Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*E*)-3-(4-Methoxyphenyl)-1-(3-methyl-2-thienyl)prop-2-en-1-one

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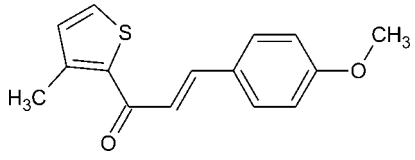
Received 30 October 2007; accepted 9 November 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; R factor = 0.059; wR factor = 0.130; data-to-parameter ratio = 13.6.

The molecule of the title compound, $C_{15}H_{14}O_2S$, is approximately planar, with a dihedral angle of 5.1 (2)° between the two rings. The pattern of bond angles within the benzene ring is influenced by the presence of the substituents; the total effect is close to the sum of the separate effects of both substituents. In the crystal structure, there are stacks of molecules connected by π–π (interplanar distances *ca* 3.3 Å) and C—H$\cdots\pi$ interactions.

Related literature

For related structures, see: Sarojini *et al.* (2007); Harrison, Yathirajan, Ashalatha *et al.* (2006); Harrison, Yathirajan, Anilkumar *et al.* (2006); Fischer *et al.* (2007). For biological applications of chalcones, see *e.g.* Dimmock *et al.* (1999); Won *et al.* (2005); Deng *et al.* (2007); Khatib *et al.* (2005). For the physical properties: see: Fichou *et al.* (1988); Sarojini *et al.* (2006). The influence of the substituents on the benzene ring geometry is summarized by Domenicano (1988).



Experimental

Crystal data

$C_{15}H_{14}O_2S$
M_r = 258.32
Monoclinic, $I2/a$
a = 21.940 (2) Å
b = 5.0674 (4) Å
c = 23.314 (3) Å
β = 96.598 (9)°
V = 2574.9 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 100 (1) K
0.4 × 0.15 × 0.1 mm

Data collection

Kuma KM4 CCD four-circle diffractometer
Absorption correction: none
7918 measured reflections
2241 independent reflections
1568 reflections with $I > 2\sigma(I)$
R_{int} = 0.094

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.130
S = 1.12
2241 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

Cg is the centroid of the benzene ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C341—H34*C*$\cdots Cg^{i}$	0.98	2.63	3.394 (4)	135

Symmetry code: (i) $x, y-1, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1989); software used to prepare material for publication: *SHELXL97*.

BKS thanks the AICTE, Government of India, for the Career Award for Young Teachers Scheme.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2354).

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  doi:10.1107/S1600536807057480

supplementary materials

Acta Cryst. (2007). E**63**, o4688 [**doi:10.1107/S1600536807057480**]

(2*E*)-3-(4-Methoxyphenyl)-1-(3-methyl-2-thienyl)prop-2-en-1-one

B. K. Sarojini, H. S. Yathirajan, B. Narayana, M. T. Swamy and M. Kubicki

Comment

Chalcones represent an important group of natural compounds with a variety of biological activities including antibacterial and antifungal ones (Dimmock *et al.*, 1999). Chalcones have found numerous applications as pesticides, photoprotectors in plastics, solar creams, food additives and are also known for anti-infective and anti-inflammatory activities, cancer chemo-preventive agents (Won *et al.*, 2005), HIV-1 integrase inhibitors (Deng *et al.*, 2007), potent tyrosinase inhibitors (Khatib *et al.*, 2005) and are known for their excellent blue light transmittance and good crystallizability (Fichou *et al.*, 1988, Sarojini *et al.*, 2006). The structures of some related chalcone derivatives *viz*., (2*E*)-1-(3-methyl-2-thienyl)-3- (3-nitrophenyl)prop-2-en-1-one (Sarojini *et al.*, 2007), (2E)-1-(3-bromo-2- thienyl)-3-(4-methoxyphenyl)prop-2-en-1-one (Harrison, Yathirajan, Ashalatha *et al.*, 2006), (2*E*)1-(4-fluorophenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (Harrison, Yathirajan, Anilkumar *et al.*, 2006) and (2E)-3-(biphenyl-4-yl)-1- (4-methoxyphenyl)prop-2-en-1-one (Fischer *et al.*, 2007) have been published. As a continuation of our studies on the structure of chalcones, a new chalcone, (**1**), $C_{15}H_{14}O_2S$ was synthesized and its structure is reported.

The bond angles pattern within the benzene ring is influenced by the presence of substituents; the overall effect is close to the sum of the separate effects of C═CHR and OMe groups (Domenicano, 1988).

The molecule as a whole does not deviate significantly from planarity (Fig. 1); the largest deviation from the least-squares plane calculated through all 18 non-hydrogen atoms is 0.121 (3) Å. The dihedral angles between the planar fragments: the thienyl ring (maximum deviation 0.002 (2) Å), and the benzene ring (0.011 (2) Å) is 5.1 (2)°.

In the crystal structure there are stacks of these planar molecules, related by the unit cell translation along b. The distances between the middlepoint of the C═C bond and the centroids of the thienyl and benzene rings of two neighboring molecules (Fig. 2) are 3.615 (3)Å and 3.559 (3) Å, respectively. Taking into account the offset, the mean interplanar distance is as short as 3.3 Å. These stacks are additionally connected by relatively short C—H···π contact between methoxy group and the centroid (*Cg*) of neighboring benzene ring (Table 1). In the crystal structure there are also short intermolecular S11···O34(1 − *x*,3/2 + *y*,1/2 − *z*) contacts of 3.281 (2) Å.

Experimental

To a thoroughly stirred solution of 1-(3-methyl-2-thienyl)ethanone (1.40 g, 0.01 mol) and 4-methoxybenzaldehyde (1.36 g, 0.01 mol) in 25 ml of ethanol, 5 ml of 40% KOH solution was added, stirred overnight and filtered. The product was crystallized from methanol (m.p.:329–332 K). Analysis for $C_{15}H_{14}O_2S$: Found (Calculated): C: 69.67 (69.74); H: 5.40 (5.46); S: 12.32% (12.41%).(2.03%).

Refinement

The non-standard space group I2/a was chosen because of the large value of the β angle (129.8°) in the standard *C*2/*c* setup. The hydrogen atoms were located in the idealized positions and refined as 'riding model'. Isotropic displacement parameters for hydrogen atoms were set at 1.2 (1.3 for methyl group) times the U_{eq} values of appropriate carrier atoms.

Figures

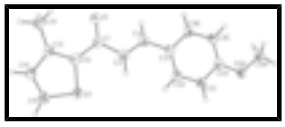

Fig. 1. Anisotropic ellipsoid representation of molecule **1** together with atom labelling scheme. The ellipsoids are drawn at 50% probability level.

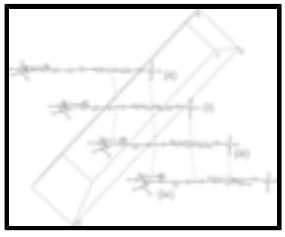

Fig. 2. The crystal packing as seen approximately perpendicular to the molecular plane. Intermolecular contacts are shown as dashed lines. Symmetry codes: (i) *x*,*y*,*z* (ii) *x*,1 + *y*,*z* (iii) *x*,-1 + *y*,*z* (iv) *x*,-2 + *y*,*z*.

(2E)-3-(4-Methoxyphenyl)-1-(3-methyl-2-thienyl)prop-2-en-1-one

Crystal data

$C_{15}H_{14}O_2S$	F_{000} = 1088
M_r = 258.32	D_x = 1.333 Mg m^{-3}
Monoclinic, *I*2/*a*	Melting point: 329-332 K
Hall symbol: -I 2ya	Mo *K*α radiation λ = 0.71073 Å
a = 21.940 (2) Å	Cell parameters from 2730 reflections
b = 5.0674 (4) Å	θ = 1.9–28.3º
c = 23.314 (3) Å	μ = 0.24 mm^{-1}
β = 96.598 (9)º	*T* = 100 (1) K
V = 2574.9 (5) Å^3	Prism, pale-yellow
Z = 8	0.4 × 0.15 × 0.1 mm

Data collection

Kuma KM4 CCD four-circle diffractometer	2241 independent reflections
Radiation source: fine-focus sealed tube	1568 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.094
Detector resolution: 8.1929 pixels mm^{-1}	θ_{max} = 25.0º
T = 100(2) K	θ_{min} = 3.5º
ω scan	*h* = −26→26
Absorption correction: none	*k* = −6→6

7918 measured reflections	$l = -27 \rightarrow 25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.059$	H-atom parameters constrained
$wR(F^2) = 0.130$	$w = 1/[\sigma^2(F_o^2) + (0.02P)^2 + 8.3074P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{max} < 0.001$
2241 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
165 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.59700 (14)	1.1256 (7)	0.09385 (14)	0.0261 (8)
O1	0.59740 (11)	1.1313 (5)	0.04137 (10)	0.0376 (6)
S11	0.62999 (4)	1.32936 (19)	0.20462 (4)	0.0300 (3)
C12	0.63564 (14)	1.3080 (7)	0.13130 (14)	0.0264 (8)
C13	0.67803 (14)	1.4874 (7)	0.11534 (14)	0.0269 (8)
C131	0.69449 (16)	1.5273 (8)	0.05474 (14)	0.0350 (9)
H13A	0.6976	1.3554	0.0360	0.045*
H13B	0.7339	1.6192	0.0563	0.045*
H13C	0.6626	1.6332	0.0326	0.045*
C14	0.70465 (14)	1.6379 (7)	0.16247 (14)	0.0291 (8)
H14	0.7345	1.7711	0.1592	0.035*
C15	0.68333 (15)	1.5737 (7)	0.21304 (15)	0.0318 (8)
H15	0.6967	1.6557	0.2489	0.038*
C2	0.55736 (14)	0.9369 (7)	0.12123 (14)	0.0271 (8)
H2	0.5608	0.9235	0.1621	0.033*
C3	0.51713 (14)	0.7865 (7)	0.08911 (14)	0.0264 (8)
H3	0.5149	0.8120	0.0486	0.032*

C31	0.47591 (14)	0.5869 (7)	0.10832 (13)	0.0252 (7)
C32	0.46711 (14)	0.5482 (7)	0.16659 (14)	0.0273 (8)
H32	0.4889	0.6544	0.1957	0.033*
C33	0.42719 (14)	0.3577 (7)	0.18170 (14)	0.0267 (8)
H33	0.4218	0.3330	0.2212	0.032*
C34	0.39447 (13)	0.1997 (6)	0.13953 (14)	0.0250 (7)
O34	0.35547 (10)	0.0212 (5)	0.16009 (9)	0.0306 (6)
C341	0.32194 (15)	−0.1493 (7)	0.11903 (14)	0.0320 (8)
H34A	0.2945	−0.0439	0.0919	0.042*
H34B	0.2977	−0.2737	0.1392	0.042*
H34C	0.3507	−0.2473	0.0978	0.042*
C35	0.40305 (14)	0.2311 (6)	0.08219 (14)	0.0252 (8)
H35	0.3821	0.1211	0.0533	0.030*
C36	0.44272 (14)	0.4263 (7)	0.06746 (14)	0.0263 (8)
H36	0.4475	0.4515	0.0278	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0246 (17)	0.0240 (18)	0.0300 (19)	0.0045 (16)	0.0048 (13)	0.0014 (16)
O1	0.0448 (14)	0.0402 (15)	0.0288 (14)	−0.0128 (14)	0.0080 (10)	−0.0036 (12)
S11	0.0289 (5)	0.0307 (5)	0.0314 (5)	−0.0044 (4)	0.0073 (3)	−0.0004 (4)
C12	0.0221 (16)	0.0235 (18)	0.0340 (19)	0.0084 (16)	0.0049 (13)	0.0016 (16)
C13	0.0198 (16)	0.0290 (19)	0.0319 (19)	0.0032 (16)	0.0032 (14)	0.0063 (16)
C131	0.0321 (19)	0.041 (2)	0.033 (2)	−0.0075 (19)	0.0062 (15)	0.0045 (18)
C14	0.0228 (16)	0.031 (2)	0.0340 (19)	−0.0012 (17)	0.0041 (14)	0.0007 (17)
C15	0.0289 (18)	0.034 (2)	0.033 (2)	−0.0016 (17)	0.0041 (14)	−0.0034 (17)
C2	0.0270 (17)	0.0267 (18)	0.0279 (18)	0.0037 (16)	0.0041 (14)	0.0004 (16)
C3	0.0257 (17)	0.0247 (18)	0.0294 (18)	0.0073 (16)	0.0065 (14)	0.0000 (15)
C31	0.0216 (16)	0.0237 (18)	0.0300 (18)	0.0058 (15)	0.0022 (13)	−0.0001 (15)
C32	0.0247 (17)	0.0273 (18)	0.0291 (18)	0.0024 (16)	0.0001 (14)	−0.0006 (16)
C33	0.0279 (17)	0.0279 (18)	0.0252 (17)	0.0038 (17)	0.0072 (13)	0.0005 (16)
C34	0.0167 (15)	0.0179 (17)	0.041 (2)	0.0070 (15)	0.0058 (13)	0.0016 (16)
O34	0.0302 (12)	0.0264 (13)	0.0361 (14)	−0.0050 (12)	0.0084 (10)	−0.0021 (11)
C341	0.0275 (18)	0.0271 (19)	0.042 (2)	−0.0020 (17)	0.0057 (15)	−0.0029 (17)
C35	0.0240 (17)	0.0229 (18)	0.0285 (18)	0.0040 (15)	0.0020 (13)	−0.0029 (15)
C36	0.0266 (17)	0.0243 (18)	0.0290 (18)	0.0043 (16)	0.0079 (14)	0.0006 (15)

Geometric parameters (Å, °)

C1—O1	1.225 (4)	C3—H3	0.9500
C1—C12	1.471 (5)	C31—C36	1.393 (4)
C1—C2	1.485 (5)	C31—C32	1.408 (4)
S11—C15	1.700 (4)	C32—C33	1.377 (5)
S11—C12	1.731 (3)	C32—H32	0.9500
C12—C13	1.382 (4)	C33—C34	1.400 (4)
C13—C14	1.408 (5)	C33—H33	0.9500
C13—C131	1.511 (4)	C34—O34	1.369 (4)
C131—H13A	0.9800	C34—C35	1.381 (4)

C131—H13B	0.9800	O34—C341	1.429 (4)
C131—H13C	0.9800	C341—H34A	0.9800
C14—C15	1.357 (4)	C341—H34B	0.9800
C14—H14	0.9500	C341—H34C	0.9800
C15—H15	0.9500	C35—C36	1.386 (4)
C2—C3	1.330 (4)	C35—H35	0.9500
C2—H2	0.9500	C36—H36	0.9500
C3—C31	1.461 (5)		
O1—C1—C12	120.5 (3)	C31—C3—H3	115.9
O1—C1—C2	121.1 (3)	C36—C31—C32	117.4 (3)
C12—C1—C2	118.4 (3)	C36—C31—C3	119.2 (3)
C15—S11—C12	91.89 (16)	C32—C31—C3	123.4 (3)
C13—C12—C1	127.8 (3)	C33—C32—C31	120.4 (3)
C13—C12—S11	110.6 (2)	C33—C32—H32	119.8
C1—C12—S11	121.6 (2)	C31—C32—H32	119.8
C12—C13—C14	112.2 (3)	C32—C33—C34	120.7 (3)
C12—C13—C131	125.5 (3)	C32—C33—H33	119.6
C14—C13—C131	122.3 (3)	C34—C33—H33	119.6
C13—C131—H13A	109.5	O34—C34—C35	125.1 (3)
C13—C131—H13B	109.5	O34—C34—C33	115.0 (3)
H13A—C131—H13B	109.5	C35—C34—C33	119.9 (3)
C13—C131—H13C	109.5	C34—O34—C341	117.3 (2)
H13A—C131—H13C	109.5	O34—C341—H34A	109.5
H13B—C131—H13C	109.5	O34—C341—H34B	109.5
C15—C14—C13	113.2 (3)	H34A—C341—H34B	109.5
C15—C14—H14	123.4	O34—C341—H34C	109.5
C13—C14—H14	123.4	H34A—C341—H34C	109.5
C14—C15—S11	112.1 (3)	H34B—C341—H34C	109.5
C14—C15—H15	124.0	C34—C35—C36	118.8 (3)
S11—C15—H15	124.0	C34—C35—H35	120.6
C3—C2—C1	120.7 (3)	C36—C35—H35	120.6
C3—C2—H2	119.7	C35—C36—C31	122.8 (3)
C1—C2—H2	119.7	C35—C36—H36	118.6
C2—C3—C31	128.2 (3)	C31—C36—H36	118.6
C2—C3—H3	115.9		
O1—C1—C12—C13	−4.4 (5)	C1—C2—C3—C31	178.3 (3)
C2—C1—C12—C13	176.0 (3)	C2—C3—C31—C36	−171.8 (3)
O1—C1—C12—S11	172.9 (3)	C2—C3—C31—C32	8.9 (5)
C2—C1—C12—S11	−6.6 (4)	C36—C31—C32—C33	0.2 (5)
C15—S11—C12—C13	0.1 (3)	C3—C31—C32—C33	179.5 (3)
C15—S11—C12—C1	−177.7 (3)	C31—C32—C33—C34	−0.3 (5)
C1—C12—C13—C14	177.3 (3)	C32—C33—C34—O34	−178.8 (3)
S11—C12—C13—C14	−0.2 (3)	C32—C33—C34—C35	1.3 (5)
C1—C12—C13—C131	−1.7 (5)	C35—C34—O34—C341	1.3 (4)
S11—C12—C13—C131	−179.2 (3)	C33—C34—O34—C341	−178.6 (3)
C12—C13—C14—C15	0.3 (4)	O34—C34—C35—C36	178.0 (3)
C131—C13—C14—C15	179.4 (3)	C33—C34—C35—C36	−2.1 (4)
C13—C14—C15—S11	−0.3 (4)	C34—C35—C36—C31	2.1 (5)

C12—S11—C15—C14	0.1 (3)	C32—C31—C36—C35	−1.1 (5)
O1—C1—C2—C3	−6.3 (5)	C3—C31—C36—C35	179.5 (3)
C12—C1—C2—C3	173.2 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C341—H34C···Cg^{i}	0.98	2.63	3.394 (4)	135

Symmetry codes: (i) *x*, *y*−1, *z*.

Fig. 1

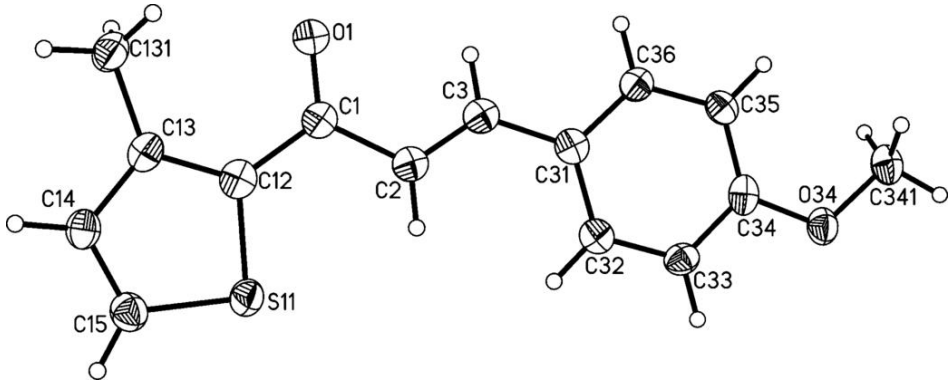
O1
C131
C1
C3
C36
C35
C13
C12
C2
C31
C14
O34
C341
C34
C32
C33
S11
C15

Fig. 2