)	0.0027 (14)
C10A	0.0186 (16)	0.0145 (14)	0.0199 (18)	−0.0024 (12)	−0.0009 (15)	0.0019 (14)
C11A	0.0148 (15)	0.0098 (14)	0.026 (2)	0.0010 (12)	−0.0032 (14)	0.0013 (13)
C12A	0.0163 (15)	0.0144 (15)	0.026 (2)	0.0043 (12)	−0.0027 (15)	0.0001 (14)
C13A	0.0123 (15)	0.0177 (15)	0.026 (2)	−0.0050 (12)	−0.0013 (14)	0.0003 (14)
C14A	0.0170 (16)	0.0170 (15)	0.023 (2)	0.0005 (12)	0.0005 (16)	0.0023 (15)
C15A	0.0229 (17)	0.0099 (14)	0.030 (2)	0.0017 (12)	−0.0045 (16)	0.0019 (15)
C16A	0.0217 (18)	0.0180 (16)	0.030 (2)	−0.0017 (13)	0.0027 (16)	−0.0041 (15)
S1B	0.0189 (8)	0.0084 (8)	0.0322 (10)	0.0019 (7)	0.0018 (7)	−0.0026 (8)
O1B	0.0207 (12)	0.0134 (11)	0.051 (2)	0.0005 (9)	−0.0075 (14)	−0.0025 (12)
O2B	0.0140 (11)	0.0120 (10)	0.0339 (16)	−0.0022 (8)	−0.0061 (12)	−0.0020 (11)
O3B	0.0193 (12)	0.0106 (10)	0.0276 (15)	0.0017 (8)	−0.0013 (11)	0.0009 (10)
O4B	0.0166 (11)	0.0112 (10)	0.0304 (16)	−0.0005 (8)	−0.0018 (11)	−0.0001 (10)
C1B	0.016 (4)	0.020 (3)	0.022 (5)	0.005 (2)	−0.004 (3)	0.005 (3)
C2B	0.027 (4)	0.020 (4)	0.033 (5)	−0.004 (3)	0.003 (3)	−0.008 (4)
C3B	0.044 (5)	0.021 (4)	0.031 (6)	0.004 (3)	−0.007 (4)	0.001 (4)
C4B	0.0195 (17)	0.0149 (15)	0.022 (2)	−0.0038 (13)	0.0001 (15)	0.0014 (14)
C5B	0.0176 (16)	0.0120 (15)	0.029 (2)	−0.0003 (13)	0.0051 (15)	0.0019 (13)
C6B	0.0210 (17)	0.0118 (14)	0.031 (2)	−0.0005 (12)	−0.0013 (17)	−0.0014 (16)
C7B	0.0179 (16)	0.0152 (15)	0.027 (2)	−0.0043 (13)	−0.0013 (15)	−0.0005 (14)
C8B	0.0196 (16)	0.0113 (13)	0.0215 (18)	−0.0004 (12)	0.0031 (15)	−0.0009 (14)
C9B	0.0216 (17)	0.0143 (15)	0.020 (2)	−0.0038 (13)	0.0010 (15)	0.0039 (13)
C10B	0.0184 (16)	0.0136 (15)	0.026 (2)	−0.0001 (12)	0.0005 (16)	0.0022 (15)
C11B	0.0218 (17)	0.0103 (14)	0.022 (2)	−0.0003 (12)	0.0023 (15)	0.0003 (13)
C12B	0.0196 (15)	0.0103 (13)	0.0229 (19)	−0.0015 (12)	−0.0013 (16)	0.0023 (14)
C13B	0.0198 (17)	0.0169 (15)	0.0184 (18)	−0.0013 (13)	0.0021 (15)	−0.0001 (14)
C14B	0.0178 (16)	0.0197 (16)	0.034 (2)	−0.0041 (13)	−0.0060 (17)	0.0000 (16)
C15B	0.0227 (18)	0.0125 (15)	0.046 (3)	0.0051 (13)	−0.0004 (19)	0.0013 (18)
C16B	0.0190 (17)	0.0185 (16)	0.025 (2)	−0.0040 (13)	−0.0026 (15)	0.0000 (14)

Geometric parameters (Å, °)

S1A—C1A	1.697 (4)	O2B—C14B	1.429 (4)
S1A—C4A	1.729 (3)	O3B—C11B	1.364 (4)
O1A—C5A	1.219 (4)	O3B—C15B	1.437 (4)
O2A—C9A	1.374 (4)	O4B—C13B	1.356 (4)
O2A—C14A	1.438 (4)	O4B—C16B	1.434 (4)
O3A—C11A	1.359 (4)	C1B—C2B	1.399 (13)
O3A—C15A	1.448 (4)	C1B—H1B	0.9300
O4A—C13A	1.371 (4)	C2B—C3B	1.408 (15)
O4A—C16A	1.430 (4)	C2B—H2B	0.9300
C1A—C2A	1.368 (5)	C3B—C4B	1.380 (12)
C1A—H1A	0.9300	C3B—H3B	0.9300
C2A—C3A	1.437 (5)	S1X—C4B	1.657 (7)

C2A—H2A	0.9300	S1X—C1X	1.69 (2)
C3A—C4A	1.413 (5)	C1X—C2X	1.37 (3)
C3A—H3A	0.9300	C1X—H1BX	0.9300
C4A—C5A	1.491 (5)	C2X—C3X	1.46 (3)
C5A—C6A	1.478 (4)	C2X—H2BX	0.9300
C6A—C7A	1.337 (5)	C3X—C4B	1.41 (2)
C6A—H6A	0.9300	C3X—H3BX	0.9300
C7A—C8A	1.462 (4)	C4B—C5B	1.485 (5)
C7A—H7A	0.9300	C5B—C6B	1.473 (5)
C8A—C13A	1.408 (5)	C6B—C7B	1.353 (5)
C8A—C9A	1.421 (5)	C6B—H6B	0.9300
C9A—C10A	1.382 (4)	C7B—C8B	1.458 (4)
C10A—C11A	1.397 (5)	C7B—H7B	0.9300
C10A—H10A	0.9300	C8B—C9B	1.412 (5)
C11A—C12A	1.383 (5)	C8B—C13B	1.414 (5)
C12A—C13A	1.390 (4)	C9B—C10B	1.393 (5)
C12A—H12A	0.9300	C10B—C11B	1.397 (5)
C14A—H14A	0.9600	C10B—H10B	0.9300
C14A—H14B	0.9600	C11B—C12B	1.394 (5)
C14A—H14C	0.9600	C12B—C13B	1.407 (5)
C15A—H15A	0.9600	C12B—H12B	0.9300
C15A—H15B	0.9600	C14B—H14D	0.9600
C15A—H15C	0.9600	C14B—H14E	0.9600
C16A—H16A	0.9600	C14B—H14F	0.9600
C16A—H16B	0.9600	C15B—H15D	0.9600
C16A—H16C	0.9600	C15B—H15E	0.9600
S1B—C4B	1.699 (4)	C15B—H15F	0.9600
S1B—C1B	1.717 (10)	C16B—H16D	0.9600
O1B—C5B	1.226 (4)	C16B—H16E	0.9600
O2B—C9B	1.360 (4)	C16B—H16F	0.9600
C1A—S1A—C4A	91.40 (17)	C1B—C2B—H2B	125.0
C9A—O2A—C14A	116.6 (3)	C3B—C2B—H2B	125.0
C11A—O3A—C15A	116.5 (3)	C4B—C3B—C2B	114.6 (9)
C13A—O4A—C16A	117.8 (3)	C4B—C3B—H3B	122.7
C2A—C1A—S1A	112.9 (3)	C2B—C3B—H3B	122.7
C2A—C1A—H1A	123.5	C4B—S1X—C1X	93.0 (9)
S1A—C1A—H1A	123.5	C2X—C1X—S1X	113 (2)
C1A—C2A—C3A	113.9 (3)	C2X—C1X—H1BX	123.6
C1A—C2A—H2A	123.1	S1X—C1X—H1BX	123.6
C3A—C2A—H2A	123.1	C1X—C2X—C3X	111 (2)
C4A—C3A—C2A	109.0 (3)	C1X—C2X—H2BX	124.6
C4A—C3A—H3A	125.5	C3X—C2X—H2BX	124.6
C2A—C3A—H3A	125.5	C4B—C3X—C2X	110 (2)
C3A—C4A—C5A	129.9 (3)	C4B—C3X—H3BX	125.2
C3A—C4A—S1A	112.8 (3)	C2X—C3X—H3BX	125.2
C5A—C4A—S1A	117.3 (3)	C3B—C4B—C3X	109.3 (12)
O1A—C5A—C6A	124.4 (3)	C3B—C4B—C5B	130.2 (6)
O1A—C5A—C4A	119.3 (3)	C3X—C4B—C5B	120.2 (12)
C6A—C5A—C4A	116.2 (3)	C3X—C4B—S1X	112.9 (12)

C7A—C6A—C5A	119.9 (3)	C5B—C4B—S1X	126.9 (4)
C7A—C6A—H6A	120.1	C3B—C4B—S1B	110.7 (5)
C5A—C6A—H6A	120.1	C5B—C4B—S1B	118.7 (3)
C6A—C7A—C8A	130.5 (3)	S1X—C4B—S1B	114.4 (3)
C6A—C7A—H7A	114.7	O1B—C5B—C6B	124.3 (3)
C8A—C7A—H7A	114.7	O1B—C5B—C4B	118.8 (3)
C13A—C8A—C9A	115.9 (3)	C6B—C5B—C4B	116.9 (3)
C13A—C8A—C7A	125.1 (3)	C7B—C6B—C5B	119.4 (3)
C9A—C8A—C7A	119.0 (3)	C7B—C6B—H6B	120.3
O2A—C9A—C10A	122.3 (3)	C5B—C6B—H6B	120.3
O2A—C9A—C8A	115.5 (3)	C6B—C7B—C8B	130.2 (3)
C10A—C9A—C8A	122.1 (3)	C6B—C7B—H7B	114.9
C9A—C10A—C11A	119.4 (3)	C8B—C7B—H7B	114.9
C9A—C10A—H10A	120.3	C9B—C8B—C13B	116.6 (3)
C11A—C10A—H10A	120.3	C9B—C8B—C7B	119.0 (3)
O3A—C11A—C12A	124.4 (3)	C13B—C8B—C7B	124.4 (3)
O3A—C11A—C10A	114.9 (3)	O2B—C9B—C10B	121.5 (3)
C12A—C11A—C10A	120.7 (3)	O2B—C9B—C8B	115.4 (3)
C11A—C12A—C13A	119.1 (3)	C10B—C9B—C8B	123.1 (3)
C11A—C12A—H12A	120.4	C9B—C10B—C11B	117.7 (3)
C13A—C12A—H12A	120.4	C9B—C10B—H10B	121.2
O4A—C13A—C12A	121.5 (3)	C11B—C10B—H10B	121.2
O4A—C13A—C8A	115.8 (3)	O3B—C11B—C12B	114.1 (3)
C12A—C13A—C8A	122.7 (3)	O3B—C11B—C10B	123.5 (3)
O2A—C14A—H14A	109.5	C12B—C11B—C10B	122.4 (3)
O2A—C14A—H14B	109.5	C11B—C12B—C13B	118.2 (3)
H14A—C14A—H14B	109.5	C11B—C12B—H12B	120.9
O2A—C14A—H14C	109.5	C13B—C12B—H12B	120.9
H14A—C14A—H14C	109.5	O4B—C13B—C12B	121.3 (3)
H14B—C14A—H14C	109.5	O4B—C13B—C8B	116.7 (3)
O3A—C15A—H15A	109.5	C12B—C13B—C8B	122.0 (3)
O3A—C15A—H15B	109.5	O2B—C14B—H14D	109.5
H15A—C15A—H15B	109.5	O2B—C14B—H14E	109.5
O3A—C15A—H15C	109.5	H14D—C14B—H14E	109.5
H15A—C15A—H15C	109.5	O2B—C14B—H14F	109.5
H15B—C15A—H15C	109.5	H14D—C14B—H14F	109.5
O4A—C16A—H16A	109.5	H14E—C14B—H14F	109.5
O4A—C16A—H16B	109.5	O3B—C15B—H15D	109.5
H16A—C16A—H16B	109.5	O3B—C15B—H15E	109.5
O4A—C16A—H16C	109.5	H15D—C15B—H15E	109.5
H16A—C16A—H16C	109.5	O3B—C15B—H15F	109.5
H16B—C16A—H16C	109.5	H15D—C15B—H15F	109.5
C4B—S1B—C1B	92.4 (4)	H15E—C15B—H15F	109.5
C9B—O2B—C14B	118.2 (3)	O4B—C16B—H16D	109.5
C11B—O3B—C15B	117.3 (3)	O4B—C16B—H16E	109.5
C13B—O4B—C16B	117.3 (3)	H16D—C16B—H16E	109.5
C2B—C1B—S1B	112.0 (7)	O4B—C16B—H16F	109.5
C2B—C1B—H1B	124.0	H16D—C16B—H16F	109.5
S1B—C1B—H1B	124.0	H16E—C16B—H16F	109.5

C1B—C2B—C3B	110.0 (10)
C4A—S1A—C1A—C2A	1.0 (3)
S1A—C1A—C2A—C3A	−1.3 (5)
C1A—C2A—C3A—C4A	0.9 (5)
C2A—C3A—C4A—C5A	176.5 (4)
C2A—C3A—C4A—S1A	−0.2 (5)
C1A—S1A—C4A—C3A	−0.5 (3)
C1A—S1A—C4A—C5A	−177.6 (3)
C3A—C4A—C5A—O1A	−171.1 (4)
S1A—C4A—C5A—O1A	5.4 (5)
C3A—C4A—C5A—C6A	11.1 (6)
S1A—C4A—C5A—C6A	−172.3 (3)
O1A—C5A—C6A—C7A	5.8 (6)
C4A—C5A—C6A—C7A	−176.6 (4)
C5A—C6A—C7A—C8A	−178.5 (4)
C6A—C7A—C8A—C13A	−5.4 (7)
C6A—C7A—C8A—C9A	176.6 (4)
C14A—O2A—C9A—C10A	−5.3 (5)
C14A—O2A—C9A—C8A	177.2 (3)
C13A—C8A—C9A—O2A	179.4 (3)
C7A—C8A—C9A—O2A	−2.4 (5)
C13A—C8A—C9A—C10A	1.9 (5)
C7A—C8A—C9A—C10A	−179.9 (4)
O2A—C9A—C10A—C11A	−177.8 (4)
C8A—C9A—C10A—C11A	−0.5 (6)
C15A—O3A—C11A—C12A	3.7 (5)
C15A—O3A—C11A—C10A	−176.6 (3)
C9A—C10A—C11A—O3A	179.4 (3)
C9A—C10A—C11A—C12A	−1.0 (6)
O3A—C11A—C12A—C13A	−179.4 (3)
C10A—C11A—C12A—C13A	1.0 (6)
C16A—O4A—C13A—C12A	0.3 (5)
C16A—O4A—C13A—C8A	−179.5 (3)
C11A—C12A—C13A—O4A	−179.2 (4)
C11A—C12A—C13A—C8A	0.6 (6)
C9A—C8A—C13A—O4A	177.9 (3)
C7A—C8A—C13A—O4A	−0.2 (6)
C9A—C8A—C13A—C12A	−1.9 (5)
C7A—C8A—C13A—C12A	−180.0 (4)
C4B—S1B—C1B—C2B	1.4 (8)
S1B—C1B—C2B—C3B	1.8 (12)
C1B—C2B—C3B—C4B	−5.1 (14)
C4B—S1X—C1X—C2X	−2(2)
S1X—C1X—C2X—C3X	7(3)
C1X—C2X—C3X—C4B	−9(3)
C2B—C3B—C4B—C3X	5.4 (16)
C2B—C3B—C4B—C5B	179.0 (7)
C2B—C3B—C4B—S1X	−121 (7)
C2B—C3B—C4B—S1B	6.2 (11)
C2X—C3X—C4B—C3B	3(2)
C2X—C3X—C4B—C5B	−171.8 (15)
C2X—C3X—C4B—S1X	8(2)
C1X—S1X—C4B—C3B	51 (7)
C1X—S1X—C4B—C3X	−3.7 (16)
C1X—S1X—C4B—C5B	176.0 (10)
C1X—S1X—C4B—S1B	−3.0 (10)
C1B—S1B—C4B—C3B	−4.2 (7)
C1B—S1B—C4B—C5B	−178.0 (4)
C1B—S1B—C4B—S1X	1.1 (5)
C3B—C4B—C5B—O1B	−176.9 (7)
C3X—C4B—C5B—O1B	−3.9 (14)
S1X—C4B—C5B—O1B	176.4 (5)
S1B—C4B—C5B—O1B	−4.6 (5)
C3B—C4B—C5B—C6B	4.9 (9)
C3X—C4B—C5B—C6B	177.9 (13)
S1X—C4B—C5B—C6B	−1.7 (6)
S1B—C4B—C5B—C6B	177.3 (3)
O1B—C5B—C6B—C7B	6.6 (6)
C4B—C5B—C6B—C7B	−175.3 (4)
C5B—C6B—C7B—C8B	−176.7 (4)
C6B—C7B—C8B—C9B	169.3 (4)
C6B—C7B—C8B—C13B	−9.2 (7)
C14B—O2B—C9B—C10B	0.6 (5)
C14B—O2B—C9B—C8B	−177.6 (3)
C13B—C8B—C9B—O2B	179.6 (3)
C7B—C8B—C9B—O2B	1.0 (5)
C13B—C8B—C9B—C10B	1.5 (6)
C7B—C8B—C9B—C10B	−177.1 (4)
O2B—C9B—C10B—C11B	−177.9 (3)
C8B—C9B—C10B—C11B	0.2 (6)
C15B—O3B—C11B—C12B	178.0 (3)
C15B—O3B—C11B—C10B	−1.0 (5)
C9B—C10B—C11B—O3B	177.6 (3)
C9B—C10B—C11B—C12B	−1.4 (6)
O3B—C11B—C12B—C13B	−178.2 (3)
C10B—C11B—C12B—C13B	0.9 (6)
C16B—O4B—C13B—C12B	0.6 (5)
C16B—O4B—C13B—C8B	−179.7 (3)
C11B—C12B—C13B—O4B	−179.4 (3)
C11B—C12B—C13B—C8B	0.9 (6)
C9B—C8B—C13B—O4B	178.2 (3)
C7B—C8B—C13B—O4B	−3.2 (6)
C9B—C8B—C13B—C12B	−2.1 (6)
C7B—C8B—C13B—C12B	176.5 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2B—H2B···O3A	0.93	2.56	3.482 (10)	171
C6A—H6A···O4A	0.93	2.22	2.815 (4)	121
C6B—H6B···O4B	0.93	2.24	2.824 (4)	120
C15A—H15C···O1B^{i}	0.96	2.51	3.451 (5)	166
C15B—H15F···O1A^{ii}	0.96	2.55	3.355 (5)	141
C16B—H16E···O3B^{iii}	0.96	2.59	3.401 (4)	142

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*−1; (ii) −*x*+1, −*y*+1, *z*−1/2; (iii) −*x*+1, −*y*+1, *z*+1/2.

Fig. 1

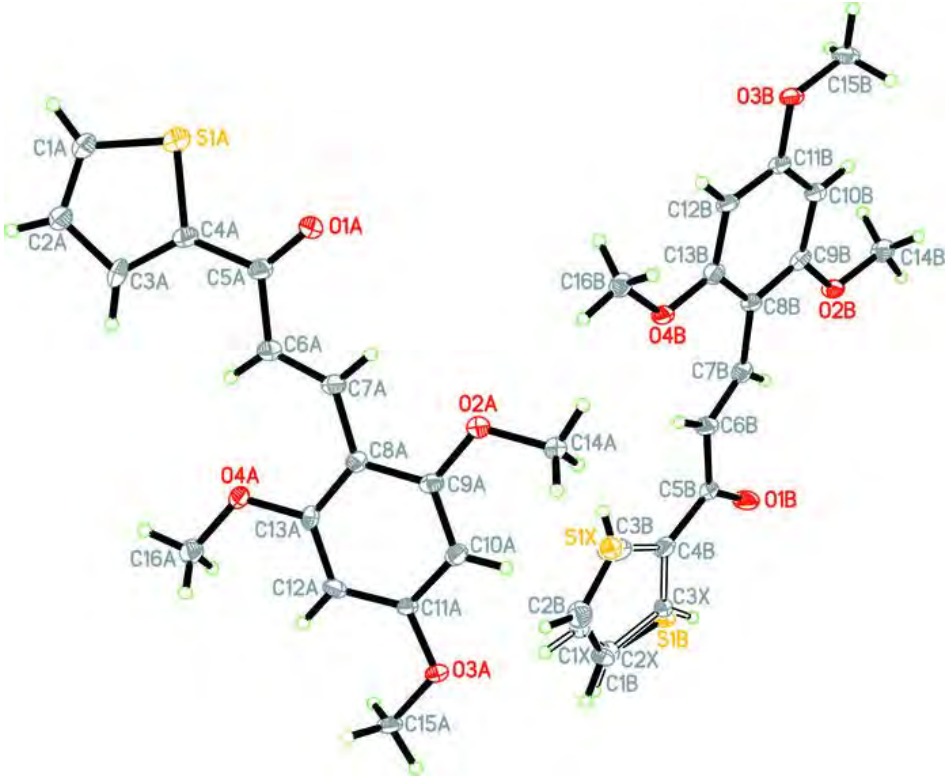
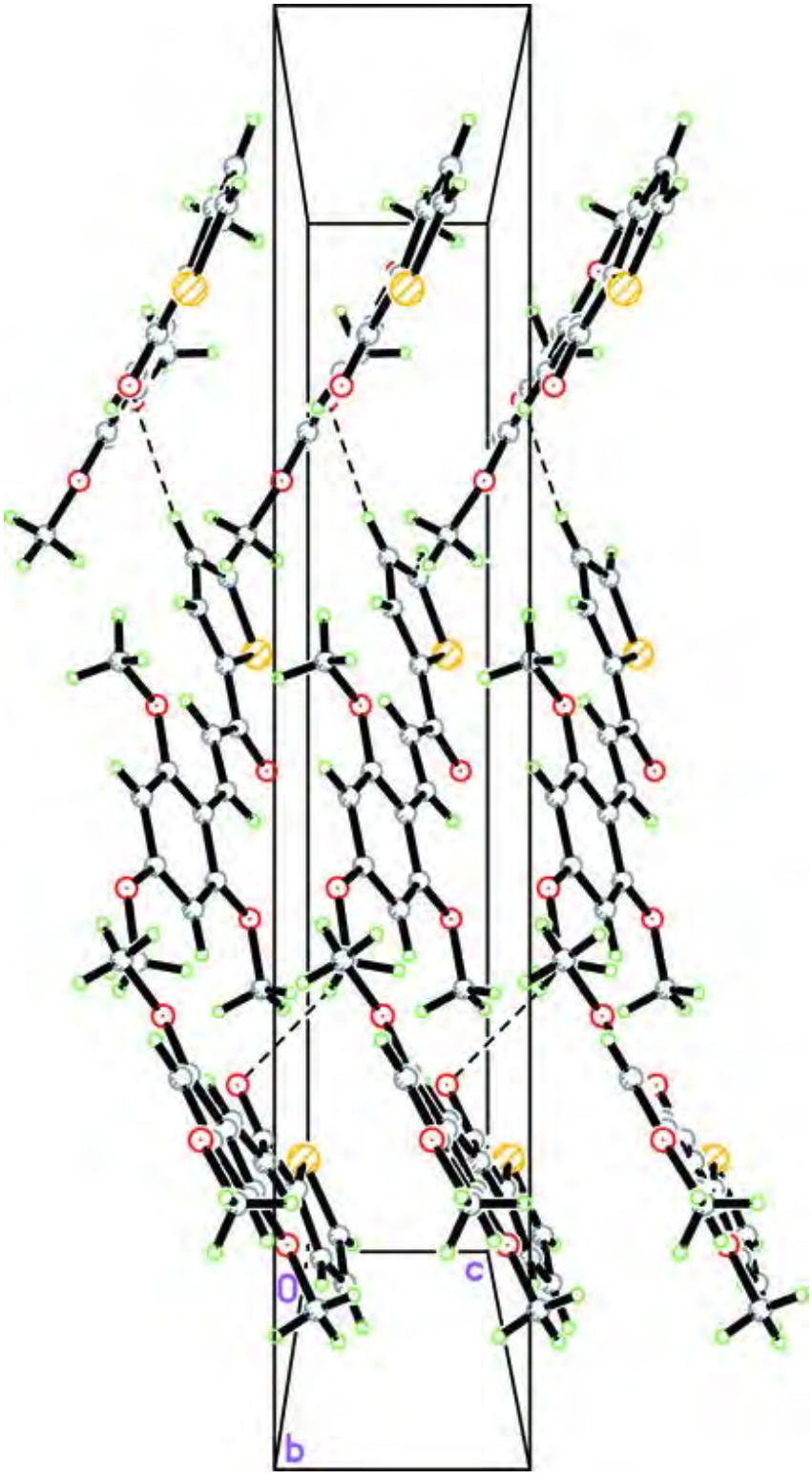


Fig. 2

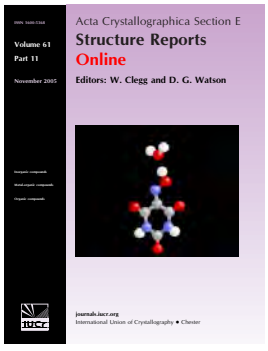


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, J. Simpson and M. Weil**

(*E*)-1-(2-Furyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun, Thitipone Suwunwong, Suchada Chantrapromma and Chatchanok Karalai

Acta Cryst. (2010). E**66**, o3070–o3071



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(2-Furyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

Hoong-Kun Fun,[a]*‡ Thitipone Suwunwong,[b] Suchada Chantrapromma[b]§ and Chatchanok Karalai[b]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

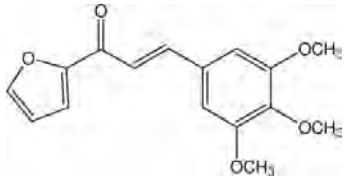
Received 28 October 2010; accepted 31 October 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.092; data-to-parameter ratio = 8.1.

The title molecule, $C_{16}H_{16}O_5$, is twisted; the dihedral angle between the furan and 3,4,5-trimethoxyphenyl rings is 12.14 (13)°. The two methoxy groups at the *meta* positions of the benzene ring are close to being coplanar with the ring [C—O—C—C = −0.6 (3) and 1.4 (3)°], whereas the third methoxy group, at the *para* position, is (+)-anticlinal with respect to the benzene ring [C—O—C—C = 104.9 (2)°]. In the crystal, molecules are linked by weak C—H···O bonds to stack along the *b* axis and further C—H···O interactions consolidate the structure.

Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Fun *et al.* (2010); Suwunwong *et al.* (2009). For background to and applications of chalcones, see: Batovska *et al.* (2007); Gu *et al.* (2009); Jung *et al.* (2008); Prasad *et al.* (2008); Saxena *et al.* (2007); Tewtrakul *et al.* (2003).



Experimental

Crystal data

$C_{16}H_{16}O_5$
M_r = 288.29
Orthorhombic, $Pna2_1$
a = 24.2677 (4) Å
b = 3.9916 (1) Å
c = 14.0816 (2) Å
V = 1364.04 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.35 × 0.24 × 0.21 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.965, T_{max} = 0.978
17261 measured reflections
2063 independent reflections
1907 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.092
S = 1.06
2063 reflections
254 parameters
1 restraint
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14B···O1^{i}	0.99 (3)	2.54 (3)	3.503 (3)	166 (2)
C15—H15B···O4^{ii}	1.01 (3)	2.36 (3)	3.337 (3)	162 (2)

Symmetry codes: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, z-\frac{1}{2}$; (ii) $x, y+1, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (TRF) for the research grant (RSA5280033) and the Prince of Songkla University for financial support. The authors also thank Universiti Sains Malaysia for the Research University grant No. 1001/PFIZIK/811160.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5713).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Batovska, D., Parushev, St., Slovova, A., Bankova, V., Tsvetkova, I., Ninova, M. & Najdenski, H. (2007). *Eur. J. Med. Chem.* **42**, 87–92.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fun, H.-K., Suwunwong, T., Chantrapromma, S. & Karalai, C. (2010). *Acta Cryst.* E**66**, o2559–o2560.
Gu, B., Ji, W., Huang, X.-Q., Patil, P. S. & Dharmaprakash, S. M. (2009). *Opt. Express*, **17**, 1126–1135.
Jung, Y. J., Son, K. I., Oh, Y. E. & Noh, D. Y. (2008). *Polyhedron*, **27**, 861–867.
Prasad, Y. R., Kumar, P. R., Smile, D. J. & Babu, P. A. (2008). *ARKIVOC*, **11**, 266–276.
Saxena, H. O., Faridi, U., Kumar, J. K., Luqman, S., Darokar, M. P., Shanker, K., Chanotiya, C. S., Gupta, M. M. & Negi, A. S. (2007). *Steroids*, **72**, 892–900.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Suwunwong, T., Chantrapromma, S., Pakdeevanich, P. & Fun, H.-K. (2009). *Acta Cryst.* E**65**, o1575–o1576.

Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol*, **25**, 503–508.

supplementary materials

Acta Cryst. (2010). E**66**, o3070-o3071 [**doi:10.1107/S160053681004451X**]

(*E*)-1-(2-Furyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

H.-K. Fun, T. Suwunwong, S. Chantrapromma and C. Karalai

Comment

Chalcones are known to exhibit bioactivity including antimicrobial (Prasad *et al.*, 2008), antifungal (Batovska *et al.*, 2007), anticancer (Saxena *et al.*, 2007) and HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003), as well as Non Linear Optical (NLO) (Gu *et al.*, 2009) and fluorescence properties (Jung *et al.*, 2008). In our on-going research on antibacterial activities, NLO and fluorescence properties of aryl/heteroaryl chalcones, we previously reported the crystal structures of (*E*)-1-(2-furyl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one (I) (Fun *et al.*, 2010) and (*E*)-1-(2-thienyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one (II) (Suwunwong *et al.*, 2009). The title compound (III) was synthesized and its crystal structure was determined in order to gain structural details to explain the affects of the substituent groups and their positions on the fluorescence properties and how their crystal packing would affect the NLO properties of the compounds. Compound (I) crystallized out in the centrosymmetric *C2/c* space group which prohibits second order NLO properties whereas compounds (II) and (III) crystallized in non-centrosymmetric $Pna2_1$ space group and should possess the second order NLO properties.

The molecule of the title heteroaryl chalcone (Fig. 1) exists in an *E* configuration with respect to the C6═C7 double bond [1.340 (3) Å] with torsion angle C5–C6–C7–C8 = 174.83 (19)°. The molecule is twisted with the dihedral angle between the furan and 3,4,5-trimethoxyphenyl rings being 12.14 (13)°. The propenone unit (C5—C7/O1) is slightly deviated with the torsion angle O1–C5–C6–C7 = 8.0 (3)°. The three methoxy groups of the 3,4,5-trimethoxyphenyl unit have two different orientations: the two methoxy groups at the *meta* positions (at atom C10 and C12 positions) are co-planar with the attached benzene ring with torsion angles C14–O3–C10–C9 = -0.6 (3)° and C16–O5–C12–C13 = 1.4 (3)° whereas the third one at *para* position (at atom C11) is (+)-anti-clinally attached with the torsion angle C15–O4–C11—C10 = 104.9 (2)°. Otherwise, the bond distances in (III) are of normal values (Allen *et al.*, 1987) and are comparable with the closely related structures (Fun *et al.*, 2010; Suwunwong *et al.*, 2009).

In the crystal (Fig. 2), the molecules are stacked into columns along the *b* axis and molecules within the stacks are linked by weak C15—H15B···O4 (Table 1) interactions.

Experimental

The title compound was synthesized by the condensation of 3,4,5-trimethoxybenzaldehyde (0.39 g, 2 mmol) in ethanol (15 ml) with 2-furyl methylketone (0.22 g, 2 mmol) in ethanol (15 ml) in the presence of 20% NaOH(aq) (5 ml). After stirring for 4 h, the resulting pale yellow solid appeared and was then collected by filtration, washed with distilled water and dried (69% yield). Pale yellow blocks of (III) were recrystalized from acetone/methanol (1:1 *v*/*v*) by the slow evaporation of the solvent at room temperature after several days, Mp. 420–421 K.

Refinement

All H atoms were located in difference maps and refined isotropically. The highest residual electron density peak is located at 0.89 Å from C2 and the deepest hole is located at 0.70 Å from O2. A total of 1892 Friedel pairs were merged before final refinement.

Figures

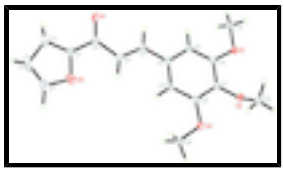


Fig. 1. The molecular structure of (III), showing 50% probability displacement ellipsoids.

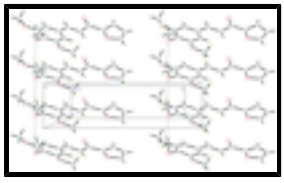


Fig. 2. The crystal packing of (III), showing column along the *b* axis. C—H···O weak interactions are shown as dashed lines.

(*E*)-1-(2-Furyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{16}H_{16}O_5$	D_x = 1.404 Mg m^{-3}
M_r = 288.29	Melting point = 420–421 K
Orthorhombic, *Pna*2$_1$	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 2063 reflections
a = 24.2677 (4) Å	θ = 2.2–30.0°
b = 3.9916 (1) Å	μ = 0.11 mm^{-1}
c = 14.0816 (2) Å	*T* = 100 K
V = 1364.04 (5) Å^3	Block, pale yellow
Z = 4	0.35 × 0.24 × 0.21 mm
F(000) = 608	

Data collection

Bruker APEXII CCD diffractometer	2063 independent reflections
Radiation source: sealed tube	1907 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.035
φ and ω scans	θ_{max} = 30.0°, θ_{min} = 2.2°
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	*h* = −29→34
T_{min} = 0.965, T_{max} = 0.978	*k* = −5→5
17261 measured reflections	*l* = −19→19

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.092$	All H-atom parameters refined
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.4265P]$ where $P = (F_o^2 + 2F_c^2)/3$
2063 reflections	$(\Delta/\sigma)_{max} = 0.001$
254 parameters	$\Delta\rho_{max} = 0.36$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.25005 (6)	0.6063 (4)	0.72174 (11)	0.0251 (3)
O2	0.34121 (7)	0.1635 (4)	0.87377 (12)	0.0271 (4)
O3	0.41052 (6)	0.5004 (4)	0.26993 (10)	0.0181 (3)
O4	0.50168 (6)	0.1761 (4)	0.32189 (11)	0.0181 (3)
O5	0.51882 (6)	−0.0146 (4)	0.50322 (11)	0.0190 (3)
C1	0.33965 (11)	0.1379 (8)	0.96954 (19)	0.0333 (6)
H1A	0.3722 (14)	0.001 (7)	0.995 (2)	0.036 (8)*
C2	0.29645 (12)	0.3011 (8)	1.00503 (17)	0.0342 (6)
H2A	0.2846 (17)	0.331 (11)	1.057 (3)	0.064 (13)*
C3	0.26677 (10)	0.4471 (7)	0.92549 (17)	0.0247 (5)
H3A	0.2426 (12)	0.568 (8)	0.922 (2)	0.026 (8)*
C4	0.29621 (8)	0.3538 (5)	0.84793 (14)	0.0171 (4)
C5	0.28952 (8)	0.4380 (5)	0.74728 (14)	0.0168 (4)
C6	0.33353 (8)	0.3224 (5)	0.68267 (15)	0.0171 (4)
H6A	0.3628 (11)	0.190 (7)	0.709 (2)	0.020 (6)*
C7	0.33640 (8)	0.4294 (5)	0.59260 (14)	0.0167 (4)

H7A	0.3082 (12)	0.566 (7)	0.570 (2)	0.023 (7)*
C8	0.37985 (8)	0.3565 (5)	0.52384 (13)	0.0155 (4)
C9	0.37237 (8)	0.4630 (5)	0.43008 (15)	0.0154 (3)
H9A	0.3401 (11)	0.568 (7)	0.410 (2)	0.019 (6)*
C10	0.41374 (8)	0.4064 (5)	0.36290 (13)	0.0143 (3)
C11	0.46246 (8)	0.2434 (5)	0.38936 (13)	0.0145 (3)
C12	0.46971 (8)	0.1383 (5)	0.48394 (14)	0.0151 (4)
C13	0.42857 (8)	0.1923 (5)	0.55057 (14)	0.0155 (4)
H13A	0.4332 (10)	0.125 (6)	0.615 (2)	0.014 (6)*
C14	0.36071 (8)	0.6651 (6)	0.24098 (15)	0.0189 (4)
H14C	0.3554 (11)	0.873 (7)	0.274 (2)	0.020 (7)*
H14B	0.3290 (12)	0.513 (7)	0.248 (2)	0.019 (7)*
H14A	0.3628 (12)	0.717 (7)	0.177 (2)	0.023 (7)*
C15	0.54913 (9)	0.3915 (6)	0.32550 (18)	0.0224 (4)
H15A	0.5639 (17)	0.395 (12)	0.393 (3)	0.069 (13)*
H15B	0.5367 (14)	0.627 (8)	0.309 (2)	0.040 (9)*
H15C	0.5741 (12)	0.315 (8)	0.277 (2)	0.032 (8)*
C16	0.52677 (9)	−0.1308 (6)	0.59880 (14)	0.0198 (4)
H16A	0.4975 (11)	−0.287 (7)	0.617 (2)	0.022 (7)*
H16B	0.5618 (12)	−0.249 (7)	0.598 (2)	0.023 (7)*
H16C	0.5293 (11)	0.058 (8)	0.645 (2)	0.021 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0218 (7)	0.0366 (8)	0.0169 (7)	0.0091 (7)	0.0035 (6)	0.0052 (7)
O2	0.0229 (7)	0.0313 (9)	0.0272 (9)	−0.0017 (7)	−0.0050 (6)	0.0068 (7)
O3	0.0184 (7)	0.0243 (7)	0.0114 (6)	0.0016 (6)	0.0005 (5)	0.0028 (5)
O4	0.0166 (6)	0.0244 (7)	0.0133 (6)	−0.0004 (6)	0.0038 (5)	−0.0033 (6)
O5	0.0166 (6)	0.0255 (7)	0.0149 (7)	0.0045 (6)	0.0004 (5)	0.0027 (6)
C1	0.0318 (12)	0.0426 (15)	0.0256 (12)	−0.0123 (11)	−0.0095 (9)	0.0101 (10)
C2	0.0455 (15)	0.0456 (15)	0.0113 (9)	−0.0270 (12)	0.0002 (10)	−0.0011 (10)
C3	0.0218 (9)	0.0340 (12)	0.0182 (10)	−0.0103 (10)	0.0068 (8)	−0.0077 (9)
C4	0.0156 (8)	0.0214 (9)	0.0142 (9)	−0.0018 (7)	0.0018 (7)	0.0023 (7)
C5	0.0161 (8)	0.0217 (9)	0.0126 (8)	−0.0029 (7)	0.0028 (7)	0.0009 (7)
C6	0.0154 (8)	0.0185 (9)	0.0175 (9)	0.0017 (7)	0.0017 (7)	−0.0001 (8)
C7	0.0126 (8)	0.0226 (10)	0.0149 (8)	0.0008 (7)	0.0017 (7)	−0.0017 (7)
C8	0.0142 (8)	0.0190 (9)	0.0134 (8)	−0.0022 (7)	0.0008 (6)	−0.0009 (7)
C9	0.0145 (7)	0.0187 (9)	0.0130 (8)	−0.0004 (7)	0.0001 (7)	−0.0006 (7)
C10	0.0162 (8)	0.0154 (8)	0.0112 (8)	−0.0023 (7)	−0.0006 (7)	−0.0001 (7)
C11	0.0141 (7)	0.0167 (8)	0.0127 (8)	−0.0017 (7)	0.0035 (6)	−0.0018 (7)
C12	0.0139 (8)	0.0160 (9)	0.0153 (9)	0.0003 (7)	−0.0017 (6)	−0.0016 (7)
C13	0.0164 (8)	0.0195 (9)	0.0107 (8)	−0.0022 (7)	0.0008 (7)	0.0002 (7)
C14	0.0185 (9)	0.0226 (10)	0.0157 (9)	0.0005 (8)	−0.0033 (7)	0.0026 (8)
C15	0.0196 (9)	0.0214 (10)	0.0262 (10)	−0.0027 (8)	0.0090 (8)	−0.0005 (9)
C16	0.0193 (9)	0.0240 (10)	0.0161 (9)	0.0014 (8)	−0.0025 (8)	0.0046 (8)

Geometric parameters (Å, °)

O1—C5	1.224 (3)	C7—C8	1.461 (3)
O2—C1	1.353 (3)	C7—H7A	0.93 (3)
O2—C4	1.379 (3)	C8—C9	1.399 (3)
O3—C10	1.364 (2)	C8—C13	1.403 (3)
O3—C14	1.435 (2)	C9—C10	1.398 (3)
O4—C11	1.372 (2)	C9—H9A	0.93 (3)
O4—C15	1.438 (3)	C10—C11	1.400 (3)
O5—C12	1.366 (2)	C11—C12	1.407 (3)
O5—C16	1.437 (2)	C12—C13	1.387 (3)
C1—C2	1.332 (5)	C13—H13A	0.96 (3)
C1—H1A	1.03 (3)	C14—H14C	0.96 (3)
C2—C3	1.454 (4)	C14—H14B	0.99 (3)
C2—H2A	0.80 (5)	C14—H14A	0.93 (3)
C3—C4	1.357 (3)	C15—H15A	1.02 (5)
C3—H3A	0.76 (3)	C15—H15B	1.01 (3)
C4—C5	1.466 (3)	C15—H15C	0.96 (3)
C5—C6	1.477 (3)	C16—H16A	0.98 (3)
C6—C7	1.340 (3)	C16—H16B	0.97 (3)
C6—H6A	0.96 (3)	C16—H16C	1.00 (3)
C1—O2—C4	106.4 (2)	O3—C10—C9	124.33 (18)
C10—O3—C14	116.54 (16)	O3—C10—C11	115.57 (17)
C11—O4—C15	114.47 (16)	C9—C10—C11	120.10 (17)
C12—O5—C16	116.60 (16)	O4—C11—C10	119.52 (17)
C2—C1—O2	111.0 (2)	O4—C11—C12	120.68 (17)
C2—C1—H1A	137.1 (19)	C10—C11—C12	119.74 (17)
O2—C1—H1A	111.8 (19)	O5—C12—C13	124.26 (18)
C1—C2—C3	107.3 (2)	O5—C12—C11	115.50 (17)
C1—C2—H2A	135 (3)	C13—C12—C11	120.25 (17)
C3—C2—H2A	118 (3)	C12—C13—C8	119.85 (18)
C4—C3—C2	104.4 (2)	C12—C13—H13A	121.0 (15)
C4—C3—H3A	122 (3)	C8—C13—H13A	119.1 (15)
C2—C3—H3A	133 (3)	O3—C14—H14C	111.7 (17)
C3—C4—O2	110.8 (2)	O3—C14—H14B	110.3 (16)
C3—C4—C5	131.1 (2)	H14C—C14—H14B	112 (2)
O2—C4—C5	117.99 (17)	O3—C14—H14A	109.5 (18)
O1—C5—C4	119.79 (18)	H14C—C14—H14A	107 (3)
O1—C5—C6	123.80 (19)	H14B—C14—H14A	106 (2)
C4—C5—C6	116.36 (18)	O4—C15—H15A	109 (3)
C7—C6—C5	121.40 (19)	O4—C15—H15B	107.9 (19)
C7—C6—H6A	120.0 (17)	H15A—C15—H15B	108 (3)
C5—C6—H6A	118.1 (17)	O4—C15—H15C	106.8 (19)
C6—C7—C8	126.96 (19)	H15A—C15—H15C	116 (3)
C6—C7—H7A	118.4 (18)	H15B—C15—H15C	109 (3)
C8—C7—H7A	114.6 (18)	O5—C16—H16A	110.7 (17)
C9—C8—C13	120.28 (17)	O5—C16—H16B	105.3 (17)
C9—C8—C7	118.13 (18)	H16A—C16—H16B	109 (2)

C13—C8—C7	121.57 (17)	O5—C16—H16C	111.9 (18)
C10—C9—C8	119.77 (17)	H16A—C16—H16C	111 (2)
C10—C9—H9A	118.2 (18)	H16B—C16—H16C	109 (2)
C8—C9—H9A	122.0 (18)		
C4—O2—C1—C2	−0.1 (3)	C14—O3—C10—C11	179.43 (17)
O2—C1—C2—C3	0.1 (3)	C8—C9—C10—O3	−179.95 (18)
C1—C2—C3—C4	−0.1 (3)	C8—C9—C10—C11	0.1 (3)
C2—C3—C4—O2	0.0 (2)	C15—O4—C11—C10	104.9 (2)
C2—C3—C4—C5	−175.9 (2)	C15—O4—C11—C12	−77.7 (2)
C1—O2—C4—C3	0.1 (3)	O3—C10—C11—O4	−2.9 (3)
C1—O2—C4—C5	176.5 (2)	C9—C10—C11—O4	177.11 (18)
C3—C4—C5—O1	−4.4 (4)	O3—C10—C11—C12	179.71 (18)
O2—C4—C5—O1	179.97 (19)	C9—C10—C11—C12	−0.3 (3)
C3—C4—C5—C6	172.9 (2)	C16—O5—C12—C13	1.4 (3)
O2—C4—C5—C6	−2.7 (3)	C16—O5—C12—C11	−178.70 (17)
O1—C5—C6—C7	8.0 (3)	O4—C11—C12—O5	3.4 (3)
C4—C5—C6—C7	−169.2 (2)	C10—C11—C12—O5	−179.21 (17)
C5—C6—C7—C8	174.83 (19)	O4—C11—C12—C13	−176.68 (18)
C6—C7—C8—C9	173.3 (2)	C10—C11—C12—C13	0.7 (3)
C6—C7—C8—C13	−8.3 (3)	O5—C12—C13—C8	179.05 (18)
C13—C8—C9—C10	−0.2 (3)	C11—C12—C13—C8	−0.8 (3)
C7—C8—C9—C10	178.14 (18)	C9—C8—C13—C12	0.6 (3)
C14—O3—C10—C9	−0.6 (3)	C7—C8—C13—C12	−177.69 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14B···O1^{i}	0.99 (3)	2.54 (3)	3.503 (3)	166 (2)
C15—H15B···O4^{ii}	1.01 (3)	2.36 (3)	3.337 (3)	162 (2)

Symmetry codes: (i) −*x*+1/2, *y*−1/2, *z*−1/2; (ii) *x*, *y*+1, *z*.

Fig. 1

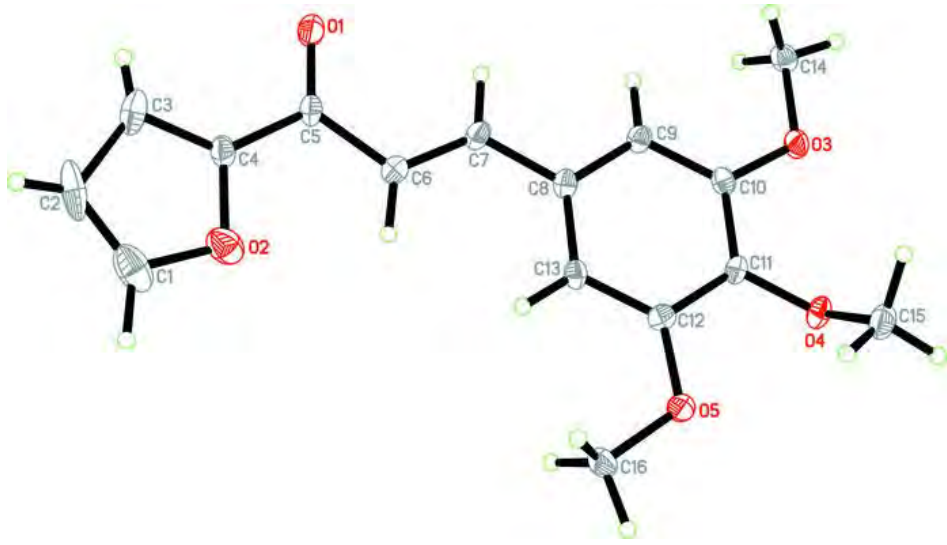
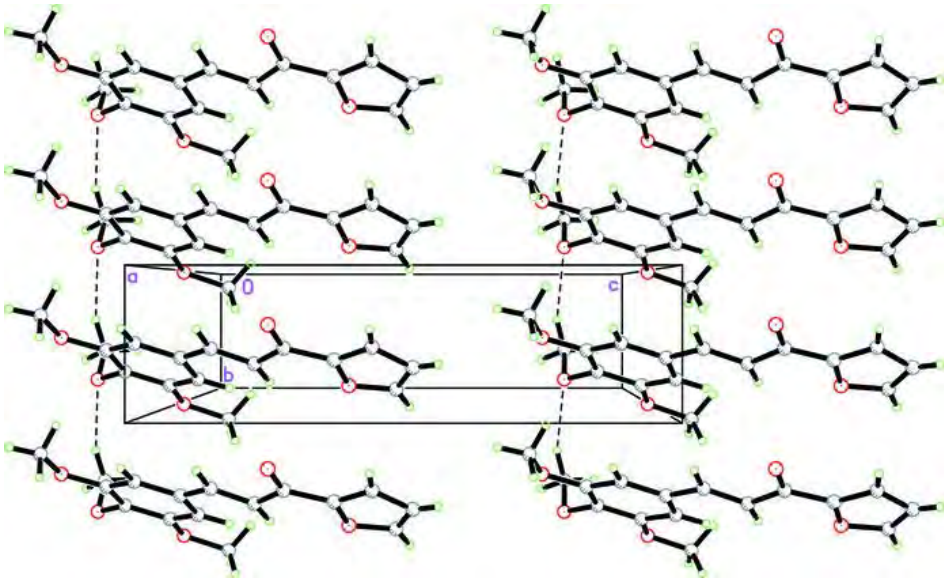


Fig. 2

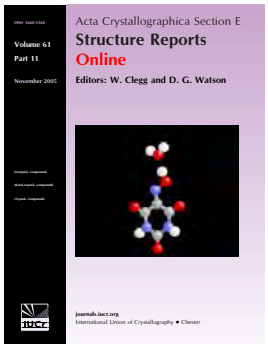


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, H. Stoeckli-Evans, E. R. T. Tiekink and M. Weil**

3-(4-Chlorophenyl)-5-(thiophen-2-yl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide

Hoong-Kun Fun, Thitipone Suwunwong and Suchada Chantrapromma

Acta Cryst. (2012). E**68**, o259–o260



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(4-Chlorophenyl)-5-(thiophen-2-yl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide

Hoong-Kun Fun,[a]*‡ Thitipone Suwunwong[b] and Suchada Chantrapromma[b]§

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

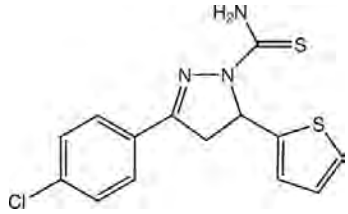
Received 3 December 2011; accepted 20 December 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.049; wR factor = 0.138; data-to-parameter ratio = 19.9.

In the title pyrazoline derivative, $C_{14}H_{12}ClN_3S_2$, the thiophene ring is disordered over two orientations with a refined site-occupancy ratio of 0.832 (4):0.168 (4). The pyrazoline ring adopts an envelope conformation with the C atom linking the thiophene ring at the flap. The dihedral angles between the benzene ring and the major and minor components of the thiophene ring are 88.6 (3) and 85.6 (15)°, respectively while the dihedral angle between the disorder components of the ring is 3.1 (16)°. The mean plane of the pyrazoline ring makes dihedral angles of 11.86 (13), 80.1 (3) and 83.0 (15)°, respectively, with the benzene ring, and the major and minor components of the thiophene ring. An intramolecular N(amide)—H···N(pyrazoline) hydrogen bond generates an S(5) ring motif. In the crystal, molecules are linked by weak C—H···S and N(amide)—H···S interactions into a tape along [10$\bar{1}$]. C—H···π interactions are also observed.

Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For ring conformations, see: Cremer & Pople (1975). For related structures, see: Fun *et al.* (2011); Nonthason *et al.* (2011). For background to and applications of pyrazoline derivatives, see: Bai *et al.* (2007); Gong *et al.* (2011); Husain *et al.* (2008); Khode *et al.* (2009); Shoman *et al.* (2009); Taj *et al.* (2011). For the stability of the temperature controller, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{14}H_{12}ClN_3S_2$
M_r = 321.86
Monoclinic, $P2_1/n$
a = 6.7784 (3) Å
b = 25.2104 (11) Å
c = 8.4628 (4) Å
β = 90.339 (2)°
V = 1446.15 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 100 K
0.56 × 0.09 × 0.08 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.749, T_{max} = 0.958
32828 measured reflections
4206 independent reflections
3801 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.138
S = 1.10
4206 reflections
211 parameters
10 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 and *Cg*2 are the centroids of the S1*A*/C1*A*–C3*A*/C4 and S1*B*/C1*B*–C3*B*/C4 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1*N*3···N2	0.90 (4)	2.28 (4)	2.656 (3)	105 (3)
N3—H2*N*3···S2^{i}	0.89 (4)	2.52 (4)	3.400 (3)	170 (3)
C5—H5*A*···S1*A*^{ii}	1.00	2.86	3.664 (3)	138
C9—H9*A*···*Cg*1^{iii}	0.95	2.79	3.628 (4)	148
C9—H9*A*···*Cg*2^{iii}	0.95	2.77	3.595 (18)	145

Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $-x+1, -y+2, -z+2$; (iii) $x-1, y, z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Prince of Songkla University for financial support. The authors also thank the Thailand Research Fund (TRF) for a research grant (RSA5280033) and Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5024).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bai, G., Li, J., Li, D., Dong, C., Han, X. & Lin, P. (2007). *Dyes Pigm.* **75**, 93–98.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Fun, H.-K., Suwunwong, T. & Chantrapromma, S. (2011). *Acta Cryst.* E**67**, o701–o702.

Gong, Z.-L., Xie, Y.-S., Zhao, B.-X., Lv, H.-S. & Liu, W.-Y. (2011). *J. Fluoresc.* **21**, 355–364.

Husain, K., Abid, M. & Azam, A. (2008). *Eur. J. Med. Chem.* **43**, 393–403.

Khode, S., Maddi, V., Aragade, P., Palkar, M., Ronad, P. K., Mamledesai, S., Thippeswamy, A. H. M. & Satyanarayana, D. (2009). *Eur. J. Med. Chem.* **44**, 1682–1688.

Nonthason, P., Suwunwong, T., Chantrapromma, S. & Fun, H.-K. (2011). *Acta Cryst.* E**67**, o3501–o3502.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Shoman, M. E., Abdel-Aziz, M., Aly, O. M., Farag, H. H. & Morsy, M. A. (2009). *Eur. J. Med. Chem.* **44**, 3068–3076.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Taj, T., Kamble, R. R., Gireesh, T. M., Hunnur, R. K. & Margankop, S. B. (2011). *Eur. J. Med. Chem.* **46**, 4366–4373.

supplementary materials

Acta Cryst. (2012). E**68**, o259-o260 [**doi:10.1107/S1600536811054754**]

3-(4-Chlorophenyl)-5-(thiophen-2-yl)-4,5-dihydro-1*H*-pyrazole-1-carbothioamide

H.-K. Fun, T. Suwunwong and S. Chantrapromma

Comment

The synthesis of pyrazoline derivatives which contain 5-membered heterocyclic structure have attracted a lot of interests in many fields, for example as in medicinal chemistry owing to their biological properties such as antiamoebic (Husain *et al.*, 2008), anti-inflammatory (Shoman *et al.*, 2009), analgesic (Khode *et al.*, 2009) and antioxidant (Taj *et al.*, 2011) activities, as well as in fluorescence (Bai *et al.*, 2007; Gong *et al.*, 2011) studies. Our on-going research on biological activities and fluorescent property of pyrazoline derivatives has led us to synthesize the title compound (I) in order to compare its biological activity with the related compounds (Fun *et al.*, 2011; Nonthason *et al.*, 2011).

In the molecule of (I), $C_{14}H_{12}ClN_3S_2$, the thiophene ring is disordered over two positions with the refined site-occupancy ratio of 0.832 (4):0.168 (4). The dihedral angles between the benzene and the major and minor components of the thiophene rings are 88.6 (3) and 85.6 (15)° respectively. The pyrazoline ring is in an envelope conformation [pucker atom at C5 with deviation of -0.125 (3) Å] with puckering parameter Q = 0.206 (3) Å and φ = 137.6 (7)° (Cremer & Pople, 1975). The dihedral angle between the mean plane through pyrazoline ring and the benzene ring is 11.86 (13)°, whereas these values are 80.1 (3) and 83.0 (15)° between the pyrazoline and the major and minor components of the thiophene ring. The carbothioamide unit lies almost on the same plane with pyrazoline ring as can be indicated by the torsion angles N2–N1–C14–N3 = -3.3 (4)° and C5–N1–C14–S2 = 0.4 (3)°. Intramolecular N3—H1N3···N2 hydrogen bond generate an S(5) ring motif (Bernstein *et al.*, 1995). Bond distances of (I) are in normal range (Allen *et al.*, 1987)

In the crystal packing, (Fig. 2), the molecules are linked by weak C5—H5A···S1A intermolecular interactions (Table 1) into cyclic centrosymmetric $R^2_2(8)$ dimers (Bernstein *et al.*, 1995). These dimers are further linked by N3—H2N3···S2 hydrogen bonds (Table 1) into a tape along the [10$\bar{1}$] direction (Fig. 2). The crystal is stabilized by N—H···S hydrogen bonds together with weak C—H···S and C—H···π interactions (Table 1).

Experimental

The title compound was synthesized by dissolving (*E*)-1-(4-chlorophenyl)-3-(2-thienyl)prop-2-en-1-one (0.25 g, 1.0 mmol) in a solution of KOH (0.06 g, 1.0 mmol) in ethanol (20 ml). An excess thiosemicarbazide (0.14 g, 1.5 mmol) in ethanol (20 ml) was then added, and the reaction mixture was vigorously stirred and refluxed for 4 h. The pale-yellow solid of the title compound obtained after cooling of the reaction was filtered off under vacuum. Pale yellow needle-shaped single crystals of the title compound suitable for *X*-ray structure determination were recrystalized from CH_3OH/CH_2Cl_2 (1:1 *v*/*v*) by slow evaporation of the solvent at room temperature after several days.

Refinement

Amide H atoms were located in a difference map and refined isotropically. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C—H) = 0.95 Å for aromatic and 0.99 Å for CH_2 atoms. The

U_{iso} values were constrained to be $1.2U_{eq}$ of the carrier atoms. The thiophene ring is disordered over two positions with the refined site-occupancy ratio of 0.832 (4):0.168 (4). In the final refinement, distances restraint was used. The highest residual electron density peak is located at 1.35 Å from Cl1 and the deepest hole is located at 0.52 Å from Cl1. The crystal was a pseudo-merohedral twin and the structure was refined with the twin law (-1 0 0 0 -1 0 0 0 1). The BASF was refined to 0.138 (1).

Figures

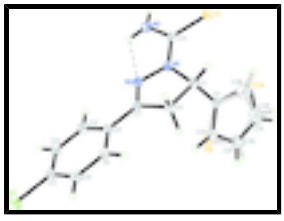


Fig. 1. The molecular structure of the title compound, showing 45% probability displacement ellipsoids and the atom-numbering scheme. Open bond show the minor *B* component. Intramolecular N—H···N hydrogen bond was shown as dash line.

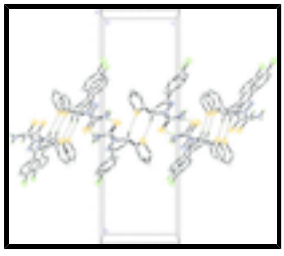


Fig. 2. The crystal packing of the title compound viewed along the *a* axis. Only the major component was shown. For clarify, only H atoms involved in hydrogen bonds were shown. Hydrogen bonds were shown as dashed lines.

3-(4-Chlorophenyl)-5-(thiophen-2-yl)-4,5-dihydro-1*H*- pyrazole-1-carbothioamide

Crystal data

$C_{14}H_{12}ClN_3S_2$	$F(000) = 664$
$M_r = 321.86$	$D_x = 1.478$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 4206 reflections
$a = 6.7784$ (3) Å	$\theta = 0.8$–$30.0°$
$b = 25.2104$ (11) Å	$\mu = 0.55$ mm^{-1}
$c = 8.4628$ (4) Å	$T = 100$ K
$\beta = 90.339$ (2)°	Needle, pale-yellow
$V = 1446.15$ (11) Å^3	0.56 × 0.09 × 0.08 mm
$Z = 4$	

Data collection

Bruker APEX DUO CCD area-detector diffractometer	4206 independent reflections
Radiation source: sealed tube	3801 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.047$
φ and ω scans	$\theta_{max} = 30.0°$, $\theta_{min} = 0.8°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	$h = -9 \rightarrow 9$

T_{min} = 0.749, T_{max} = 0.958	k = −35→35
32828 measured reflections	l = −11→11

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.049	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.138	H atoms treated by a mixture of independent and constrained refinement
S = 1.10	$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 1.9543P]$ where $P = (F_o^2 + 2F_c^2)/3$
4206 reflections	$(\Delta/\sigma)_{max}$ = 0.002
211 parameters	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
10 restraints	$\Delta\rho_{min}$ = −0.59 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	−0.33129 (11)	0.74126 (3)	0.43185 (9)	0.02922 (17)	
S2	0.81787 (10)	1.01564 (3)	0.70895 (8)	0.02383 (16)	
N1	0.5530 (3)	0.93930 (8)	0.6751 (3)	0.0187 (4)	
N2	0.4447 (3)	0.90237 (8)	0.5877 (2)	0.0182 (4)	
C9	−0.0433 (4)	0.85079 (10)	0.6843 (3)	0.0200 (5)	
H9A	−0.0680	0.8721	0.7747	0.024*	
N3	0.7384 (4)	0.95592 (10)	0.4561 (3)	0.0264 (5)	
C4	0.5995 (4)	0.91382 (10)	0.9528 (3)	0.0190 (4)	
S1A	0.81128 (18)	0.93898 (4)	1.03729 (11)	0.0196 (2)	0.832 (4)
C1A	0.8584 (10)	0.8815 (2)	1.1384 (9)	0.0242 (10)	0.832 (4)
H1AA	0.9681	0.8764	1.2071	0.029*	0.832 (4)
C2A	0.7184 (16)	0.8437 (3)	1.1078 (12)	0.0289 (15)	0.832 (4)
H2AA	0.7203	0.8090	1.1519	0.035*	0.832 (4)

C3A	0.5701 (18)	0.8623 (3)	1.0024 (14)	0.0263 (16)	0.832 (4)
H3AA	0.4610	0.8413	0.9692	0.032*	0.832 (4)
S1B	0.544 (2)	0.8499 (4)	0.9965 (18)	0.0243 (19)	0.168 (4)
C1B	0.751 (7)	0.8414 (15)	1.110 (8)	0.038 (14)*	0.168 (4)
H1BA	0.7880	0.8089	1.1588	0.046*	0.168 (4)
C2B	0.856 (7)	0.8873 (15)	1.122 (7)	0.041 (9)*	0.168 (4)
H2BA	0.9726	0.8912	1.1832	0.049*	0.168 (4)
C3B	0.770 (4)	0.9289 (10)	1.031 (4)	0.041 (9)*	0.168 (4)
H3BA	0.8240	0.9636	1.0250	0.049*	0.168 (4)
C5	0.4780 (4)	0.94575 (10)	0.8380 (3)	0.0186 (4)	
H5A	0.4751	0.9840	0.8691	0.022*	
C6	0.2675 (4)	0.92375 (11)	0.8167 (3)	0.0210 (5)	
H6A	0.2293	0.9009	0.9067	0.025*	
H6B	0.1698	0.9527	0.8047	0.025*	
C7	0.2870 (3)	0.89198 (9)	0.6660 (3)	0.0170 (4)	
C8	0.1379 (4)	0.85466 (9)	0.6075 (3)	0.0174 (4)	
C10	−0.1881 (4)	0.81612 (10)	0.6300 (3)	0.0207 (5)	
H10A	−0.3117	0.8140	0.6819	0.025*	
C11	−0.1501 (4)	0.78485 (10)	0.4997 (3)	0.0213 (5)	
C12	0.0296 (4)	0.78746 (11)	0.4212 (3)	0.0238 (5)	
H12A	0.0537	0.7655	0.3321	0.029*	
C13	0.1730 (4)	0.82256 (10)	0.4750 (3)	0.0219 (5)	
H13A	0.2957	0.8249	0.4217	0.026*	
C14	0.6989 (4)	0.96747 (10)	0.6069 (3)	0.0205 (5)	
H1N3	0.673 (6)	0.9294 (15)	0.408 (5)	0.030 (9)*	
H2N3	0.851 (6)	0.9675 (15)	0.416 (4)	0.032 (9)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0269 (3)	0.0216 (3)	0.0391 (4)	−0.0052 (2)	−0.0061 (3)	−0.0048 (3)
S2	0.0228 (3)	0.0227 (3)	0.0259 (3)	−0.0056 (2)	−0.0034 (2)	0.0011 (2)
N1	0.0178 (9)	0.0199 (10)	0.0183 (9)	−0.0026 (7)	−0.0012 (7)	−0.0010 (8)
N2	0.0172 (9)	0.0188 (9)	0.0186 (9)	−0.0006 (7)	−0.0026 (7)	0.0008 (7)
C9	0.0199 (11)	0.0196 (11)	0.0204 (11)	0.0018 (9)	−0.0006 (8)	−0.0018 (9)
N3	0.0265 (11)	0.0329 (13)	0.0198 (10)	−0.0107 (10)	0.0001 (9)	0.0018 (9)
C4	0.0185 (10)	0.0215 (11)	0.0171 (10)	−0.0012 (8)	0.0002 (8)	−0.0041 (9)
S1A	0.0192 (4)	0.0206 (4)	0.0188 (4)	0.0009 (4)	−0.0021 (3)	−0.0030 (3)
C1A	0.029 (2)	0.024 (2)	0.019 (2)	0.0046 (15)	−0.0052 (13)	−0.0011 (15)
C2A	0.037 (3)	0.023 (2)	0.027 (3)	−0.003 (2)	−0.006 (2)	0.0051 (14)
C3A	0.028 (3)	0.027 (4)	0.024 (2)	−0.009 (3)	−0.005 (2)	0.000 (3)
S1B	0.026 (4)	0.022 (4)	0.025 (3)	−0.007 (3)	−0.007 (2)	0.000 (3)
C5	0.0171 (10)	0.0196 (11)	0.0191 (10)	−0.0004 (8)	0.0004 (8)	−0.0037 (8)
C6	0.0185 (11)	0.0222 (11)	0.0224 (11)	−0.0010 (9)	0.0003 (9)	−0.0052 (9)
C7	0.0172 (10)	0.0163 (10)	0.0174 (10)	0.0007 (8)	−0.0031 (8)	0.0001 (8)
C8	0.0196 (11)	0.0150 (10)	0.0175 (10)	0.0009 (8)	−0.0020 (8)	0.0010 (8)
C10	0.0182 (10)	0.0182 (11)	0.0257 (12)	−0.0002 (9)	−0.0008 (9)	0.0008 (9)
C11	0.0218 (11)	0.0158 (10)	0.0263 (12)	−0.0019 (9)	−0.0047 (9)	−0.0003 (9)

C12	0.0270 (12)	0.0211 (12)	0.0231 (12)	−0.0001 (10)	−0.0027 (10)	−0.0062 (9)
C13	0.0219 (11)	0.0216 (11)	0.0222 (11)	0.0004 (10)	0.0011 (9)	−0.0028 (9)
C14	0.0200 (11)	0.0213 (11)	0.0201 (11)	−0.0015 (9)	−0.0029 (9)	0.0045 (9)

Geometric parameters (Å, °)

Cl1—C11	1.743 (3)	C2A—H2AA	0.9500
S2—C14	1.692 (3)	C3A—H3AA	0.9500
N1—C14	1.350 (3)	S1B—C1B	1.71 (2)
N1—N2	1.395 (3)	C1B—C2B	1.363 (18)
N1—C5	1.481 (3)	C1B—H1BA	0.9500
N2—C7	1.288 (3)	C2B—C3B	1.42 (2)
C9—C10	1.390 (3)	C2B—H2BA	0.9500
C9—C8	1.397 (3)	C3B—H3BA	0.9500
C9—H9A	0.9500	C5—C6	1.541 (3)
N3—C14	1.338 (3)	C5—H5A	1.0000
N3—H1N3	0.90 (4)	C6—C7	1.512 (3)
N3—H2N3	0.89 (4)	C6—H6A	0.9900
C4—C3A	1.379 (8)	C6—H6B	0.9900
C4—C3B	1.381 (18)	C7—C8	1.465 (3)
C4—C5	1.503 (3)	C8—C13	1.405 (3)
C4—S1B	1.696 (10)	C10—C11	1.381 (4)
C4—S1A	1.721 (3)	C10—H10A	0.9500
S1A—C1A	1.713 (6)	C11—C12	1.392 (4)
C1A—C2A	1.368 (6)	C12—C13	1.389 (4)
C1A—H1AA	0.9500	C12—H12A	0.9500
C2A—C3A	1.421 (12)	C13—H13A	0.9500
C14—N1—N2	120.6 (2)	C4—C3B—H3BA	123.4
C14—N1—C5	126.6 (2)	C2B—C3B—H3BA	123.4
N2—N1—C5	112.60 (19)	N1—C5—C4	110.7 (2)
C7—N2—N1	107.4 (2)	N1—C5—C6	100.05 (19)
C10—C9—C8	120.7 (2)	C4—C5—C6	112.7 (2)
C10—C9—H9A	119.6	N1—C5—H5A	111.0
C8—C9—H9A	119.6	C4—C5—H5A	111.0
C14—N3—H1N3	120 (3)	C6—C5—H5A	111.0
C14—N3—H2N3	118 (3)	C7—C6—C5	101.7 (2)
H1N3—N3—H2N3	119 (4)	C7—C6—H6A	111.4
C3A—C4—C3B	103.6 (13)	C5—C6—H6A	111.4
C3A—C4—C5	128.4 (5)	C7—C6—H6B	111.4
C3B—C4—C5	128.0 (11)	C5—C6—H6B	111.4
C3B—C4—S1B	110.0 (11)	H6A—C6—H6B	109.3
C5—C4—S1B	121.9 (5)	N2—C7—C8	122.0 (2)
C3A—C4—S1A	110.0 (5)	N2—C7—C6	113.8 (2)
C5—C4—S1A	121.56 (18)	C8—C7—C6	124.2 (2)
S1B—C4—S1A	116.4 (5)	C9—C8—C13	119.0 (2)
C1A—S1A—C4	92.7 (2)	C9—C8—C7	119.6 (2)
C2A—C1A—S1A	111.6 (4)	C13—C8—C7	121.4 (2)
C2A—C1A—H1AA	124.2	C11—C10—C9	119.2 (2)
S1A—C1A—H1AA	124.2	C11—C10—H10A	120.4

C1A—C2A—C3A	112.1 (5)	C9—C10—H10A	120.4
C1A—C2A—H2AA	124.0	C10—C11—C12	121.4 (2)
C3A—C2A—H2AA	124.0	C10—C11—Cl1	119.3 (2)
C4—C3A—C2A	113.5 (6)	C12—C11—Cl1	119.3 (2)
C4—C3A—H3AA	123.2	C13—C12—C11	119.1 (2)
C2A—C3A—H3AA	123.2	C13—C12—H12A	120.4
C4—S1B—C1B	93.5 (11)	C11—C12—H12A	120.4
C2B—C1B—S1B	111.0 (19)	C12—C13—C8	120.5 (2)
C2B—C1B—H1BA	124.5	C12—C13—H13A	119.8
S1B—C1B—H1BA	124.5	C8—C13—H13A	119.8
C1B—C2B—C3B	112 (2)	N3—C14—N1	116.4 (2)
C1B—C2B—H2BA	124.0	N3—C14—S2	123.1 (2)
C3B—C2B—H2BA	124.0	N1—C14—S2	120.50 (19)
C4—C3B—C2B	113.3 (17)		
C14—N1—N2—C7	−163.4 (2)	S1B—C4—C5—N1	−89.2 (7)
C5—N1—N2—C7	12.0 (3)	S1A—C4—C5—N1	86.6 (2)
C3A—C4—S1A—C1A	−0.4 (7)	C3A—C4—C5—C6	20.2 (8)
C3B—C4—S1A—C1A	9(13)	C3B—C4—C5—C6	−161.3 (19)
C5—C4—S1A—C1A	−178.3 (4)	S1B—C4—C5—C6	21.9 (7)
S1B—C4—S1A—C1A	−2.2 (7)	S1A—C4—C5—C6	−162.34 (18)
C4—S1A—C1A—C2A	0.7 (8)	N1—C5—C6—C7	19.1 (2)
S1A—C1A—C2A—C3A	−0.8 (14)	C4—C5—C6—C7	−98.4 (2)
C3B—C4—C3A—C2A	−1.1 (19)	N1—N2—C7—C8	179.6 (2)
C5—C4—C3A—C2A	177.7 (7)	N1—N2—C7—C6	2.6 (3)
S1B—C4—C3A—C2A	165 (11)	C5—C6—C7—N2	−14.8 (3)
S1A—C4—C3A—C2A	0.0 (12)	C5—C6—C7—C8	168.2 (2)
C1A—C2A—C3A—C4	0.5 (16)	C10—C9—C8—C13	−0.6 (4)
C3A—C4—S1B—C1B	−17 (10)	C10—C9—C8—C7	179.3 (2)
C3B—C4—S1B—C1B	−3(3)	N2—C7—C8—C9	−170.3 (2)
C5—C4—S1B—C1B	174 (3)	C6—C7—C8—C9	6.4 (4)
S1A—C4—S1B—C1B	−2(3)	N2—C7—C8—C13	9.6 (4)
C4—S1B—C1B—C2B	3(6)	C6—C7—C8—C13	−173.7 (2)
S1B—C1B—C2B—C3B	−3(8)	C8—C9—C10—C11	0.8 (4)
C3A—C4—C3B—C2B	4(4)	C9—C10—C11—C12	−0.3 (4)
C5—C4—C3B—C2B	−175 (3)	C9—C10—C11—Cl1	179.84 (19)
S1B—C4—C3B—C2B	2(4)	C10—C11—C12—C13	−0.4 (4)
S1A—C4—C3B—C2B	−168 (16)	Cl1—C11—C12—C13	179.5 (2)
C1B—C2B—C3B—C4	0(7)	C11—C12—C13—C8	0.5 (4)
C14—N1—C5—C4	−86.0 (3)	C9—C8—C13—C12	−0.1 (4)
N2—N1—C5—C4	98.9 (2)	C7—C8—C13—C12	−179.9 (2)
C14—N1—C5—C6	154.9 (2)	N2—N1—C14—N3	−3.3 (4)
N2—N1—C5—C6	−20.1 (3)	C5—N1—C14—N3	−178.0 (2)
C3A—C4—C5—N1	−90.9 (8)	N2—N1—C14—S2	175.10 (17)
C3B—C4—C5—N1	87.6 (19)	C5—N1—C14—S2	0.4 (3)

Hydrogen-bond geometry (Å, °)

*Cg*1 and *Cg*2 are the centroids of the S1*A*/C1*A*–C3*A*/C4 and S1*B*/C1*B*–C3*B*/C4 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N3—H1N3···N2	0.90 (4)	2.28 (4)	2.656 (3)	105 (3)
N3—H2N3···S2^{i}	0.89 (4)	2.52 (4)	3.400 (3)	170 (3)
C5—H5A···S1A^{ii}	1.00	2.86	3.664 (3)	138
C9—H9A···Cg1^{iii}	0.95	2.79	3.628 (4)	148
C9—H9A···Cg2^{iii}	0.95	2.77	3.595 (18)	145

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*+1; (ii) −*x*+1, −*y*+2, −*z*+2; (iii) *x*−1, *y*, *z*.

Fig. 1

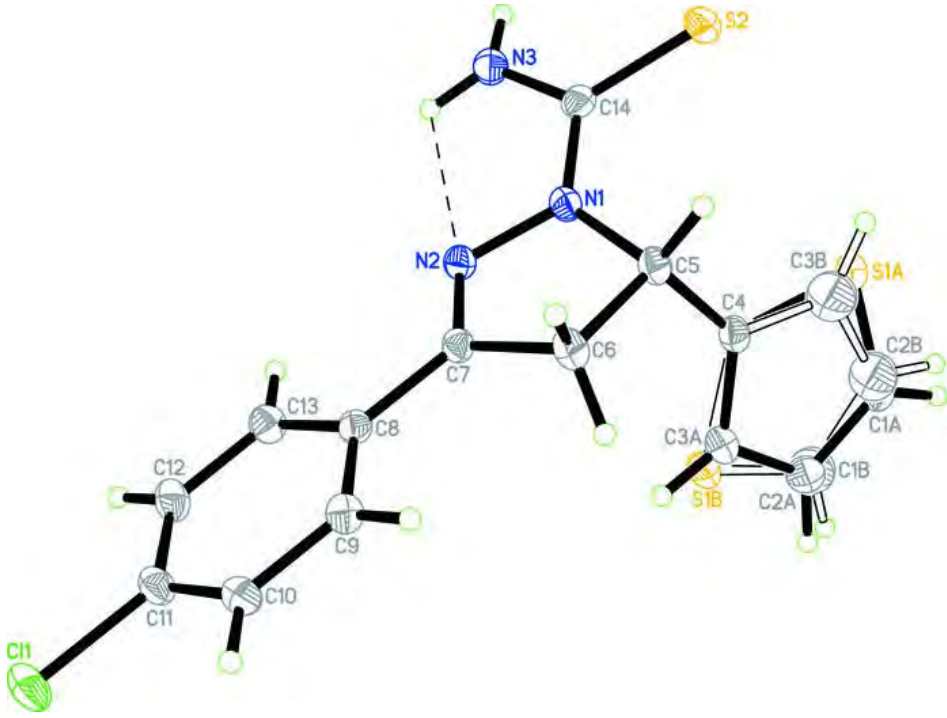
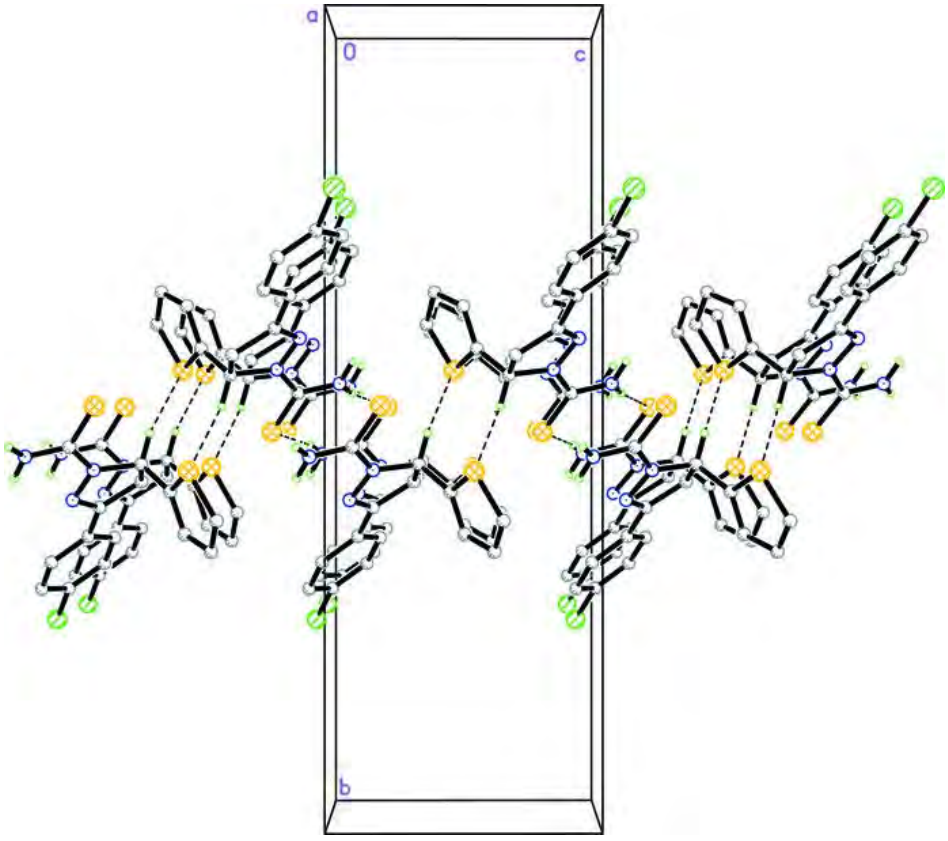


Fig. 2

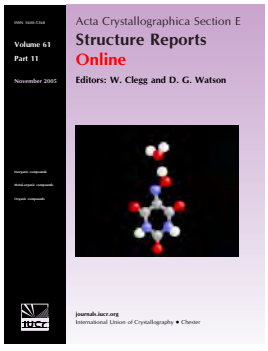


Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, H. Stoeckli-Evans, E. R. T. Tiekink and M. Weil**

(*E*)-1-(Furan-2-yl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

Thitipone Suwunwong, Suchada Chantrapromma, Chatchanok Karalai, Pitikan Wisitsak and Hoong-Kun Fun

Acta Cryst. (2012). E**68**, o317–o318



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(Furan-2-yl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

Thitipone Suwunwong,[a] Suchada Chantrapromma,[a]*‡ Chatchanok Karalai,[a] Pitikan Wisitsak[b] and Hoong-Kun Fun[c]§

[a]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]Excellence Center, Mae Fah Luang University, Thasud, Muang, Chaing Rai 57100, Thailand, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

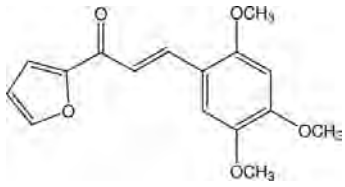
Received 21 December 2011; accepted 1 January 2012

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; R factor = 0.064; wR factor = 0.175; data-to-parameter ratio = 13.7.

In the title chalcone derivative, $C_{16}H_{16}O_5$, the dihedral angle between the furan and benzene rings is 2.06 (17)°. The two methoxy groups at the *ortho* and *para* positions are essentially coplanar with the benzene ring [C—O—C—C angles = −1.0 (5) and 178.5 (3)°], whereas the third one at the *meta* position is slightly twisted [C—O—C—C = 9.6 (5)°]. In the crystal, weak C—H···O interactions link the molecules into a sheet parallel to ($\bar{1}$02). An intermolecular π–π interaction between the furan and benzene rings is present [centroid–centroid distance = 3.772 (2) Å]. A short C···C contact [3.173 (5) Å] is also observed between neighbouring furan rings.

Related literature

For background to and applications of chalcones, see: Cheng *et al.* (2008); Jung *et al.* (2008); Lee *et al.* (2006); Liu *et al.* (2011); Nerya *et al.* (2004); Suwunwong *et al.* (2011); Tewtrakul *et al.* (2003). For related structures, see: Fun *et al.* (2010*a*,*b*, 2011). For the stability of the temperature controller, see: Cosier & Glazer, (1986). For standard bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{16}O_5$
M_r = 288.29
Monoclinic, $P2_1/c$
a = 8.338 (2) Å
b = 8.610 (2) Å
c = 18.923 (5) Å
β = 94.467 (4)°
V = 1354.4 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.28 × 0.21 × 0.09 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.971, T_{max} = 0.991
8001 measured reflections
2647 independent reflections
1663 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.175
S = 1.08
2647 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14B···O2^{i}	0.96	2.47	3.363 (5)	154
C15—H15C···O1^{ii}	0.96	2.43	3.365 (4)	165

Symmetry codes: (i) $x, y+1, z$; (ii) $x-1, -y+\frac{5}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Thailand Research Fund (grant No. RSA5280033) and the Prince of Songkla University for financial support. TS thanks the Graduate School, Prince of Songkla University, for partial financial support. The authors also thank Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5037).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cheng, J.-H., Hung, C.-F., Yang, S.-C., Wang, J.-P., Won, S.-J. & Lin, C.-N. (2008). *Bioorg. Med. Chem.* **16**, 7270–7276.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Fun, H.-K., Chantrapromma, S. & Suwunwong, T. (2011). *Acta Cryst.* E**67**, o2789–o2790.
Fun, H.-K., Suwunwong, T., Chantrapromma, S. & Karalai, C. (2010*a*). *Acta Cryst.* E**66**, o2559–o2560.
Fun, H.-K., Suwunwong, T., Chantrapromma, S. & Karalai, C. (2010*b*). *Acta Cryst.* E**66**, o3070–o3071.
Jung, Y. J., Son, K. I., Oh, Y. E. & Noh, D. Y. (2008). *Polyhedron*, **27**, 861–867.

‡ Thomson Reuters ResearcherID: A-5085-2009.
§ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Lee, S. H., Seo, G. S., Kim, J. Y., Jin, X. Y., Kim, H.-D. & Sohn, D. H. (2006). *Eur. J. Pharmacol.* **532**, 178–186.
Liu, X.-F., Zheng, C.-J., Sun, L.-P., Liu, X.-K. & Piao, H.-R. (2011). *Eur. J. Med. Chem.* **46**, 3469–3473.
Nerya, O., Musa, R., Khatib, S., Tamir, S. & Vaya, J. (2004). *Phytochemistry*, **65**, 1389–1395.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suwunwong, T., Chantrapromma, S. & Fun, H. K. (2011). *Chem. Pap.* **65**, 890–897.
Tewtrakul, S., Subhadhirasakul, S., Puripattanavong, J. & Panphadung, T. (2003). *Songklanakarin J. Sci. Technol.* **25**, 503–508.

supplementary materials

Acta Cryst. (2012). E**68**, o317-o318 [**doi:10.1107/S1600536812000037**]

(*E*)-1-(Furan-2-yl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

T. Suwunwong, S. Chantrapromma, C. Karalai, P. Wisitsak and H.-K. Fun

Comment

Chalcones and heteroaryl chalcones have been reported to possess a wide range of biological activities such as antibacterial (Liu *et al.*, 2011), anti-inflammatory (Lee *et al.*, 2006), anti-oxidant (Cheng *et al.*, 2008), HIV-1 protease inhibitory (Tewtrakul *et al.*, 2003) as well as anti-tyrosinase activities (Nerya *et al.*, 2004), including fluorescent property (Jung *et al.*, 2008; Suwunwong *et al.*, 2011). The title heteroaryl chalcones (I) was synthesized to study for its fluorescence property and tyrosinase inhibitory activity and also to compare its property with the previously published related compounds (Suwunwong *et al.*, 2011). Our experiment shows that (I) exhibits fluorescence property (Suwunwong *et al.*, 2011) and tyrosinase inhibitory activity with the IC_{50} value of 0.143±0.002 $mg.ml^{-1}$. Herein the crystal structure of (I) is reported.

The molecule of (I) in Fig. 1 exists in an *E* configuration with respect to the C6═C7 double bond [1.335 (5) Å]. The molecule is planar with the dihedral angle between the furan and the benzene rings being 2.06 (17)°. The middle prop-2-en-1-one unit (O2/C5–C7) is also planar with the *r.m.s*. 0.0013 (2) and the torsion angle O1–C5–C6–C7 = -0.4 (5)°. The mean plane through this unit makes dihedral angles of 4.1 (2)° and 3.6 (2)° with the furan and the benzene rings, respectively. The two methoxy groups at *ortho* (at atom 9) and *para* (at atom C11) positions of 2,4,5-trimethoxyphenyl unit are essentially coplanar with the attached benzene ring with torsion angles C14–O3–C9–C10 = -1.0 (5)° and C15–O4–C11–C12 = 178.5 (3)°, whereas the third one at *meta* (at atom C12) position is slightly twisted with the torsion angle of C16–O5–C12–C13 = 9.6 (5)°. These angle values also indicated that the methyl group at *para* position points toward the one at *ortho* but point away from the one at *meta* positions due to the sterric effect. The bond distances have normal values (Allen *et al.*, 1987) and are comparable with closely related structures (Fun *et al.*, 2010*a*,*b*, 2011).

In the crystal packing (Fig. 2), weak C14—H14B···$O2^{i}$ and C15—H15C···$O1^{ii}$ interactions (Table 1) link the molecules into sheets parallel to the ($\bar{1}$ 0 2) plane and these sheets are stacked along the *a* axis by π–π interactions with *Cg*1···$Cg2^{iii}$ = 3.772 (2) Å [symmetry code: (iii) 1-x, 2-y, 1-z]; *Cg*1 and *Cg*2 are the centroids of C1–C4/O2 furan and C8–C13 benzene rings, respectively. A C1···$C1^{iv}$[3.173 (5) Å; symmetry code: (iv) 2-x, 1-y, 1-z] short contact is also observed.

Experimental

The title compound was prepared by the condensation of the solution of 2-furyl methylketone (2 mmol, 0.22 g) in ethanol (15 ml) with the solution of 2,4,5-trimethoxybenzaldehyde (2 mmol, 0.40 g) in ethanol (15 ml) in the presence of 20% NaOH (aq) 5 ml at 278 K for 4 hr. The resulting solid which was obtained was collected by filtration, washed with distilled water and dried in air. Yellow slab-shaped single crystals of the title compound suitable for X-ray structure determination were recrystalized from acetone:ethanol (1:1 *v*/*v*) by the slow evaporation of the solvent at room temperature after several days (m.p. 356–357 K).

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with *d*(C—H) = 0.93 Å for aromatic and CH, and 0.96 Å for CH_3 atoms. The U_{iso}(H) values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.83 Å from C10 and the deepest hole is located at 0.89 Å from C4.

Figures

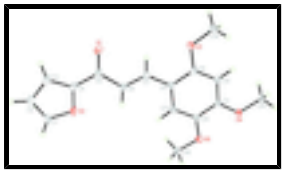


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

Fig. 2. The crystal packing of the title compound viewd along the *b* axis, showing molecular sheets parallel to the ($\bar{1}$ 0 2) plane. Hydrogen bonds are shown as dashed lines.

(*E*)-1-(Furan-2-yl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{16}H_{16}O_5$	$F(000)$ = 608
M_r = 288.29	D_x = 1.414 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point = 356–357 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 8.338 (2) Å	Cell parameters from 2647 reflections
b = 8.610 (2) Å	θ = 2.2–26.0°
c = 18.923 (5) Å	μ = 0.11 mm^{-1}
β = 94.467 (4)°	*T* = 100 K
V = 1354.4 (6) Å^3	Plate, yellow
Z = 4	0.28 × 0.21 × 0.09 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer	2647 independent reflections
Radiation source: sealed tube	1663 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.068
φ and ω scans	θ_{max} = 26.0°, θ_{min} = 2.2°
Absorption correction: multi-scan	*h* = −8→10

(*SADABS*; Bruker, 2009)

T_{min} = 0.971, T_{max} = 0.991

8001 measured reflections

k = −10→7

l = −23→23

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.064

$wR(F^2)$ = 0.175

S = 1.08

2647 reflections

193 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 1.5283P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.33 e Å^{-3}

$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.8947 (3)	1.0580 (3)	0.56583 (11)	0.0221 (6)
O2	0.8198 (3)	0.6581 (3)	0.53014 (11)	0.0219 (6)
O3	0.5846 (3)	1.3849 (3)	0.41496 (11)	0.0234 (6)
O4	0.1930 (3)	1.2112 (3)	0.22782 (11)	0.0203 (6)
O5	0.2463 (3)	0.9265 (3)	0.26329 (11)	0.0211 (6)
C1	0.8927 (5)	0.5329 (4)	0.56403 (17)	0.0249 (9)
H1A	0.8673	0.4297	0.5539	0.030*
C2	1.0063 (4)	0.5797 (4)	0.61407 (17)	0.0244 (9)
H2A	1.0717	0.5167	0.6440	0.029*
C3	1.0061 (4)	0.7441 (4)	0.61196 (17)	0.0218 (8)
H3A	1.0719	0.8098	0.6404	0.026*
C4	0.8920 (4)	0.7884 (4)	0.56070 (15)	0.0177 (7)
C5	0.8371 (4)	0.9414 (4)	0.53558 (15)	0.0181 (8)

C6	0.7141 (4)	0.9497 (4)	0.47546 (15)	0.0177 (8)
H6A	0.6743	0.8590	0.4539	0.021*
C7	0.6597 (4)	1.0876 (4)	0.45188 (16)	0.0174 (8)
H7A	0.7039	1.1740	0.4756	0.021*
C8	0.5399 (4)	1.1191 (4)	0.39374 (15)	0.0157 (7)
C9	0.5031 (4)	1.2735 (4)	0.37474 (16)	0.0172 (8)
C10	0.3898 (4)	1.3086 (4)	0.31901 (15)	0.0162 (7)
H10A	0.3691	1.4115	0.3065	0.019*
C11	0.3084 (4)	1.1903 (4)	0.28248 (15)	0.0145 (7)
C12	0.3391 (4)	1.0334 (4)	0.30161 (15)	0.0152 (7)
C13	0.4544 (4)	1.0014 (4)	0.35535 (15)	0.0155 (7)
H13A	0.4769	0.8982	0.3668	0.019*
C14	0.5532 (5)	1.5429 (4)	0.39800 (19)	0.0317 (10)
H14B	0.6202	1.6080	0.4291	0.048*
H14C	0.4421	1.5656	0.4036	0.048*
H14D	0.5759	1.5621	0.3498	0.048*
C15	0.1539 (4)	1.3692 (4)	0.20725 (16)	0.0190 (8)
H15C	0.0763	1.3687	0.1670	0.029*
H15D	0.2495	1.4216	0.1951	0.029*
H15A	0.1098	1.4221	0.2460	0.029*
C16	0.2548 (5)	0.7704 (4)	0.28869 (17)	0.0234 (8)
H16D	0.1717	0.7097	0.2640	0.035*
H16A	0.2406	0.7694	0.3385	0.035*
H16B	0.3580	0.7272	0.2806	0.035*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0219 (15)	0.0236 (14)	0.0194 (11)	−0.0015 (11)	−0.0062 (10)	−0.0033 (10)
O2	0.0214 (15)	0.0215 (14)	0.0214 (11)	0.0010 (11)	−0.0076 (10)	0.0023 (10)
O3	0.0271 (16)	0.0136 (13)	0.0269 (12)	−0.0014 (11)	−0.0148 (11)	0.0006 (10)
O4	0.0225 (15)	0.0167 (13)	0.0198 (11)	0.0022 (11)	−0.0106 (10)	0.0018 (9)
O5	0.0236 (15)	0.0138 (13)	0.0239 (11)	−0.0038 (11)	−0.0105 (10)	−0.0012 (9)
C1	0.029 (2)	0.0191 (19)	0.0261 (17)	0.0075 (18)	0.0009 (16)	0.0063 (15)
C2	0.020 (2)	0.033 (2)	0.0205 (16)	0.0054 (17)	0.0015 (15)	0.0070 (15)
C3	0.016 (2)	0.030 (2)	0.0187 (15)	−0.0001 (16)	−0.0019 (14)	0.0027 (14)
C4	0.0135 (19)	0.0238 (19)	0.0153 (14)	−0.0026 (16)	−0.0016 (13)	−0.0002 (14)
C5	0.015 (2)	0.024 (2)	0.0155 (15)	0.0011 (16)	−0.0012 (13)	0.0006 (14)
C6	0.014 (2)	0.0200 (19)	0.0183 (15)	−0.0021 (15)	−0.0049 (13)	−0.0002 (13)
C7	0.0129 (19)	0.0218 (19)	0.0174 (15)	−0.0026 (14)	0.0011 (13)	−0.0004 (13)
C8	0.0157 (19)	0.0169 (18)	0.0140 (14)	−0.0004 (15)	−0.0018 (13)	0.0002 (13)
C9	0.0143 (19)	0.0176 (18)	0.0189 (15)	−0.0029 (15)	−0.0032 (14)	−0.0008 (13)
C10	0.0144 (19)	0.0155 (18)	0.0184 (15)	0.0016 (15)	0.0000 (13)	0.0022 (13)
C11	0.0096 (18)	0.0189 (18)	0.0146 (14)	0.0037 (14)	−0.0017 (13)	0.0016 (13)
C12	0.0115 (18)	0.0181 (18)	0.0155 (14)	−0.0002 (15)	−0.0023 (13)	−0.0017 (13)
C13	0.0132 (19)	0.0161 (18)	0.0166 (15)	0.0008 (14)	−0.0035 (13)	0.0006 (12)
C14	0.044 (3)	0.0117 (19)	0.036 (2)	−0.0027 (19)	−0.0158 (18)	0.0021 (16)
C15	0.019 (2)	0.0172 (18)	0.0196 (15)	0.0027 (15)	−0.0046 (14)	0.0063 (13)

C16	0.029 (2)	0.0140 (19)	0.0259 (17)	−0.0021 (16)	−0.0058 (15)	−0.0004 (14)

Geometric parameters (Å, °)

O1—C5	1.234 (4)	C7—C8	1.453 (5)
O2—C1	1.372 (4)	C7—H7A	0.9300
O2—C4	1.379 (4)	C8—C9	1.405 (5)
O3—C9	1.371 (4)	C8—C13	1.408 (4)
O3—C14	1.418 (4)	C9—C10	1.393 (5)
O4—C11	1.369 (4)	C10—C11	1.379 (5)
O4—C15	1.445 (4)	C10—H10A	0.9300
O5—C12	1.373 (4)	C11—C12	1.416 (5)
O5—C16	1.427 (4)	C12—C13	1.372 (4)
C1—C2	1.347 (5)	C13—H13A	0.9300
C1—H1A	0.9300	C14—H14B	0.9600
C2—C3	1.416 (5)	C14—H14C	0.9600
C2—H2A	0.9300	C14—H14D	0.9600
C3—C4	1.359 (5)	C15—H15C	0.9600
C3—H3A	0.9300	C15—H15D	0.9600
C4—C5	1.462 (5)	C15—H15A	0.9600
C5—C6	1.473 (4)	C16—H16D	0.9600
C6—C7	1.335 (5)	C16—H16A	0.9600
C6—H6A	0.9300	C16—H16B	0.9600
C1—O2—C4	106.3 (3)	C11—C10—C9	119.9 (3)
C9—O3—C14	118.1 (3)	C11—C10—H10A	120.1
C11—O4—C15	117.3 (3)	C9—C10—H10A	120.1
C12—O5—C16	116.2 (3)	O4—C11—C10	124.8 (3)
C2—C1—O2	110.8 (3)	O4—C11—C12	114.9 (3)
C2—C1—H1A	124.6	C10—C11—C12	120.2 (3)
O2—C1—H1A	124.6	C13—C12—O5	126.2 (3)
C1—C2—C3	106.3 (3)	C13—C12—C11	118.9 (3)
C1—C2—H2A	126.9	O5—C12—C11	114.9 (3)
C3—C2—H2A	126.9	C12—C13—C8	122.3 (3)
C4—C3—C2	107.4 (3)	C12—C13—H13A	118.8
C4—C3—H3A	126.3	C8—C13—H13A	118.8
C2—C3—H3A	126.3	O3—C14—H14B	109.5
C3—C4—O2	109.2 (3)	O3—C14—H14C	109.5
C3—C4—C5	132.0 (3)	H14B—C14—H14C	109.5
O2—C4—C5	118.8 (3)	O3—C14—H14D	109.5
O1—C5—C4	118.8 (3)	H14B—C14—H14D	109.5
O1—C5—C6	122.7 (3)	H14C—C14—H14D	109.5
C4—C5—C6	118.5 (3)	O4—C15—H15C	109.5
C7—C6—C5	120.0 (3)	O4—C15—H15D	109.5
C7—C6—H6A	120.0	H15C—C15—H15D	109.5
C5—C6—H6A	120.0	O4—C15—H15A	109.5
C6—C7—C8	128.0 (3)	H15C—C15—H15A	109.5
C6—C7—H7A	116.0	H15D—C15—H15A	109.5
C8—C7—H7A	116.0	O5—C16—H16D	109.5
C9—C8—C13	117.2 (3)	O5—C16—H16A	109.5

C9—C8—C7	119.6 (3)	H16D—C16—H16A	109.5
C13—C8—C7	123.1 (3)	O5—C16—H16B	109.5
O3—C9—C10	123.1 (3)	H16D—C16—H16B	109.5
O3—C9—C8	115.5 (3)	H16A—C16—H16B	109.5
C10—C9—C8	121.4 (3)		
C4—O2—C1—C2	0.1 (4)	C7—C8—C9—O3	−1.1 (4)
O2—C1—C2—C3	−0.1 (4)	C13—C8—C9—C10	−1.6 (5)
C1—C2—C3—C4	0.1 (4)	C7—C8—C9—C10	179.5 (3)
C2—C3—C4—O2	0.0 (4)	O3—C9—C10—C11	−177.7 (3)
C2—C3—C4—C5	179.1 (3)	C8—C9—C10—C11	1.6 (5)
C1—O2—C4—C3	0.0 (3)	C15—O4—C11—C10	−0.1 (4)
C1—O2—C4—C5	−179.3 (3)	C15—O4—C11—C12	178.5 (3)
C3—C4—C5—O1	−3.5 (5)	C9—C10—C11—O4	178.8 (3)
O2—C4—C5—O1	175.6 (3)	C9—C10—C11—C12	0.3 (4)
C3—C4—C5—C6	177.0 (3)	C16—O5—C12—C13	9.6 (5)
O2—C4—C5—C6	−3.9 (4)	C16—O5—C12—C11	−170.0 (3)
O1—C5—C6—C7	−0.4 (5)	O4—C11—C12—C13	179.2 (3)
C4—C5—C6—C7	179.1 (3)	C10—C11—C12—C13	−2.1 (4)
C5—C6—C7—C8	179.9 (3)	O4—C11—C12—O5	−1.1 (4)
C6—C7—C8—C9	−177.3 (3)	C10—C11—C12—O5	177.6 (3)
C6—C7—C8—C13	4.0 (5)	O5—C12—C13—C8	−177.5 (3)
C14—O3—C9—C10	−1.0 (5)	C11—C12—C13—C8	2.1 (5)
C14—O3—C9—C8	179.6 (3)	C9—C8—C13—C12	−0.2 (5)
C13—C8—C9—O3	177.7 (3)	C7—C8—C13—C12	178.5 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14B···O2^{i}	0.96	2.47	3.363 (5)	154
C15—H15C···O1^{ii}	0.96	2.43	3.365 (4)	165

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*−1, −*y*+5/2, *z*−1/2.

Fig. 1

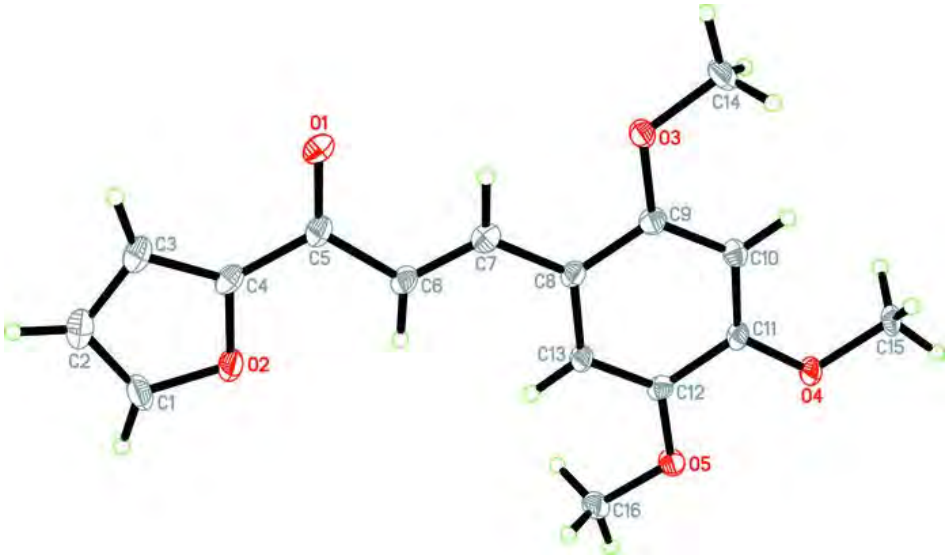
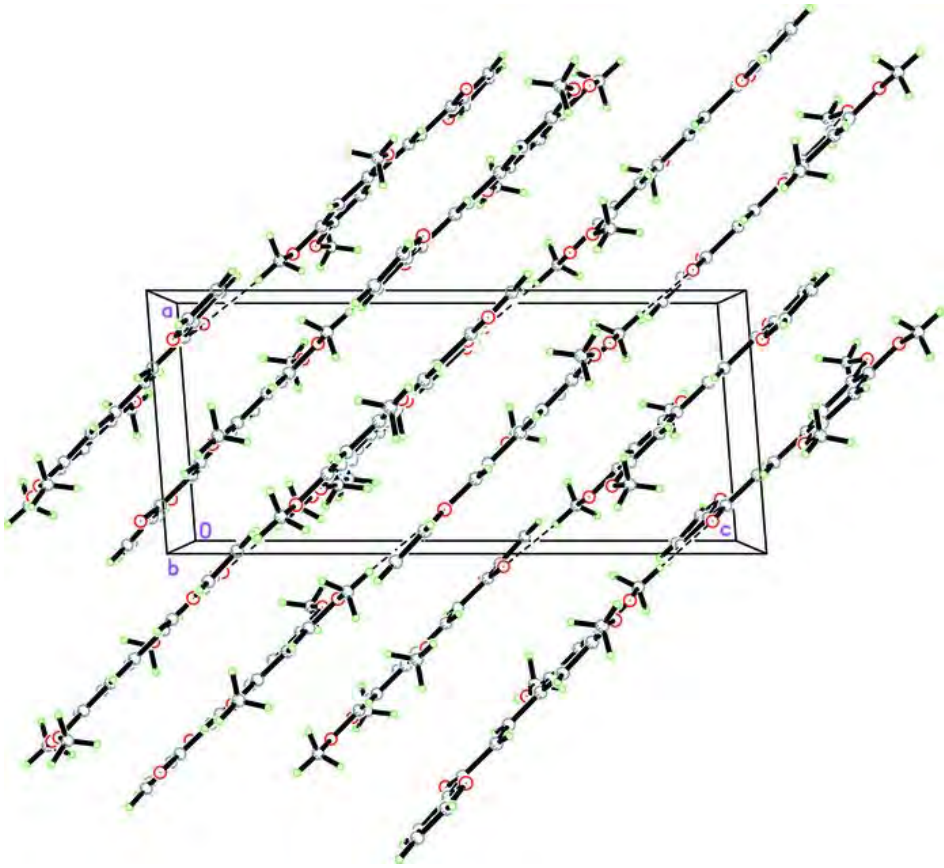


Fig. 2



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368
Editors: **W. T. A. Harrison, H. Stoeckli-Evans, E. R. T. Tiekink and M. Weil**

6-(4-Aminophenyl)-4-(4-ethoxyphenyl)-2-methoxynicotinonitrile

Thitipone Suwunwong, Suchada Chantrapromma and Hoong-Kun Fun

Acta Cryst. (2012). E**68**, o2812–o2813

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-(4-Aminophenyl)-4-(4-ethoxyphenyl)-2-methoxynicotinonitrile

Thitipone Suwunwong,[a] Suchada Chantrapromma[b]*‡ and Hoong-Kun Fun[c]§

[a]Department of Chemistry and Center of Excellence for Innovation in Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: suchada.c@psu.ac.th

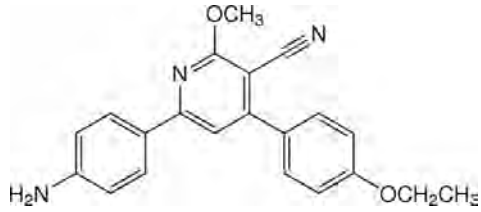
Received 6 August 2012; accepted 20 August 2012

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.052; wR factor = 0.159; data-to-parameter ratio = 21.3.

In the title molecule, $C_{21}H_{19}N_3O_2$, the central pyridine ring makes dihedral angles of 14.46 (9) and 34.67 (8)° with the 4-amino- and 4-ethoxy-substituted benzene rings, respectively. The ethoxy group is essentially coplanar with the attached benzene ring [C—O—C—C torsion angle = 178.70 (16)°] as is the methoxy group with the pyridine ring [C—O—C—N torsion angle = −3.0 (3)°]. In the crystal, molecules are linked by N—H···N hydrogen bonds into chains along [201]. Weak C—H···O hydrogen bonds and C—H···π interactions are also present.

Related literature

The title nicotinonitrile derivative is a cyclized product of a chalcone and a malononitrile in the present of sodium methoxide. For the synthesis and applications of substituted pyridines and nicotinonitrile derivatives, see: Al-Jaber *et al.* (2012); Brandt *et al.* (2010); El-Sayed *et al.* (2011); Goda *et al.* (2004); Ji *et al.* (2007); Kamal *et al.* (2007); Kim *et al.* (2005); Kolev *et al.* (2005); Koner *et al.* (2012); Zhou *et al.* (2006). For a related structure, see: Chantrapromma *et al.* (2010). For standard bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{21}H_{19}N_3O_2$
M_r = 345.39
Monoclinic, $P2_1/c$
a = 5.3924 (2) Å
b = 16.5111 (5) Å
c = 20.1415 (6) Å
β = 91.315 (2)°
V = 1792.82 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 K
0.54 × 0.25 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.956, T_{max} = 0.982
17814 measured reflections
5216 independent reflections
3013 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.159
S = 1.04
5216 reflections
245 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

Cg is the centroid of the C12–C17 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N3—H2*N*3···N2^{i}	0.88 (3)	2.20 (3)	3.084 (3)	177 (2)
C21—H21*A*···O1^{ii}	0.96	2.52	3.439 (2)	160
C18—H18*A*···*Cg*^{iii}	0.96	2.84	3.7135 (18)	151

Symmetry codes: (i) $x+1, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x-1, y, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

TS thanks the Thailand Research Fund through the Royal Golden Jubilee PhD Program and the Center of Excellence for Innovation in Chemistry (PERCH-CIC), Office of the Higher Education, Ministry of Education, Thailand for financial support. The authors thank the Thailand Research Fund (grant No. RSA 5280033), Prince of Songkla University and Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

‡ Thomson Reuters ResearcherID: A-5085-2009.
§ Additional correspondence author, e-mail: hkfun@usm.my. Thomson Reuters ResearcherID: A-3561-2009.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5514).

References

Al-Jaber, N. A., Bougasim, A. S. A. & Karah, M. M. S. (2012). *J. Saudi Chem. Soc*. **16**, 45–53.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Brandt, W., Mologni, L., Preu, L., Lemcke, T., Gambacorti-Passerini, C. & Kunick, C. (2010). *Eur. J. Med. Chem.* **45**, 2919–2927.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chantrapromma, S., Fun, H.-K., Suwunwong, T., Padaki, M. & Isloor, A. M. (2010). *Acta Cryst.* E**66**, o79–o80.

El-Sayed, H. A., Moustafa, A. H., Haikal, A. E.-F. Z., Abu-El-Halawa, R. & Ashry, E. S. H. E. (2011). *Eur. J. Med. Chem.* **46**, 2948–2954.

Goda, F. E., Abdel-Aziz, Alaa A.-M. & Attef, O. A. (2004). *Bioorg. Med. Chem.* **12**, 1845–1852.

Ji, J., Bunnelle, W. H., Anderson, D. J., Faltynek, C., Dyhring, T., Ahring, P. K., Rueter, L. E., Curzon, P., Buckley, M. J., Marsh, K. C., Kempf-Grote, A. & Meyer, M. D. (2007). *Biochem. Pharmacol.* **74**, 1253–1262.

Kamal, A., Khan, M. N. A., Srinivasa, Reddy, K. S. & Rohini, K. (2007). *Bio. Med. Chem*. **15**, 1004–1013.

Kim, K.-R., Rhee, S.-D., Kim, H. Y., Jung, W. H., Yang, S.-D., Kim, S. S., Ahn, J. H. & Cheon, H. G. (2005). *Eur. J. Pharmacol.* **518**, 63–70.

Kolev, T., Stamboliyska, B. & Yancheva, D. (2005). *Chem. Phys.* **324**, 489–496.

Koner, R. R., Sinha, S., Kumar, S., Nandi, C. K. & Ghosh, S. (2012). *Tetrahedron Lett.* **53**, 2302–2307.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Zhou, W.-J., Ji, S.-J. & Shen, Z.-L. (2006). *J. Organomet. Chem.* **691**, 1356–1360.

supplementary materials

Acta Cryst. (2012). E**68**, o2812–o2813 [doi:10.1107/S1600536812036276]

6-(4-Aminophenyl)-4-(4-ethoxyphenyl)-2-methoxynicotinonitrile

Thitipone Suwunwong, Suchada Chantrapromma and Hoong-Kun Fun

Comment

Pyridines have been reported for their applications in a number of areas (Goda *et al.*, 2004; Kamal *et al.*, 2007; Kolev *et al.*, 2005). There are several methods reported for the synthesis of substituted pyridine derivatives including nicotinonitrile derivatives (Al-Jaber *et al.*, 2012; Zhou *et al.*, 2006). Nicotinonitrile derivatives have a wide range of applications such as antitumor, antimicrobial, analgesic, anti-hyperglycemic and antiproliferative activities (Brandt *et al.*, 2010; El-Sayed *et al.*, 2011; Ji *et al.*, 2007; Kim *et al.*, 2005) and fluorescent materials (Koner *et al.*, 2012). Our research is aimed at the synthesis and preliminary fluorescent and antibacterial screening of nicotinonitrile derivatives. The title compound (I) was synthesized by the cyclization of a chalcone derivative with malononitrile to investigate its fluorescent properties. It was found that (I) exhibits fluorescence with the maximum emission at 498 nm when was excited at 370 nm in DMSO.

The molecular structure of the title compound is shown in Fig. 1. The central pyridine ring is inclined to the 4-aminophenyl and 4-ethoxyphenyl rings with the dihedral angles of 14.46 (9) and 34.67 (8)°, respectively. The dihedral angle between these two substituted benzene rings is 44.84 (9)°. The ethoxy substituent of the 4-ethoxyphenyl group is essentially co-planar with the attached benzene ring with the torsion angle C15–O1–C18–C19 = 178.70 (16)° and C18–O1–C15–C16 = 1.8 (3)°. The methoxy group is also approximately co-planar to the pyridine ring as indictated by the torsion angle C21–O2–C11–N1 = -3.0 (3)°. The bond distances agree with the literature values (Allen *et al.*, 1987) and are comparable with those for a related structure (Chantrapromma *et al.*, 2010).

In the crystal (Fig. 2), molecules are linked by N—H···N hydrogen bonds into chains along [201]. Weak C—H···O hydrogen bonds and C—H···π interactions are also present (Table 1).

Experimental

The title compound (I) was synthesized by stirring the solution of (*E*)-1-(4-aminophenyl)-3-(4-ethoxyphenyl)prop-2-en-1-one (0.27 g, 1 mmol) in methanol (10 ml) with a freshly prepared sodium methoxide (1.0 mmol of sodium in 20 ml of methanol). Excess malononitrile (0.13 g, 2.0 mmol) was then added with continuous stirring at room temperature until the precipitate was separated out. The resulting solid was filtered. Pale brown block-shaped single crystals of the title compound suitable for X-ray structure determination were recrystallized from methanol/ethanol (1:1 *v*/*v*) by the slow evaporation of the solvent at room temperature over several days, Mp. 477–478 K.

Refinement

Amino H atoms were located in difference maps and refined isotropically. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C—H) = 0.93 Å for aromatic, 0.97 for CH_2 and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

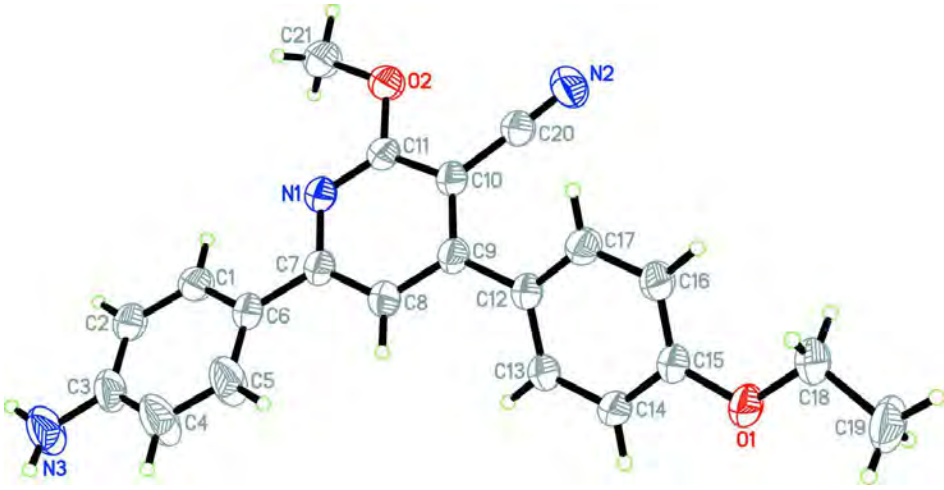


Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

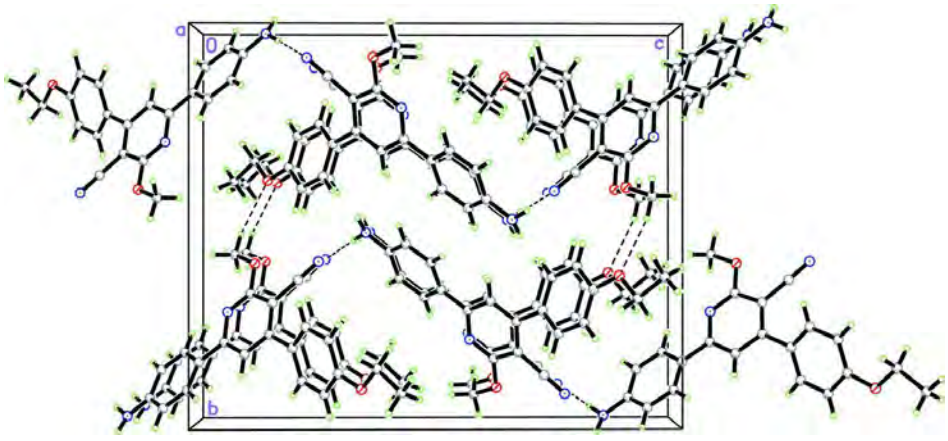


Figure 2

The crystal packing of the title compound viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

6-(4-Aminophenyl)-4-(4-ethoxyphenyl)-2-methoxynicotinonitrile

Crystal data

$C_{21}H_{19}N_3O_2$
$M_r = 345.39$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.3924$ (2) Å
$b = 16.5111$ (5) Å
$c = 20.1415$ (6) Å
$\beta = 91.315$ (2)°
$V = 1792.82$ (10) Å^3
$Z = 4$
$F(000) = 728$
$D_x = 1.280$ Mg m^{-3}
Melting point = 477–478 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5216 reflections
$\theta = 2.0$–30.0°

μ = 0.08 mm^{-1}
T = 298 K

Block, pale-brown
0.54 × 0.25 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.956, T_{max} = 0.982

17814 measured reflections
5216 independent reflections
3013 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 30.0°, θ_{min} = 2.0°
h = −7→7
k = −23→23
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.159
S = 1.04
5216 reflections
245 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2 + 0.3033P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.3134 (2)	0.38320 (7)	0.14470 (6)	0.0637 (3)
O2	1.3927 (3)	0.09389 (7)	0.37553 (7)	0.0737 (4)
N1	1.3663 (3)	0.21531 (8)	0.43089 (6)	0.0543 (3)
N2	1.0455 (4)	0.08668 (10)	0.23879 (9)	0.0858 (6)
N3	1.6313 (5)	0.48378 (15)	0.64781 (10)	0.0987 (7)
C1	1.5687 (4)	0.32043 (10)	0.52550 (9)	0.0654 (5)
H1A	1.6556	0.2743	0.5133	0.078*
C2	1.6575 (4)	0.36652 (11)	0.57804 (9)	0.0675 (5)
H2A	1.8018	0.3506	0.6006	0.081*
C3	1.5363 (4)	0.43554 (12)	0.59760 (9)	0.0663 (5)
C4	1.3212 (5)	0.45547 (16)	0.56331 (12)	0.1042 (9)
H4A	1.2332	0.5012	0.5759	0.125*
C5	1.2329 (4)	0.40959 (14)	0.51089 (11)	0.0874 (7)
H5A	1.0877	0.4254	0.4887	0.105*

C6	1.3538 (3)	0.34094 (10)	0.49045 (8)	0.0514 (4)
C7	1.2622 (3)	0.28980 (9)	0.43520 (8)	0.0501 (4)
C8	1.0805 (3)	0.31513 (10)	0.38963 (8)	0.0540 (4)
H8A	1.0110	0.3663	0.3941	0.065*
C9	1.0004 (3)	0.26572 (9)	0.33750 (7)	0.0485 (4)
C10	1.1104 (3)	0.18863 (9)	0.33383 (8)	0.0509 (4)
C11	1.2909 (3)	0.16807 (9)	0.38214 (8)	0.0537 (4)
C12	0.8139 (3)	0.29470 (9)	0.28753 (7)	0.0484 (4)
C13	0.8087 (3)	0.37611 (10)	0.26827 (8)	0.0547 (4)
H13A	0.9214	0.4122	0.2876	0.066*
C14	0.6399 (3)	0.40372 (10)	0.22128 (8)	0.0564 (4)
H14A	0.6392	0.4581	0.2093	0.068*
C15	0.4699 (3)	0.35068 (10)	0.19151 (8)	0.0511 (4)
C16	0.4713 (3)	0.27011 (10)	0.21019 (8)	0.0544 (4)
H16A	0.3585	0.2341	0.1908	0.065*
C17	0.6410 (3)	0.24309 (10)	0.25784 (8)	0.0534 (4)
H17A	0.6391	0.1889	0.2703	0.064*
C18	0.1324 (3)	0.33169 (12)	0.11351 (9)	0.0643 (5)
H18A	0.0257	0.3082	0.1465	0.077*
H18B	0.2131	0.2881	0.0900	0.077*
C19	−0.0167 (4)	0.38315 (14)	0.06586 (11)	0.0864 (7)
H19A	−0.1479	0.3513	0.0461	0.130*
H19B	0.0887	0.4030	0.0317	0.130*
H19C	−0.0865	0.4281	0.0893	0.130*
C20	1.0686 (3)	0.13264 (10)	0.28075 (9)	0.0608 (5)
C21	1.5900 (4)	0.07236 (12)	0.42137 (11)	0.0818 (7)
H21A	1.6516	0.0196	0.4105	0.123*
H21B	1.5285	0.0719	0.4657	0.123*
H21C	1.7217	0.1112	0.4185	0.123*
H2N3	1.747 (5)	0.4641 (15)	0.6750 (13)	0.101 (8)*
H1N3	1.536 (5)	0.5205 (19)	0.6634 (15)	0.129 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0660 (7)	0.0607 (7)	0.0629 (8)	−0.0029 (6)	−0.0309 (6)	0.0055 (6)
O2	0.0944 (9)	0.0481 (6)	0.0765 (9)	0.0129 (6)	−0.0432 (8)	−0.0048 (6)
N1	0.0654 (8)	0.0489 (7)	0.0478 (7)	−0.0002 (6)	−0.0171 (6)	0.0033 (6)
N2	0.1065 (14)	0.0644 (10)	0.0843 (12)	0.0134 (9)	−0.0443 (11)	−0.0191 (9)
N3	0.1089 (16)	0.1055 (16)	0.0794 (13)	0.0313 (13)	−0.0496 (12)	−0.0399 (12)
C1	0.0775 (12)	0.0515 (9)	0.0655 (11)	0.0122 (8)	−0.0303 (9)	−0.0019 (8)
C2	0.0783 (12)	0.0623 (10)	0.0603 (11)	0.0073 (9)	−0.0347 (9)	0.0019 (8)
C3	0.0720 (11)	0.0775 (12)	0.0485 (10)	0.0090 (9)	−0.0192 (9)	−0.0112 (8)
C4	0.0993 (16)	0.1191 (19)	0.0918 (16)	0.0551 (14)	−0.0506 (13)	−0.0563 (15)
C5	0.0796 (13)	0.1055 (16)	0.0750 (14)	0.0393 (12)	−0.0402 (11)	−0.0366 (12)
C6	0.0576 (9)	0.0548 (9)	0.0411 (8)	0.0016 (7)	−0.0128 (7)	0.0017 (7)
C7	0.0555 (9)	0.0513 (8)	0.0431 (8)	−0.0004 (7)	−0.0106 (7)	0.0030 (6)
C8	0.0626 (10)	0.0513 (8)	0.0472 (9)	0.0068 (7)	−0.0148 (7)	−0.0027 (7)
C9	0.0510 (8)	0.0499 (8)	0.0439 (8)	−0.0027 (7)	−0.0108 (7)	0.0037 (6)
C10	0.0586 (9)	0.0466 (8)	0.0468 (8)	−0.0045 (7)	−0.0156 (7)	0.0023 (6)

C11	0.0651 (10)	0.0436 (8)	0.0516 (9)	0.0004 (7)	−0.0162 (8)	0.0048 (7)
C12	0.0505 (8)	0.0499 (8)	0.0442 (8)	0.0006 (7)	−0.0123 (7)	−0.0004 (6)
C13	0.0628 (10)	0.0474 (8)	0.0530 (9)	−0.0030 (7)	−0.0209 (8)	−0.0033 (7)
C14	0.0675 (10)	0.0444 (8)	0.0562 (10)	0.0000 (7)	−0.0212 (8)	0.0020 (7)
C15	0.0523 (8)	0.0531 (8)	0.0471 (9)	0.0016 (7)	−0.0146 (7)	0.0007 (7)
C16	0.0503 (9)	0.0545 (9)	0.0576 (10)	−0.0071 (7)	−0.0172 (7)	0.0012 (7)
C17	0.0543 (9)	0.0487 (8)	0.0568 (9)	−0.0036 (7)	−0.0122 (7)	0.0068 (7)
C18	0.0598 (10)	0.0711 (11)	0.0608 (11)	−0.0038 (8)	−0.0226 (8)	−0.0032 (8)
C19	0.0809 (14)	0.0989 (16)	0.0775 (14)	−0.0139 (12)	−0.0418 (11)	0.0149 (12)
C20	0.0703 (11)	0.0479 (8)	0.0630 (11)	0.0020 (8)	−0.0256 (9)	−0.0007 (8)
C21	0.0986 (15)	0.0603 (11)	0.0843 (14)	0.0193 (10)	−0.0444 (12)	−0.0005 (10)

Geometric parameters (Å, º)

O1—C15	1.3612 (17)	C9—C10	1.407 (2)
O1—C18	1.4291 (19)	C9—C12	1.485 (2)
O2—C11	1.3502 (19)	C10—C11	1.402 (2)
O2—C21	1.437 (2)	C10—C20	1.427 (2)
N1—C11	1.3114 (19)	C12—C17	1.388 (2)
N1—C7	1.356 (2)	C12—C13	1.399 (2)
N2—C20	1.141 (2)	C13—C14	1.376 (2)
N3—C3	1.377 (2)	C13—H13A	0.9300
N3—H2N3	0.88 (3)	C14—C15	1.393 (2)
N3—H1N3	0.86 (3)	C14—H14A	0.9300
C1—C2	1.380 (2)	C15—C16	1.382 (2)
C1—C6	1.385 (2)	C16—C17	1.385 (2)
C1—H1A	0.9300	C16—H16A	0.9300
C2—C3	1.376 (3)	C17—H17A	0.9300
C2—H2A	0.9300	C18—C19	1.501 (3)
C3—C4	1.376 (3)	C18—H18A	0.9700
C4—C5	1.375 (3)	C18—H18B	0.9700
C4—H4A	0.9300	C19—H19A	0.9600
C5—C6	1.375 (3)	C19—H19B	0.9600
C5—H5A	0.9300	C19—H19C	0.9600
C6—C7	1.473 (2)	C21—H21A	0.9600
C7—C8	1.391 (2)	C21—H21B	0.9600
C8—C9	1.391 (2)	C21—H21C	0.9600
C8—H8A	0.9300		

C15—O1—C18	118.42 (13)	O2—C11—C10	115.38 (14)
C11—O2—C21	117.24 (13)	C17—C12—C13	117.46 (14)
C11—N1—C7	117.77 (13)	C17—C12—C9	122.12 (14)
C3—N3—H2N3	119.3 (17)	C13—C12—C9	120.42 (13)
C3—N3—H1N3	117 (2)	C14—C13—C12	121.20 (14)
H2N3—N3—H1N3	117 (3)	C14—C13—H13A	119.4
C2—C1—C6	121.82 (17)	C12—C13—H13A	119.4
C2—C1—H1A	119.1	C13—C14—C15	120.40 (15)
C6—C1—H1A	119.1	C13—C14—H14A	119.8
C3—C2—C1	121.18 (16)	C15—C14—H14A	119.8
C3—C2—H2A	119.4	O1—C15—C16	124.59 (14)

C1—C2—H2A	119.4	O1—C15—C14	116.19 (14)
C2—C3—C4	117.04 (17)	C16—C15—C14	119.22 (14)
C2—C3—N3	121.22 (18)	C15—C16—C17	119.88 (14)
C4—C3—N3	121.7 (2)	C15—C16—H16A	120.1
C5—C4—C3	121.77 (19)	C17—C16—H16A	120.1
C5—C4—H4A	119.1	C16—C17—C12	121.83 (15)
C3—C4—H4A	119.1	C16—C17—H17A	119.1
C6—C5—C4	121.70 (17)	C12—C17—H17A	119.1
C6—C5—H5A	119.1	O1—C18—C19	107.14 (15)
C4—C5—H5A	119.1	O1—C18—H18A	110.3
C5—C6—C1	116.47 (15)	C19—C18—H18A	110.3
C5—C6—C7	123.06 (15)	O1—C18—H18B	110.3
C1—C6—C7	120.46 (15)	C19—C18—H18B	110.3
N1—C7—C8	121.12 (14)	H18A—C18—H18B	108.5
N1—C7—C6	115.86 (13)	C18—C19—H19A	109.5
C8—C7—C6	123.02 (14)	C18—C19—H19B	109.5
C9—C8—C7	121.57 (15)	H19A—C19—H19B	109.5
C9—C8—H8A	119.2	C18—C19—H19C	109.5
C7—C8—H8A	119.2	H19A—C19—H19C	109.5
C8—C9—C10	116.51 (13)	H19B—C19—H19C	109.5
C8—C9—C12	121.09 (14)	N2—C20—C10	177.0 (2)
C10—C9—C12	122.38 (13)	O2—C21—H21A	109.5
C11—C10—C9	118.00 (14)	O2—C21—H21B	109.5
C11—C10—C20	117.27 (14)	H21A—C21—H21B	109.5
C9—C10—C20	124.46 (13)	O2—C21—H21C	109.5
N1—C11—O2	119.58 (13)	H21A—C21—H21C	109.5
N1—C11—C10	125.03 (15)	H21B—C21—H21C	109.5

C6—C1—C2—C3	−0.5 (3)	C7—N1—C11—C10	0.1 (3)
C1—C2—C3—C4	1.2 (3)	C21—O2—C11—N1	−3.0 (3)
C1—C2—C3—N3	−176.7 (2)	C21—O2—C11—C10	176.03 (17)
C2—C3—C4—C5	−1.3 (4)	C9—C10—C11—N1	0.0 (3)
N3—C3—C4—C5	176.6 (3)	C20—C10—C11—N1	174.15 (17)
C3—C4—C5—C6	0.7 (5)	C9—C10—C11—O2	−179.00 (15)
C4—C5—C6—C1	0.1 (4)	C20—C10—C11—O2	−4.8 (2)
C4—C5—C6—C7	179.0 (2)	C8—C9—C12—C17	−146.49 (17)
C2—C1—C6—C5	−0.2 (3)	C10—C9—C12—C17	35.4 (2)
C2—C1—C6—C7	−179.14 (17)	C8—C9—C12—C13	34.1 (2)
C11—N1—C7—C8	0.3 (2)	C10—C9—C12—C13	−144.01 (17)
C11—N1—C7—C6	−179.60 (15)	C17—C12—C13—C14	−0.5 (3)
C5—C6—C7—N1	−165.10 (19)	C9—C12—C13—C14	178.94 (16)
C1—C6—C7—N1	13.8 (2)	C12—C13—C14—C15	−0.2 (3)
C5—C6—C7—C8	15.0 (3)	C18—O1—C15—C16	1.8 (3)
C1—C6—C7—C8	−166.12 (17)	C18—O1—C15—C14	−178.89 (15)
N1—C7—C8—C9	−0.8 (3)	C13—C14—C15—O1	−178.79 (16)
C6—C7—C8—C9	179.16 (16)	C13—C14—C15—C16	0.6 (3)
C7—C8—C9—C10	0.8 (2)	O1—C15—C16—C17	179.15 (16)
C7—C8—C9—C12	−177.43 (15)	C14—C15—C16—C17	−0.2 (3)
C8—C9—C10—C11	−0.4 (2)	C15—C16—C17—C12	−0.6 (3)

C12—C9—C10—C11	177.79 (15)	C13—C12—C17—C16	1.0 (3)
C8—C9—C10—C20	−174.09 (17)	C9—C12—C17—C16	−178.51 (16)
C12—C9—C10—C20	4.1 (3)	C15—O1—C18—C19	178.70 (16)
C7—N1—C11—O2	178.99 (16)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C12–C17 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H2*N*3···N2^{i}	0.88 (3)	2.20 (3)	3.084 (3)	177 (2)
C21—H21*A*···O1^{ii}	0.96	2.52	3.439 (2)	160
C18—H18*A*···*Cg*^{iii}	0.96	2.84	3.7135 (18)	151

Symmetry codes: (i) *x*+1, −*y*+1/2, *z*+1/2; (ii) −*x*+2, *y*−1/2, −*z*+1/2; (iii) *x*−1, *y*, *z*.