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Some chalcones derived from thiophene-3-carbaldehyde: synthesis and crystal structures

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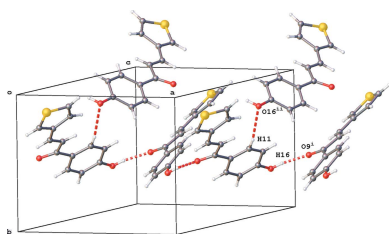
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The synthesis, spectroscopic data and crystal and molecular structures of four 3-(3-phenylprop-1-ene-3-one-1-yl)thiophene derivatives, namely 1-(4-hydroxyphenyl)-3-(thiophen-3-yl)prop-1-en-3-one, C₁₃H₁₀O₂S, (**1**), 1-(4-methoxyphenyl)-3-(thiophen-3-yl)prop-1-en-3-one, C₁₄H₁₂O₂S, (**2**), 1-(4-ethoxyphenyl)-3-(thiophen-3-yl)prop-1-en-3-one, C₁₅H₁₄O₂S, (**3**), and 1-(4-bromophenyl)-3-(thiophen-3-yl)prop-1-en-3-one, C₁₃H₉BrOS, (**4**), are described. The four chalcones have been synthesized by reaction of thiophene-3-carbaldehyde with an acetophenone derivative in an absolute ethanol solution containing potassium hydroxide, and differ in the substituent at the *para* position of the phenyl ring: –OH for **1**, –OCH₃ for **2**, –OCH₂CH₃ for **3** and –Br for **4**. The thiophene ring in **4** was found to be disordered over two orientations with occupancies 0.702 (4) and 0.298 (4). The configuration about the C=C bond is *E*. The thiophene and phenyl rings are inclined by 4.73 (12) for **1**, 12.36 (11) for **2**, 17.44 (11) for **3** and 46.1 (6) and 48.6 (6)° for **4**, indicating that the –OH derivative is almost planar and the –Br derivative deviates the most from planarity. However, the substituent has no real influence on the bond distances in the α,β -unsaturated carbonyl moiety. The molecular packing of **1** features chain formation in the *a*-axis direction by O–H···O contacts. In the case of **2** and **3**, the packing is characterized by dimer formation through C–H···O interactions. In addition, C–H··· π (thiophene) interactions in **2** and C–H···S(thiophene) interactions in **3** contribute to the three-dimensional architecture. The presence of C–H··· π (thiophene) contacts in the crystal of **4** results in chain formation in the *c*-axis direction. The Hirshfeld surface analysis shows that for all four derivatives, the highest contribution to surface contacts arises from contacts in which H atoms are involved.

1. Chemical context

Chalcones, typically referred to as Michael acceptors, can react with nucleophiles at the electrophilic β -position of the unsaturated system (Amslinger, 2010). Many chalcone derivatives containing an α,β -unsaturated carbonyl show potential biological applications such as being effective against amyloid β -induced cytotoxicity (Bukhari *et al.*, 2014) and irreversibly angiotensin-converting enzyme inhibitors (Hea-Young Park Choo *et al.*, 2000).

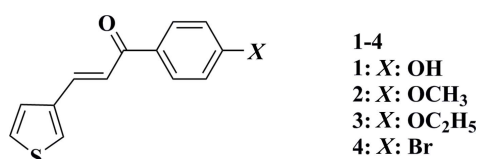
Thiophene, C₄H₄S, belongs to a class of aromatic five-membered heterocycles containing one S heteroatom. Many thiophene derivatives exhibit biological activities: anti-bacterial (Mishra *et al.*, 2012), antiallergic (Gillespie *et al.*,



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1985), analgesic (Laddi *et al.*, 1998), and act as anti-inflammatory agents (Ferreira *et al.*, 2006), antioxidant agents (Jarak *et al.*, 2005) and antitumor agents (Gadad *et al.*, 1994). With the introduction of a thiophene ring into chalcones, it was hoped to design chalcones with interesting new structures and properties. The addition of the thiophene ring to an α,β -unsaturated carbonyl group has also been investigated for a substitution at the C α atom of the thiophene ring (Harrison *et al.*, 2006).

Recently, some thiophene derivatives, such as *N*-(4-oxo-2-sulfanylidene-1,3-thiazolidin-3-yl)-2-(thiophen-3-yl)acetamide (Vu Quoc *et al.*, 2017) and 4-phenyl-3-(thiophen-3-ylmethyl)-1*H*-1,2,4-triazole-5(4*H*)-thione (Vu Quoc *et al.*, 2018), were synthesized by us and their crystal structures were investigated by X-ray diffraction.



In this study, we present the synthesis and crystal structure of four chalcones (**1–4**) containing a thiophene ring: 3-(3-phenylprop-1-ene-3-one-1-yl)thiophene derivatives containing –OH, –OCH₃, –OCH₂CH₃ and –Br at the *para* position of the phenyl ring.

2. Structural commentary

The asymmetric units of **1**, **2**, **3** and **4** are illustrated in Figs. 1, 2, 3 and 4, respectively. The thiophene group in **4** is disordered over two orientations by a rotation of about 180° about the C3–C6 bond in a 0.702 (4): 0.298 (4) ratio. Chalcone **1** bearing the –OH substituent is almost planar, with the dihedral angle between the thiophene and phenyl rings being 4.73 (12)°. For the other chalcones, the deviation from planarity is significant, as illustrated by the dihedral angles: 12.36 (11)° for **2**, 17.44 (11)° for **3** and 46.1 (6)° and 48.6 (6)° for **4**. The C6=C7 bond lengths [1.329 (3) Å for **1**, 1.328 (3) Å for **2**, 1.319 (3) Å for **3** and 1.325 (5) Å for **4**] are almost identical. The configuration of the C6=C7 bond can be described as *E* [torsion angles C3–C6–C7–C8 are –175.4 (2), –177.8 (2),

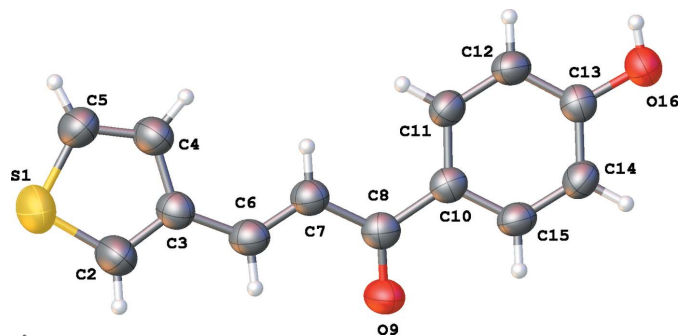


Figure 1
 The molecular structure of **1** showing 50% displacement ellipsoids.

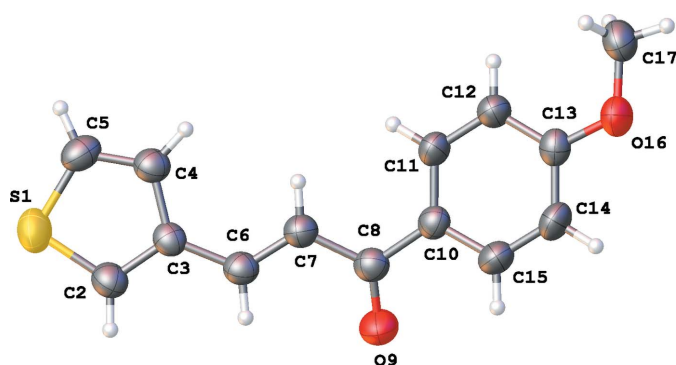


Figure 2
 The molecular structure of **2** showing 50% displacement ellipsoids.

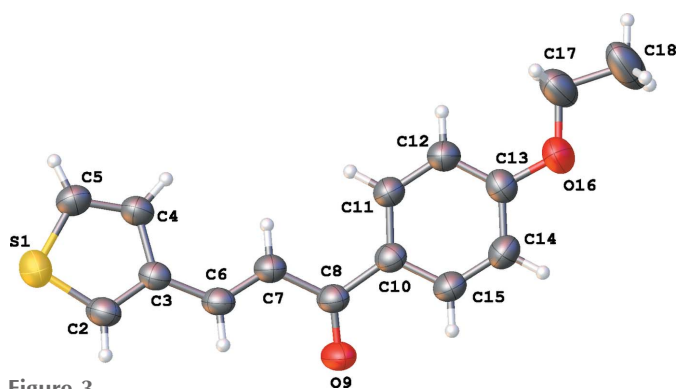


Figure 3
 The molecular structure of **3** showing 50% displacement ellipsoids.

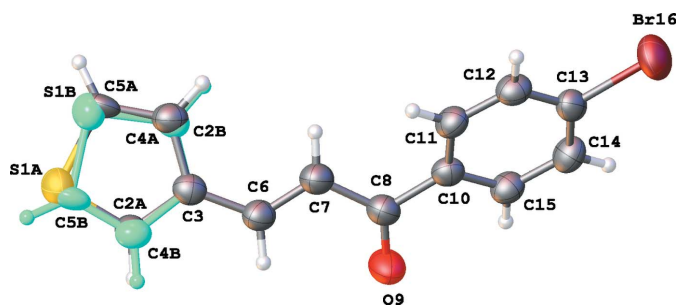


Figure 4
 The molecular structure of **4** showing 50% displacement ellipsoids. The minor-disorder component is shown in light blue.

179.75 (18) and –174.3 (3)° for **1–4**, respectively]. For **1**, this *E* configuration gives rise to an intramolecular C6–H6···O9 interaction (Table 1). The substituent at the *para*-position of the phenyl ring has no significant influence on the C8=O9 bond length [1.232 (3) Å in **1**, 1.228 (3) Å in **2**, 1.224 (2) Å in **3** and 1.224 (4) Å in **4**].

3. Supramolecular features

In chalcone derivative **1**, which crystallizes in the orthorhombic space group *Pbca*, the –OH substituent is involved as donor in intermolecular O16–H16···O9ⁱ [symmetry code: (i) $x + \frac{1}{2}, y, \frac{3}{2} - z$] hydrogen bonding, resulting in the formation of chains of molecules running in the *a*-axis direction (Fig. 5, Table 1). As acceptor, the –OH substituent interacts by

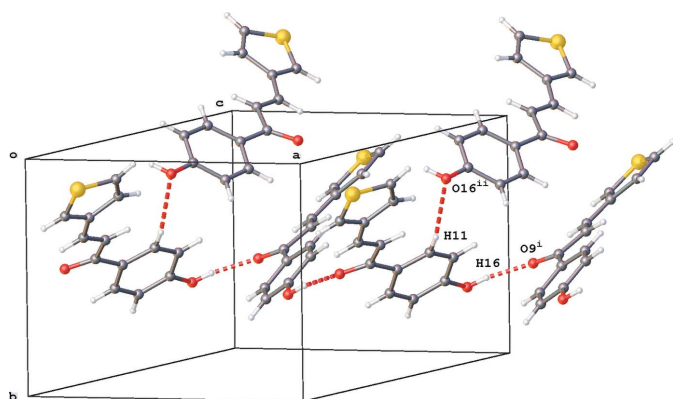


Figure 5
Partial crystal packing of **1** showing the intermolecular hydrogen-bonding interactions as red dashed lines (see Table 1 for details).

intermolecular C11–H11···O16 hydrogen bonding (Fig. 5, Table 1).

Crystals of **2–4** belong to the monoclinic space group $P2_1/c$. The crystal packing of **2** is characterized by inversion-dimer formation between the methoxy groups by weak C17–H17B···O16ⁱ interactions [H17···O16ⁱ = 2.61 Å; symmetry code (i): $-x + 1, -y + 2, -z + 2$] and C–H··· π (thiophene) interactions (C5–H5···Cg1ⁱⁱⁱ and C11–H11···Cg1ⁱⁱⁱ; for details see Table 2 and Fig. 6).

In the packing of **3**, C2–H2···O9ⁱ interactions result in dimeric units forming rings of $R_2^2(14)$ graph-set motif

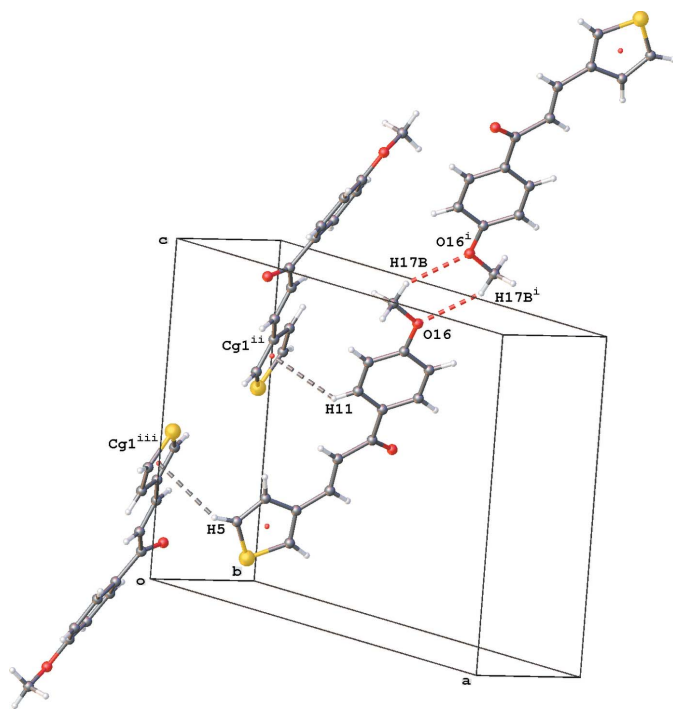


Figure 6
Partial crystal packing of **2** showing dimer formation through C–H···O (red dashed lines) and C–H··· π interactions [grey dashed lines; Cg1 is the centroid of the thiophene ring; symmetry codes: (i) $-x + 1, -y + 2, z + 2$, (ii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$, (iii) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$].

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O16–H16···O9 ⁱ	0.82	1.86	2.667 (2)	167
C6–H6···O9	0.93	2.46	2.785 (3)	100
C11–H11···O16 ⁱⁱ	0.93	2.55	3.425 (3)	157

Symmetry codes: (i) $x + \frac{1}{2}, y, -z + \frac{3}{2}$; (ii) $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$.

Table 2
Hydrogen-bond geometry (Å, °) for **2**.

Cg1 is the centroid of the S1/C2–C5 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5–H5···Cg1 ⁱⁱ	0.93	2.94	3.602 (2)	129
C11–H11···Cg1 ⁱⁱⁱ	0.93	2.99	3.598 (2)	125

Symmetry codes: (ii) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$.

Table 3
Hydrogen-bond geometry (Å, °) for **3**.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2–H2···O9 ⁱ	0.93	2.47	3.324 (2)	153

Symmetry code: (i) $-x + 1, -y + 1, -z + 2$.

Table 4
Hydrogen-bond geometry (Å, °) for **4**.

Cg1 and Cg2 are the centroids of the major- and minor-disorder components of the thiophene ring, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5A–H5A···Cg1 ⁱ	0.93	2.80	3.493 (14)	132
C5A–H5A···Cg2 ⁱ	0.93	2.85	3.52 (2)	130

Symmetry code: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$.

[symmetry code (i): $1 - x, 1 - y, 2 - z$; Table 3, Fig. 7]. In addition, two weaker interactions are present in the packing. Inversion dimers are formed by C14–H14···O16ⁱⁱ interactions [H14···O16ⁱⁱ = 2.71 Å; symmetry code: (ii) $-x + 2, -y + 1, -z + 2$] enclosing an $R_2^2(8)$ ring motif. Chains of molecules running in the a -axis direction are the consequence of C18ⁱⁱⁱ–H18Aⁱⁱⁱ···S1 interactions [H18Aⁱⁱⁱ···S1 = 3.05 Å;

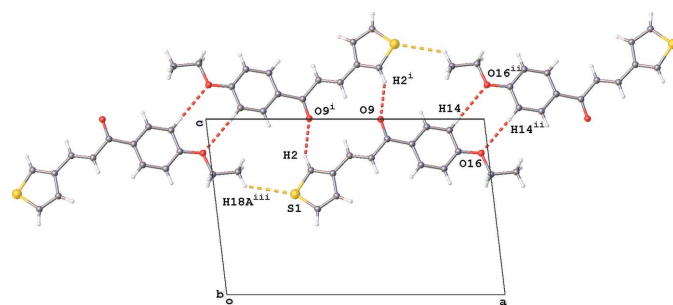
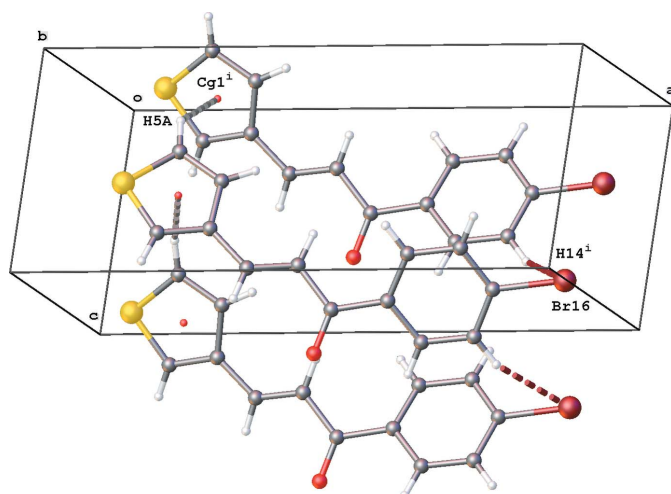


Figure 7
Formation of sheets of molecules of **3** by C–H···O and C–H···S interactions [red dashed lines; symmetry codes: (i) $-x + 1, -y + 1, -z + 2$, (ii) $-x + 2, -y + 1, -z + 2$, (iii) $x - 1, y, z$].


Figure 8

Chains of molecules in **4** running in the *c*-axis direction formed by C5A–H5A···Cg1ⁱ interactions [grey dashed lines, Cg1 is the centroid of the major-disorder component of the thiophene ring; symmetry code: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$].

symmetry code: (iii) $x - 1, y, z$]. These intermolecular interactions result in the formation of sheets of molecules parallel to the *ac* plane (Fig. 7).

In the packing of **4**, chains running in the *c*-axis direction are formed by C5–H5··· π (thiophene) interactions (Table 4, Fig. 8). At the other side of the molecule, the closest contact for the Br16 atom is with H14 [Br16···H14ⁱ = 3.23 Å; Fig. 8]. The shortest Br···Br distance [4.4621 (11) Å] in the crystal packing is Br16···Br16ⁱⁱ [symmetry code: (ii) $-x + 2, -y + 1, -z + 2$].

No voids or π – π stackings are observed in the crystal packing of **1–4**.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.40, update of February 2019; Groom *et al.*, 2016) for 3-(3-thienyl)prop-2-en-1-one gave three hits, *viz.* AYUPIU (Shalini *et al.*, 2011), IBIRUJ (Oyarce *et al.*, 2017) and UNAJIE (Baggio *et al.*, 2016).

The configuration about the double bonds in the symmetrical 1,5-bis(thiophen-3-yl)penta-1,4-dien-3-one (AYUPIU; Shalini *et al.*, 2011) is twice *E*. The dihedral angle between the terminal thiophene rings is 15.45 (10)°. In the crystal packing, C–H···O interactions link the molecules into arrays in the *ac* plane that are further connected by C–H··· π interactions.

Both thiophene rings in 3-hydroxy-1-(thiophen-2-yl)-3-(thiophen-3-yl)prop-2-en-1-one (IBIRUJ; Oyarce *et al.*, 2017) are disordered; the major-disorder components are inclined to each other by 12.1 (3)°. Chains of molecules running in the *c*-axis direction are formed through C–H···O interactions.

In the crystal of 1,3-bis(3-thienyl)prop-2-en-1-one (UNAJIE; Baggio *et al.*, 2016), the stereochemistry about the double bond is *E* and the dihedral angle between the thiophene rings is 8.88 (10)°. Columns of stacking molecules along [010] indicate that π – π interactions play an important role in

Table 5

Percentage contributions of interatomic contacts to the Hirshfeld surfaces for compounds (**1–4**).

Contact	1	2	3	4
H···H	28.8	33.5	44.5	28.5
S···H/H···S	13.0	11.6	10.2	3.9
C···H/H···C	30.4	33.9	22.1	32.6
O···H/H···O	18.5	15.8	13.8	11.1
C···C	3.2	0.7	4.5	2.4
C···S/S···C	3.7	0.9	3.5	0.0
S···S	0.0	1.5	0.0	0.0
S···O/O···S	0.3	0.0	0.1	0.0
C···O/O···C	1.8	1.6	1.0	1.0
O···O	0.0	0.6	0.0	0.0
Br···S/S···Br				2.8
Br···C/C···Br				0.6
Br···H/H···Br				16.5
Br···O/O···Br				0.0
Br···Br				0.5

the crystal packing, together with C–H···O hydrogen bonds between the columns.

A search for 1-phenyl-3-(2-thienyl)prop-2-en-1-one allowing substitution at the phenyl ring resulted in 19 hits of which the compound 1-(4-bromophenyl)-3-(2-thienyl)prop-2-en-1-one (GENXED; Patil *et al.*, 2006; GENXED01; Arshad *et al.*, 2017) is the 2-thienyl derivative of **4**. In addition to similar cell parameters, the thiophene ring also shows rotational disorder [ratio 0.791 (2):0.209 (2) for GENXED; Patil *et al.*, 2006] and the angles between thiophene and phenyl rings are comparable [46.49 (11) and 48.4 (3)° for GENXED; Patil *et al.*, 2006].

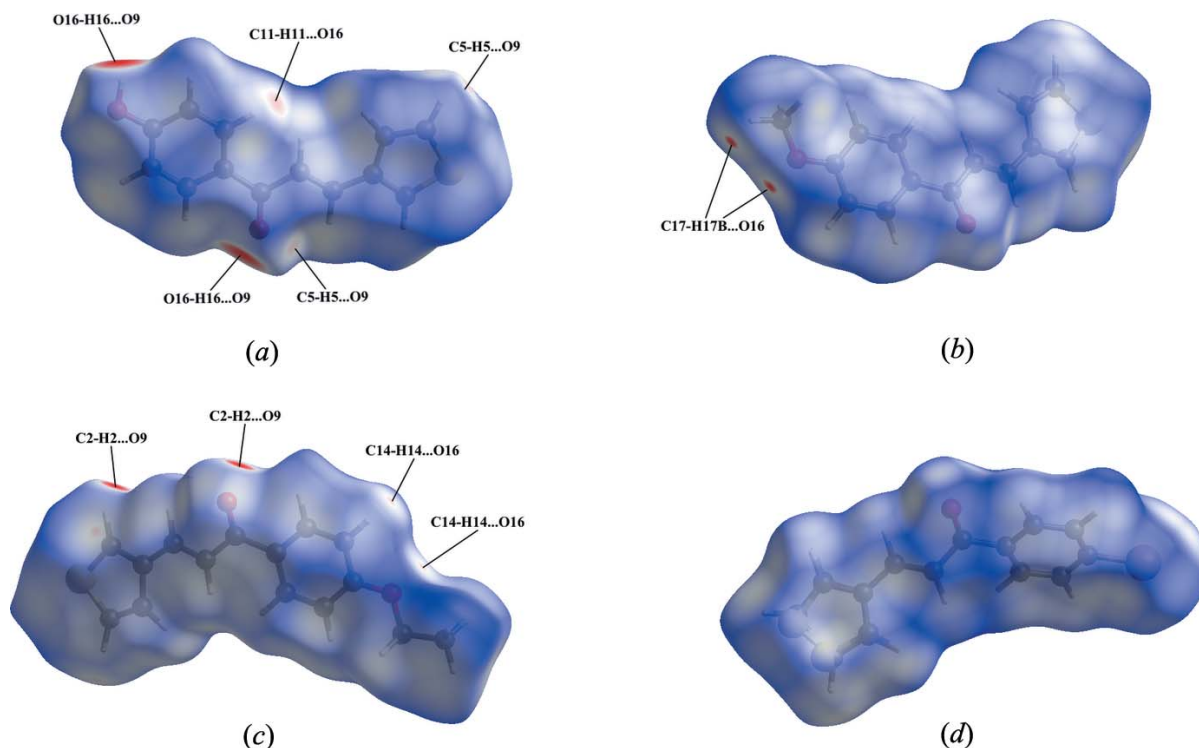
5. Hirshfeld surface analysis

The Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) and the associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) were performed using *CrystalExplorer* (Turner *et al.*, 2017). The Hirshfeld surfaces of compounds **1–4** mapped over d_{norm} are given in Fig. 9. The relative distributions from the different interatomic contacts to the Hirshfeld surfaces are presented in Table 5.

The bright-red spots in Fig. 9*a* near atoms O16 and O9 are indicative for the O16–H16···O9 hydrogen bond in the crystal packing of **1**. The additional faint-red spots illustrate C–H···O interactions. The most significant contributions to the Hirshfeld surface are from C···H/H···C (30.4%), H···H (28.8%) and O···H/H···O (18.5%) contacts (Table 5).

For compound **2**, the donor and acceptor of the relatively weak C17–H17B···O16 interaction are viewed as diminutive red spots near atoms H17B and O16 in Fig. 9*b*. The C–H··· π (thiophene) interactions are indicated by the high contribution from C···H/H···C contacts (33.9%) to the Hirshfeld surface (Table 5).

The bright-red spots in Fig. 9*c* near atoms O9 and H2 of **3** refer to the strong C2–H2···O9 dimer formation, while the faint-red spots near atoms O16 and H14 are indicative for the relatively weak C14–H14···O16 dimer formation. Near atom


Figure 9

The Hirshfeld surface mapped over d_{norm} for (a) compound **1** in the range -0.704 to 1.267 a.u., (b) compound **2** in the range -0.059 to 1.101 a.u., (c) compound **3** in the range -0.200 to 1.439 a.u. and (d) compound **4** in the range 0.007 to 0.942 a.u.

C2 another faint-red spot refers to a contact (2.73 \AA) with atom H5.

The Hirshfeld surface mapped over d_{norm} for **4** (Fig. 9d) shows no short interatomic contacts. Again the C–H $\cdots\pi$ interaction with the disordered thiophene ring is reflected in the high contribution from C \cdots H/H \cdots C contacts (32.6%) to the Hirshfeld surface (Table 5).

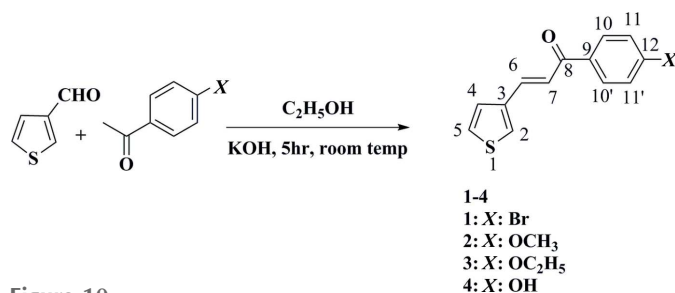
For the four derivatives, the largest contributions of interatomic contacts to the Hirshfeld surface are contacts in which H atoms are involved (Table 5).

6. Synthesis and crystallization

The reaction scheme to synthesize the title compounds **1–4** is given in Fig. 10.

Synthesis of α,β -unsaturated ketone compounds **1–4**:

In a 250 mL beaker, thiophene-3-carbaldehyde (0.1 mole) and substituted acetophenone (0.1 mol) were dissolved in


Figure 10

Reaction scheme for the title compounds **1–4**.

ethanol (100 mL). To this mixture, a 50% KOH (10 mL) solution was added and the mixture was stirred by a magnetic stirrer for 5 h at room temperature until a precipitate appeared. The products **1–4** were obtained as solids, which were filtered under low pressure and recrystallized from ethanol.

Data for 3-(3-(4-hydroxyphenyl)prop-1-ene-3-one-1-yl)-thiophene (**1**):

Yellow crystals; yield 90%; m.p. 388 K; IR (Nicolet Impact 410 FT-IR, KBr, cm^{-1}): 3456.8 (OH), 2983.3 (CH aromatic, alkene), 1643.1 (C=O), 1596.8 (C=C, C=N), 1037.4 [–CH=(*trans*)]; ¹H NMR [Bruker XL-500, 500 MHz, d_6 -CDCl₃, δ (ppm), J (Hz)]: 6.93 (*d*, 2H, $J = 9.0$, H11,11'), 7.34 (*d*, 1H, $J = 15.5$, H7), 7.37 (*d*, 1H, $J = 2$, $J = 3$, H2), 7.41 (*dd*, 1H, $J = 5$, H4), 7.59 (*dd*, 1H, $J = 5.5$, H5), 7.79 (*d*, 1H, $J = 15.5$, H6), 7.98 (*d*, 2H, $J = 8.5$, H10,10'). ¹³C NMR [Bruker XL-500, 125 MHz, d_6 -CDCl₃, δ (ppm)]: 121.63 (C2); 128.79 (C3), 126.98 (C4); 125.26 (C5); 131.39 (C6); 131.07 (C7); 189.0 (C8); 159.84 (C9); 138.33 (C10,10'); 137.64 (C11,11'); 115.4 (C12). Calculation for C₁₃H₁₀O₂S: $M = 230$ au.

Data for 3-(3-(4-methoxyphenyl)prop-1-ene-3-one-1-yl)-thiophene (**2**):

White crystals; yield 70%; m.p. 378 K; IR (Nicolet Impact 410 FT-IR, KBr, cm^{-1}): 3009.3 (CH alkane), 2974.3 (CH aromatic, alkene), 1651.1 (C=O), 1597.5 (C=C, C=N), 1017.2 [–CH=(*trans*)]; ¹H NMR [Bruker XL-500, 500 MHz, d_6 -CDCl₃, δ (ppm), J (Hz)]: 3.89 (*s*, 3H, OCH₃), 6.98 (*d*, 2H, $J = 9.0$, H11,11'), 7.35 (*d*, 1H, $J = 15.5$, H7), 7.36 (*dd*, 1H, $J = 2.5$, $J = 5$, H2), 7.42 (*d*, 1H, $J = 5$, H4), 7.58 (*dd*, 1H, $J = 2.5$, H5), 7.79 (*d*, 1H, $J = 16$, H6), 8.02 (*d*, 2H, $J = 9$, H10,10'). ¹³C NMR

Table 6
Experimental details.

	1	3	2	4
Crystal data				
Chemical formula	C ₁₃ H ₁₀ O ₂ S	C ₁₄ H ₁₂ O ₂ S	C ₁₅ H ₁₄ O ₂ S	C ₁₃ H ₉ BrOS
<i>M_r</i>	230.27	244.30	258.32	293.17
Crystal system, space group	Orthorhombic, <i>Pbca</i>	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>P2₁/c</i>
Temperature (K)	294	294	294	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.0808 (5), 9.0251 (5), 22.8157 (10)	16.4118 (13), 5.8387 (5), 12.6456 (9)	16.5120 (8), 7.7851 (5), 10.4913 (5)	14.1245 (7), 14.2016 (13), 5.8809 (4)
α , β , γ (°)	90, 90, 90	90, 97.279 (7), 90	90, 96.813 (4), 90	90, 98.081 (6), 90
<i>V</i> (Å ³)	2281.69 (19)	1201.98 (16)	1339.11 (13)	1167.93 (15)
<i>Z</i>	8	4	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.26	0.26	0.23	3.67
Crystal size (mm)	0.4 × 0.3 × 0.07	0.45 × 0.3 × 0.15	0.5 × 0.35 × 0.15	0.4 × 0.4 × 0.05
Data collection				
Diffractometer	SuperNova, single source at offset/far, Eos	SuperNova, single source at offset/far, Eos	SuperNova, single source at offset/far, Eos	SuperNova, single source at offset/far, Eos
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)
<i>T_{min}</i> , <i>T_{max}</i>	0.522, 1.000	0.803, 1.000	0.733, 1.000	0.367, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>) reflections]	9745, 2333, 1814	5075, 2457, 1771	13246, 2734, 2162	12050, 2392, 1683
<i>R_{int}</i>	0.019	0.021	0.035	0.045
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.625	0.625	0.625	0.625
Refinement				
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.049, 0.134, 1.05	0.046, 0.118, 1.04	0.047, 0.141, 1.05	0.043, 0.107, 1.02
No. of reflections	2333	2457	2734	2392
No. of parameters	146	156	165	158
No. of restraints	0	0	0	20
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å ⁻³)	0.20, -0.35	0.16, -0.26	0.19, -0.28	0.40, -0.46

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

[Bruker XL-500, 125 MHz, *d*₆-CDCl₃, δ (ppm)]: 121.70 (C2), 128.68 (C3), 126.94 (C4), 125.28 (C5), 131.19 (C6), 130.74 (C7), 188.96 (C8), 163.41 (C9), 138.37 (C10,10'), 137.44 (C11,11'), 113.85 (C12), 55.5 (OCH₃). Calculation for C₁₄H₁₂O₂S: *M* = 244 au.

Data for 3-(3-(4-ethoxyphenyl)prop-1-ene-3-one-1-yl)-thiophene (3):

White crystals; yield 50%; m.p. 380 K; IR (Nicolet Impact 410 FT-IR, KBr, cm⁻¹): 3010.6 (CH alkane), 2983.3 (CH aromatic, alkene), 1657.1 (C=O), 1596.7 (C=C, C=N), 1011.4 [-CH=(*trans*)]; ¹H NMR [Bruker XL-500, 500 MHz, *d*₆-CDCl₃, δ (ppm), *J* (Hz)]: 1.53 (*t*, 3H, *J* = 7, OCH₂CH₃), 4.12 (*q*, 2H, *J* = 7, 7, OCH₂CH₃), 6.96 (*d*, 2H, *J* = 9.0, H11,11'), 7.36 (*d*, 1H, *J* = 15.5, H7), 7.36 (*d*, 1H, *J* = 2, *J* = 3, H2), 7.42 (*dd*, 1H, *J* = 1.5, *J* = 5, H4), 7.58 (*dd*, 1H, *J* = 1.5, *J* = 5.5, H5), 7.78 (*d*, 1H, *J* = 15.5, H6), 8.01 (*d*, 2H, *J* = 9, H10,10'). ¹³C NMR [Bruker XL-500, 125 MHz, *d*₆-CDCl₃, δ (ppm)]: 121.73 (C2); 128.63 (C3), 126.93 (C4); 125.29 (C5); 131.00 (C6); 130.75 (C7); 188.96 (C8); 162.85 (C9); 138.4 (C10,10'); 137.37 (C11,11'); 114.3 (C12); 63.80 (OCH₂CH₃); 14.7 (OCH₂CH₃). Calculation for C₁₅H₁₄O₂S: *M* = 258 au.

Data for 3-(3-(4-bromophenyl)prop-1-ene-3-one-1-yl)-thiophene (4):

Bright-yellow crystals; yield 99%; m.p. 353 K; IR (Nicolet Impact 410 FT-IR, KBr, cm⁻¹): 3090.7 (CH aromatic, alkene),

1654.5 (C=O), 1595.8 (C=C, C=N), 1006.1 [-CH=(*trans*)]; ¹H NMR [Bruker XL-500, 500 MHz, *d*₆-CDCl₃, δ (ppm), *J* (Hz)]: 6.69 (*d*, 2H, *J* = 9.0, H11,11'), 7.35 (*d*, 1H, *J* = 15.5, H7), 7.36 (*dd*, 1H, *J* = 3, *J* = 5.5, H2), 7.41 (*d*, 1H, *J* = 5.5, H4), 7.56 (*dd*, 1H, *J* = 3, H5), 7.77(*d*, 1H, *J* = 15.5, H6), 7.91 (*d*, 2H, *J* = 8.5, H10,10'). ¹³C NMR [Bruker XL-500, 125 MHz, *d*₆-CDCl₃, δ (ppm)]: 121.29 (C2), 127.81 (C3), 127.18 (C4), 125.21 (C5), 129.97 (C6), 131.93 (C7), 189.66 (C8), 138.81 (C9), 138.81 (C10,10'), 138.05(C11,11'), 129.50 (C12). Calculation for C₁₃H₉OSBr: *M* = 293 au.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 6.

All H atoms were placed in idealized positions and refined in riding mode, with *U*_{iso}(H) values assigned as 1.2*U*_{eq} of the parent atoms (1.5 times for methyl groups), with C–H distances of 0.93 (aromatic and =CH), 0.96 (CH₃) and 0.97 Å (CH₂), and O–H distances of 0.82 Å (rotating OH).

In **4**, the thiophene ring was disordered over two positions [population parameters 0.702 (4) and 0.298 (4)] and was refined with restraints for the bond lengths and angles in the ring. The anisotropic temperature factors for atoms S1, C2, C4 and C5 in both orientations were constrained to be equal. In

the final cycles of refinement, two and one outliers were omitted for **1** and **2**, respectively.

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supporting information

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Some chalcones derived from thiophene-3-carbaldehyde: synthesis and crystal structures

Trung Vu Quoc, Duong Tran Thi Thuy, Thuan Dang Thanh, Thanh Phung Ngoc, Vuong Nguyen Thien, Chinh Nguyen Thuy and Luc Van Meervelt

Computing details

For all structures, data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

1-(4-Hydroxyphenyl)-3-(thiophen-3-yl)prop-1-en-3-one (1)

Crystal data

$C_{13}H_{10}O_2S$

$M_r = 230.27$

Orthorhombic, *Pbca*

$a = 11.0808$ (5) Å

$b = 9.0251$ (5) Å

$c = 22.8157$ (10) Å

$V = 2281.69$ (19) Å³

$Z = 8$

$F(000) = 960$

$D_x = 1.341$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3900 reflections

$\theta = 3.0$ – 27.0°

$\mu = 0.26$ mm⁻¹

$T = 294$ K

Block, yellow

$0.4 \times 0.3 \times 0.07$ mm

Data collection

SuperNova, single source at offset/far, Eos diffractometer

Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 15.9631 pixels mm⁻¹

ω scans

Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)

$T_{\min} = 0.522$, $T_{\max} = 1.000$

9745 measured reflections

2333 independent reflections

1814 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -13 \rightarrow 13$

$k = -4 \rightarrow 11$

$l = -27 \rightarrow 28$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.134$

$S = 1.05$

2333 reflections

146 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 1.4972P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.20$ e Å⁻³

$\Delta\rho_{\min} = -0.35$ e Å⁻³

Special details

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.89495 (8)	0.20994 (11)	0.39007 (3)	0.0907 (3)
C2	0.8168 (2)	0.3325 (4)	0.43118 (11)	0.0783 (8)
H2	0.749344	0.383495	0.417667	0.094*
C3	0.8629 (2)	0.3490 (3)	0.48637 (9)	0.0533 (6)
C4	0.9650 (2)	0.2575 (3)	0.49383 (11)	0.0637 (7)
H4	1.008933	0.252750	0.528507	0.076*
C5	0.9932 (2)	0.1760 (3)	0.44462 (11)	0.0704 (7)
H5	1.058032	0.110896	0.441931	0.085*
C6	0.8079 (2)	0.4425 (3)	0.53031 (10)	0.0550 (6)
H6	0.738492	0.493292	0.519204	0.066*
C7	0.8460 (2)	0.4637 (3)	0.58489 (9)	0.0521 (5)
H7	0.918191	0.420470	0.596709	0.062*
C8	0.77790 (19)	0.5528 (2)	0.62690 (9)	0.0486 (5)
O9	0.67534 (14)	0.5953 (2)	0.61457 (7)	0.0621 (5)
C10	0.83162 (18)	0.5889 (2)	0.68496 (9)	0.0458 (5)
C11	0.9339 (2)	0.5188 (3)	0.70693 (10)	0.0511 (5)
H11	0.973899	0.449440	0.683877	0.061*
C12	0.9773 (2)	0.5503 (3)	0.76226 (10)	0.0536 (6)
H12	1.044085	0.499832	0.776762	0.064*
C13	0.92110 (19)	0.6570 (2)	0.79603 (9)	0.0477 (5)
C14	0.8212 (2)	0.7312 (3)	0.77412 (10)	0.0545 (6)
H14	0.784258	0.804822	0.796302	0.065*
C15	0.77688 (19)	0.6960 (3)	0.71980 (10)	0.0518 (5)
H15	0.708722	0.744893	0.705920	0.062*
O16	0.95988 (15)	0.6936 (2)	0.85077 (6)	0.0601 (5)
H16	1.025310	0.654151	0.857006	0.090*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0964 (6)	0.1162 (8)	0.0596 (4)	0.0193 (5)	-0.0060 (4)	-0.0214 (4)
C2	0.0726 (17)	0.104 (2)	0.0583 (15)	0.0212 (17)	-0.0110 (13)	-0.0091 (15)
C3	0.0502 (12)	0.0622 (14)	0.0475 (12)	0.0007 (11)	0.0014 (10)	0.0023 (11)
C4	0.0598 (14)	0.0797 (17)	0.0515 (13)	0.0102 (13)	-0.0034 (11)	0.0004 (12)
C5	0.0670 (15)	0.0795 (19)	0.0647 (16)	0.0183 (15)	0.0016 (13)	-0.0065 (14)
C6	0.0479 (12)	0.0638 (15)	0.0533 (13)	0.0051 (11)	0.0010 (10)	0.0052 (11)
C7	0.0478 (12)	0.0562 (13)	0.0522 (12)	0.0038 (10)	0.0000 (10)	0.0012 (10)
C8	0.0475 (12)	0.0483 (12)	0.0500 (12)	-0.0017 (10)	0.0024 (9)	0.0060 (10)
O9	0.0497 (9)	0.0816 (12)	0.0550 (9)	0.0138 (9)	-0.0052 (7)	-0.0024 (8)

C10	0.0427 (11)	0.0441 (12)	0.0507 (12)	-0.0017 (9)	0.0037 (9)	0.0038 (9)
C11	0.0498 (12)	0.0477 (12)	0.0558 (13)	0.0051 (10)	-0.0003 (10)	-0.0054 (10)
C12	0.0482 (12)	0.0522 (13)	0.0604 (14)	0.0072 (11)	-0.0063 (10)	-0.0014 (11)
C13	0.0466 (11)	0.0492 (12)	0.0474 (11)	-0.0054 (10)	0.0022 (9)	0.0005 (10)
C14	0.0498 (12)	0.0562 (14)	0.0577 (13)	0.0065 (11)	0.0043 (10)	-0.0082 (11)
C15	0.0442 (11)	0.0523 (13)	0.0590 (13)	0.0079 (10)	-0.0022 (10)	0.0005 (11)
O16	0.0580 (10)	0.0712 (11)	0.0510 (9)	0.0040 (9)	-0.0045 (7)	-0.0082 (8)

Geometric parameters (Å, °)

S1—C2	1.689 (3)	C8—C10	1.488 (3)
S1—C5	1.682 (3)	C10—C11	1.392 (3)
C2—H2	0.9300	C10—C15	1.391 (3)
C2—C3	1.367 (3)	C11—H11	0.9300
C3—C4	1.411 (3)	C11—C12	1.380 (3)
C3—C6	1.446 (3)	C12—H12	0.9300
C4—H4	0.9300	C12—C13	1.382 (3)
C4—C5	1.378 (3)	C13—C14	1.387 (3)
C5—H5	0.9300	C13—O16	1.361 (2)
C6—H6	0.9300	C14—H14	0.9300
C6—C7	1.329 (3)	C14—C15	1.370 (3)
C7—H7	0.9300	C15—H15	0.9300
C7—C8	1.461 (3)	O16—H16	0.8200
C8—O9	1.232 (3)		
C5—S1—C2	92.31 (13)	O9—C8—C10	120.3 (2)
S1—C2—H2	123.5	C11—C10—C8	123.2 (2)
C3—C2—S1	113.0 (2)	C15—C10—C8	119.10 (19)
C3—C2—H2	123.5	C15—C10—C11	117.7 (2)
C2—C3—C4	110.3 (2)	C10—C11—H11	119.4
C2—C3—C6	123.0 (2)	C12—C11—C10	121.3 (2)
C4—C3—C6	126.6 (2)	C12—C11—H11	119.4
C3—C4—H4	123.3	C11—C12—H12	120.1
C5—C4—C3	113.3 (2)	C11—C12—C13	119.8 (2)
C5—C4—H4	123.3	C13—C12—H12	120.1
S1—C5—H5	124.5	C12—C13—C14	119.7 (2)
C4—C5—S1	111.0 (2)	O16—C13—C12	122.6 (2)
C4—C5—H5	124.5	O16—C13—C14	117.8 (2)
C3—C6—H6	116.6	C13—C14—H14	120.0
C7—C6—C3	126.9 (2)	C15—C14—C13	120.0 (2)
C7—C6—H6	116.6	C15—C14—H14	120.0
C6—C7—H7	119.0	C10—C15—H15	119.3
C6—C7—C8	122.0 (2)	C14—C15—C10	121.4 (2)
C8—C7—H7	119.0	C14—C15—H15	119.3
C7—C8—C10	119.85 (19)	C13—O16—H16	109.5
O9—C8—C7	119.9 (2)		
S1—C2—C3—C4	0.1 (3)	C7—C8—C10—C15	167.8 (2)

S1—C2—C3—C6	−177.0 (2)	C8—C10—C11—C12	−176.7 (2)
C2—S1—C5—C4	0.5 (3)	C8—C10—C15—C14	178.6 (2)
C2—C3—C4—C5	0.3 (4)	O9—C8—C10—C11	165.4 (2)
C2—C3—C6—C7	179.4 (3)	O9—C8—C10—C15	−13.6 (3)
C3—C4—C5—S1	−0.5 (3)	C10—C11—C12—C13	−2.3 (4)
C3—C6—C7—C8	−175.4 (2)	C11—C10—C15—C14	−0.5 (3)
C4—C3—C6—C7	2.7 (4)	C11—C12—C13—C14	0.3 (3)
C5—S1—C2—C3	−0.3 (3)	C11—C12—C13—O16	−179.7 (2)
C6—C3—C4—C5	177.3 (2)	C12—C13—C14—C15	1.6 (3)
C6—C7—C8—O9	9.1 (4)	C13—C14—C15—C10	−1.5 (4)
C6—C7—C8—C10	−172.3 (2)	C15—C10—C11—C12	2.4 (3)
C7—C8—C10—C11	−13.2 (3)	O16—C13—C14—C15	−178.5 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O16—H16 \cdots O9 ⁱ	0.82	1.86	2.667 (2)	167
C6—H6 \cdots O9	0.93	2.46	2.785 (3)	100
C11—H11 \cdots O16 ⁱⁱ	0.93	2.55	3.425 (3)	157

Symmetry codes: (i) $x+1/2, y, -z+3/2$; (ii) $-x+2, y-1/2, -z+3/2$.

1-(4-Methoxyphenyl)-3-(thiophen-3-yl)prop-1-en-3-one (2)

Crystal data

C₁₄H₁₂O₂S $M_r = 244.30$ Monoclinic, $P2_1/c$ $a = 16.4118$ (13) Å $b = 5.8387$ (5) Å $c = 12.6456$ (9) Å $\beta = 97.279$ (7)° $V = 1201.98$ (16) Å³ $Z = 4$ $F(000) = 512$ $D_x = 1.350$ Mg m^{−3}Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1920 reflections

 $\theta = 3.2$ – 27.3 ° $\mu = 0.26$ mm^{−1} $T = 294$ K

Block, white

 $0.45 \times 0.3 \times 0.15$ mm

Data collection

SuperNova, single source at offset/far, Eos diffractometer

Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 15.9631 pixels mm^{−1} ω scans

Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)

 $T_{\min} = 0.803, T_{\max} = 1.000$

5075 measured reflections

2457 independent reflections

1771 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.021$ $\theta_{\max} = 26.4$ °, $\theta_{\min} = 2.5$ ° $h = -19 \rightarrow 20$ $k = -4 \rightarrow 7$ $l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.046$ $wR(F^2) = 0.118$ $S = 1.04$

2457 reflections

156 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.3903P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$$

Extinction correction: SHELXL-2016/4

(Sheldrick 2015),

$$F_c^* = kFc[1 + 0.001x\lambda^3/\sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.0141 (16)

Special details

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.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.04793 (4)	0.76040 (12)	0.07886 (5)	0.0654 (3)
C2	0.10560 (14)	0.9740 (4)	0.14100 (16)	0.0519 (6)
H2	0.116506	1.111424	0.108336	0.062*
C3	0.13426 (12)	0.9216 (4)	0.24447 (15)	0.0421 (5)
C4	0.10869 (13)	0.6985 (4)	0.27105 (18)	0.0494 (6)
H4	0.122905	0.631591	0.337572	0.059*
C5	0.06133 (14)	0.5929 (4)	0.18926 (18)	0.0558 (6)
H5	0.039091	0.447129	0.193370	0.067*
C6	0.18106 (12)	1.0798 (4)	0.31699 (16)	0.0463 (5)
H6	0.191362	1.224036	0.290462	0.056*
C7	0.21052 (13)	1.0373 (4)	0.41779 (16)	0.0477 (5)
H7	0.202752	0.892707	0.445659	0.057*
C8	0.25502 (13)	1.2111 (4)	0.48691 (17)	0.0468 (5)
O9	0.25416 (11)	1.4134 (3)	0.46020 (13)	0.0657 (5)
C10	0.30162 (12)	1.1378 (4)	0.58960 (15)	0.0430 (5)
C11	0.28855 (13)	0.9319 (4)	0.63854 (16)	0.0499 (6)
H11	0.249747	0.830772	0.605098	0.060*
C12	0.33136 (13)	0.8718 (4)	0.73559 (16)	0.0522 (6)
H12	0.320424	0.733792	0.767704	0.063*
C13	0.39048 (13)	1.0180 (4)	0.78457 (16)	0.0467 (5)
C14	0.40604 (15)	1.2229 (4)	0.73538 (19)	0.0588 (7)
H14	0.446764	1.320516	0.767118	0.071*
C15	0.36170 (15)	1.2817 (4)	0.64040 (18)	0.0565 (6)
H15	0.371979	1.421195	0.609103	0.068*
O16	0.43757 (9)	0.9765 (3)	0.87912 (11)	0.0586 (5)
C17	0.42397 (17)	0.7696 (5)	0.93373 (19)	0.0679 (7)
H17A	0.367631	0.762376	0.946635	0.102*
H17B	0.459086	0.765633	1.000460	0.102*
H17C	0.436021	0.641159	0.890914	0.102*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0659 (4)	0.0704 (5)	0.0552 (4)	0.0009 (3)	-0.0104 (3)	-0.0052 (3)

C2	0.0571 (13)	0.0487 (14)	0.0489 (12)	0.0044 (11)	0.0019 (10)	0.0049 (11)
C3	0.0414 (11)	0.0426 (13)	0.0418 (11)	0.0067 (9)	0.0036 (9)	0.0015 (10)
C4	0.0497 (12)	0.0462 (14)	0.0515 (12)	0.0020 (10)	0.0033 (10)	0.0054 (11)
C5	0.0510 (13)	0.0420 (13)	0.0730 (15)	-0.0027 (11)	0.0029 (11)	-0.0003 (12)
C6	0.0475 (12)	0.0419 (13)	0.0489 (12)	0.0019 (10)	0.0042 (10)	0.0038 (10)
C7	0.0507 (12)	0.0444 (13)	0.0470 (12)	-0.0033 (10)	0.0027 (10)	0.0024 (11)
C8	0.0487 (12)	0.0454 (14)	0.0469 (12)	-0.0009 (10)	0.0086 (9)	0.0006 (11)
O9	0.0854 (13)	0.0450 (10)	0.0631 (10)	-0.0029 (9)	-0.0039 (9)	0.0039 (9)
C10	0.0458 (11)	0.0421 (12)	0.0415 (11)	-0.0038 (10)	0.0072 (9)	-0.0039 (10)
C11	0.0523 (13)	0.0500 (14)	0.0455 (12)	-0.0152 (11)	-0.0013 (10)	-0.0011 (11)
C12	0.0595 (14)	0.0490 (14)	0.0465 (12)	-0.0131 (11)	0.0007 (10)	0.0038 (11)
C13	0.0449 (12)	0.0523 (14)	0.0420 (11)	-0.0022 (10)	0.0026 (9)	-0.0064 (11)
C14	0.0611 (14)	0.0541 (15)	0.0578 (14)	-0.0200 (12)	-0.0058 (11)	-0.0068 (12)
C15	0.0686 (15)	0.0452 (14)	0.0544 (13)	-0.0158 (12)	0.0027 (11)	0.0005 (11)
O16	0.0583 (10)	0.0640 (11)	0.0495 (9)	-0.0088 (8)	-0.0083 (7)	-0.0020 (8)
C17	0.0807 (18)	0.0666 (18)	0.0513 (14)	-0.0019 (14)	-0.0108 (12)	0.0017 (13)

Geometric parameters (Å, °)

S1—C2	1.697 (2)	C10—C11	1.381 (3)
S1—C5	1.696 (2)	C10—C15	1.390 (3)
C2—H2	0.9300	C11—H11	0.9300
C2—C3	1.368 (3)	C11—C12	1.380 (3)
C3—C4	1.422 (3)	C12—H12	0.9300
C3—C6	1.451 (3)	C12—C13	1.379 (3)
C4—H4	0.9300	C13—C14	1.387 (3)
C4—C5	1.360 (3)	C13—O16	1.361 (2)
C5—H5	0.9300	C14—H14	0.9300
C6—H6	0.9300	C14—C15	1.367 (3)
C6—C7	1.328 (3)	C15—H15	0.9300
C7—H7	0.9300	O16—C17	1.423 (3)
C7—C8	1.471 (3)	C17—H17A	0.9600
C8—O9	1.228 (3)	C17—H17B	0.9600
C8—C10	1.484 (3)	C17—H17C	0.9600
C5—S1—C2	92.07 (11)	C15—C10—C8	119.1 (2)
S1—C2—H2	123.8	C10—C11—H11	119.0
C3—C2—S1	112.49 (18)	C12—C11—C10	122.0 (2)
C3—C2—H2	123.8	C12—C11—H11	119.0
C2—C3—C4	110.8 (2)	C11—C12—H12	120.3
C2—C3—C6	123.5 (2)	C13—C12—C11	119.5 (2)
C4—C3—C6	125.60 (19)	C13—C12—H12	120.3
C3—C4—H4	123.4	C12—C13—C14	119.44 (19)
C5—C4—C3	113.1 (2)	O16—C13—C12	125.0 (2)
C5—C4—H4	123.4	O16—C13—C14	115.54 (19)
S1—C5—H5	124.3	C13—C14—H14	119.9
C4—C5—S1	111.50 (18)	C15—C14—C13	120.2 (2)
C4—C5—H5	124.3	C15—C14—H14	119.9

C3—C6—H6	117.0	C10—C15—H15	119.3
C7—C6—C3	125.9 (2)	C14—C15—C10	121.4 (2)
C7—C6—H6	117.0	C14—C15—H15	119.3
C6—C7—H7	118.8	C13—O16—C17	118.08 (18)
C6—C7—C8	122.3 (2)	O16—C17—H17A	109.5
C8—C7—H7	118.8	O16—C17—H17B	109.5
C7—C8—C10	118.9 (2)	O16—C17—H17C	109.5
O9—C8—C7	120.8 (2)	H17A—C17—H17B	109.5
O9—C8—C10	120.3 (2)	H17A—C17—H17C	109.5
C11—C10—C8	123.42 (19)	H17B—C17—H17C	109.5
C11—C10—C15	117.43 (19)		
S1—C2—C3—C4	1.0 (2)	C8—C10—C11—C12	-178.1 (2)
S1—C2—C3—C6	-176.32 (16)	C8—C10—C15—C14	179.6 (2)
C2—S1—C5—C4	-0.02 (19)	O9—C8—C10—C11	162.0 (2)
C2—C3—C4—C5	-1.1 (3)	O9—C8—C10—C15	-17.9 (3)
C2—C3—C6—C7	-179.7 (2)	C10—C11—C12—C13	-1.6 (4)
C3—C4—C5—S1	0.6 (3)	C11—C10—C15—C14	-0.3 (4)
C3—C6—C7—C8	-177.8 (2)	C11—C12—C13—C14	-0.1 (3)
C4—C3—C6—C7	3.3 (4)	C11—C12—C13—O16	-178.9 (2)
C5—S1—C2—C3	-0.60 (18)	C12—C13—C14—C15	1.6 (4)
C6—C3—C4—C5	176.2 (2)	C12—C13—O16—C17	-1.6 (3)
C6—C7—C8—O9	13.0 (3)	C13—C14—C15—C10	-1.4 (4)
C6—C7—C8—C10	-166.6 (2)	C14—C13—O16—C17	179.5 (2)
C7—C8—C10—C11	-18.4 (3)	C15—C10—C11—C12	1.8 (3)
C7—C8—C10—C15	161.7 (2)	O16—C13—C14—C15	-179.5 (2)

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the S1/C2—C5 ring.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C5—H5...Cg1 ⁱ	0.93	2.94	3.602 (2)	129
C11—H11...Cg1 ⁱⁱ	0.93	2.99	3.598 (2)	125

Symmetry codes: (i) $-x, y-1/2, -z+1/2$; (ii) $x, -y+3/2, z+1/2$.**1-(4-Ethoxyphenyl)-3-(thiophen-3-yl)prop-1-en-3-one (3)***Crystal data*

C₁₅H₁₄O₂S
M_r = 258.32
 Monoclinic, *P*2₁/*c*
a = 16.5120 (8) Å
b = 7.7851 (5) Å
c = 10.4913 (5) Å
 β = 96.813 (4)°
V = 1339.11 (13) Å³
Z = 4

F(000) = 544
D_x = 1.281 Mg m⁻³
 Mo *K*α radiation, λ = 0.71073 Å
 Cell parameters from 4827 reflections
 θ = 3.3–27.9°
 μ = 0.23 mm⁻¹
T = 294 K
 Block, white
 0.5 × 0.35 × 0.15 mm

Data collection

SuperNova, single source at offset/far, Eos diffractometer

Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 15.9631 pixels mm⁻¹

ω scans

Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)

$T_{\min} = 0.733$, $T_{\max} = 1.000$

13246 measured reflections

2734 independent reflections

2162 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.5^\circ$

$h = -20 \rightarrow 20$

$k = -9 \rightarrow 9$

$l = -12 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.141$

$S = 1.05$

2734 reflections

165 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.4069P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$

Extinction correction: SHELXL-2016/4 (Sheldrick 2015),

$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0073 (17)

Special details

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.28921 (3)	0.41932 (9)	0.57261 (6)	0.0697 (3)
C2	0.35583 (12)	0.4427 (3)	0.70816 (19)	0.0558 (5)
H2	0.340852	0.481608	0.785855	0.067*
C3	0.43322 (11)	0.3985 (2)	0.69052 (17)	0.0458 (4)
C4	0.43736 (12)	0.3435 (3)	0.56166 (18)	0.0551 (5)
H4	0.485324	0.307932	0.531318	0.066*
C5	0.36332 (13)	0.3486 (3)	0.4872 (2)	0.0588 (5)
H5	0.354921	0.317238	0.401153	0.071*
C6	0.50131 (12)	0.4082 (2)	0.79107 (18)	0.0483 (5)
H6	0.489859	0.443268	0.871763	0.058*
C7	0.57803 (12)	0.3718 (3)	0.77867 (18)	0.0518 (5)
H7	0.591201	0.335993	0.699110	0.062*
C8	0.64340 (12)	0.3860 (3)	0.88615 (17)	0.0490 (5)
O9	0.62809 (9)	0.3934 (2)	0.99739 (13)	0.0670 (5)
C10	0.72986 (12)	0.3954 (2)	0.85742 (17)	0.0476 (5)
C11	0.75356 (12)	0.3597 (3)	0.73833 (18)	0.0540 (5)
H11	0.714251	0.329023	0.671266	0.065*
C12	0.83455 (13)	0.3684 (3)	0.7166 (2)	0.0594 (5)
H12	0.849281	0.344313	0.635592	0.071*

C13	0.89322 (13)	0.4129 (3)	0.8157 (2)	0.0568 (5)
C14	0.87043 (13)	0.4512 (3)	0.9360 (2)	0.0618 (6)
H14	0.909675	0.483056	1.002823	0.074*
C15	0.79065 (13)	0.4420 (3)	0.95555 (19)	0.0571 (5)
H15	0.776104	0.467265	1.036427	0.069*
O16	0.97471 (9)	0.4227 (2)	0.80525 (16)	0.0762 (5)
C17	1.00141 (16)	0.3853 (4)	0.6849 (3)	0.0901 (9)
H17A	0.978364	0.467414	0.621098	0.108*
H17B	0.983604	0.271209	0.656990	0.108*
C18	1.09277 (17)	0.3952 (5)	0.6991 (4)	0.1138 (13)
H18A	1.111988	0.364258	0.619307	0.171*
H18B	1.115025	0.317438	0.765128	0.171*
H18C	1.109771	0.510223	0.721773	0.171*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0502 (4)	0.0898 (5)	0.0684 (4)	0.0040 (3)	0.0052 (3)	0.0000 (3)
C2	0.0549 (12)	0.0668 (13)	0.0479 (11)	0.0079 (10)	0.0153 (9)	0.0016 (9)
C3	0.0481 (10)	0.0477 (10)	0.0430 (10)	0.0015 (8)	0.0115 (8)	0.0017 (8)
C4	0.0505 (11)	0.0663 (13)	0.0500 (11)	0.0015 (9)	0.0128 (9)	-0.0087 (9)
C5	0.0619 (13)	0.0676 (13)	0.0472 (11)	0.0005 (10)	0.0072 (9)	-0.0116 (10)
C6	0.0522 (11)	0.0544 (11)	0.0397 (9)	0.0043 (8)	0.0118 (8)	0.0013 (8)
C7	0.0507 (11)	0.0654 (13)	0.0400 (10)	0.0021 (9)	0.0090 (8)	-0.0020 (9)
C8	0.0506 (11)	0.0582 (12)	0.0387 (10)	0.0054 (9)	0.0077 (8)	0.0025 (8)
O9	0.0584 (9)	0.1032 (13)	0.0407 (8)	0.0094 (8)	0.0111 (6)	0.0024 (7)
C10	0.0506 (11)	0.0527 (11)	0.0393 (9)	0.0030 (8)	0.0047 (8)	0.0035 (8)
C11	0.0491 (11)	0.0707 (14)	0.0422 (10)	-0.0022 (9)	0.0049 (8)	-0.0041 (9)
C12	0.0534 (12)	0.0780 (15)	0.0476 (11)	-0.0027 (10)	0.0098 (9)	-0.0075 (10)
C13	0.0461 (11)	0.0669 (14)	0.0580 (12)	-0.0025 (9)	0.0089 (9)	0.0003 (10)
C14	0.0550 (12)	0.0813 (16)	0.0472 (11)	-0.0048 (11)	-0.0020 (9)	-0.0035 (10)
C15	0.0553 (12)	0.0759 (14)	0.0398 (10)	0.0003 (10)	0.0045 (9)	-0.0005 (9)
O16	0.0476 (9)	0.1096 (15)	0.0726 (11)	-0.0107 (8)	0.0115 (7)	-0.0139 (9)
C17	0.0590 (15)	0.121 (2)	0.095 (2)	-0.0131 (14)	0.0279 (14)	-0.0264 (17)
C18	0.0593 (16)	0.135 (3)	0.153 (3)	-0.0193 (16)	0.0391 (18)	-0.049 (2)

Geometric parameters (Å, °)

S1—C2	1.701 (2)	C11—H11	0.9300
S1—C5	1.692 (2)	C11—C12	1.385 (3)
C2—H2	0.9300	C12—H12	0.9300
C2—C3	1.357 (3)	C12—C13	1.379 (3)
C3—C4	1.427 (3)	C13—C14	1.392 (3)
C3—C6	1.450 (3)	C13—O16	1.365 (3)
C4—H4	0.9300	C14—H14	0.9300
C4—C5	1.371 (3)	C14—C15	1.359 (3)
C5—H5	0.9300	C15—H15	0.9300
C6—H6	0.9300	O16—C17	1.416 (3)

C6—C7	1.319 (3)	C17—H17A	0.9700
C7—H7	0.9300	C17—H17B	0.9700
C7—C8	1.470 (3)	C17—C18	1.500 (4)
C8—O9	1.224 (2)	C18—H18A	0.9600
C8—C10	1.496 (3)	C18—H18B	0.9600
C10—C11	1.381 (3)	C18—H18C	0.9600
C10—C15	1.398 (3)		
C5—S1—C2	92.34 (10)	C12—C11—H11	119.3
S1—C2—H2	123.7	C11—C12—H12	120.2
C3—C2—S1	112.62 (15)	C13—C12—C11	119.60 (19)
C3—C2—H2	123.7	C13—C12—H12	120.2
C2—C3—C4	110.92 (18)	C12—C13—C14	119.8 (2)
C2—C3—C6	123.30 (17)	O16—C13—C12	124.39 (19)
C4—C3—C6	125.79 (17)	O16—C13—C14	115.84 (19)
C3—C4—H4	123.5	C13—C14—H14	120.1
C5—C4—C3	113.01 (18)	C15—C14—C13	119.78 (19)
C5—C4—H4	123.5	C15—C14—H14	120.1
S1—C5—H5	124.4	C10—C15—H15	119.1
C4—C5—S1	111.11 (15)	C14—C15—C10	121.80 (19)
C4—C5—H5	124.4	C14—C15—H15	119.1
C3—C6—H6	117.0	C13—O16—C17	118.30 (18)
C7—C6—C3	126.00 (18)	O16—C17—H17A	110.0
C7—C6—H6	117.0	O16—C17—H17B	110.0
C6—C7—H7	118.8	O16—C17—C18	108.5 (2)
C6—C7—C8	122.31 (18)	H17A—C17—H17B	108.4
C8—C7—H7	118.8	C18—C17—H17A	110.0
C7—C8—C10	118.74 (16)	C18—C17—H17B	110.0
O9—C8—C7	121.26 (18)	C17—C18—H18A	109.5
O9—C8—C10	119.99 (17)	C17—C18—H18B	109.5
C11—C10—C8	123.51 (17)	C17—C18—H18C	109.5
C11—C10—C15	117.56 (18)	H18A—C18—H18B	109.5
C15—C10—C8	118.92 (17)	H18A—C18—H18C	109.5
C10—C11—H11	119.3	H18B—C18—H18C	109.5
C10—C11—C12	121.47 (19)		
S1—C2—C3—C4	-0.1 (2)	C8—C10—C15—C14	179.3 (2)
S1—C2—C3—C6	179.55 (15)	O9—C8—C10—C11	168.8 (2)
C2—S1—C5—C4	-0.05 (18)	O9—C8—C10—C15	-10.9 (3)
C2—C3—C4—C5	0.1 (3)	C10—C11—C12—C13	0.3 (3)
C2—C3—C6—C7	-177.9 (2)	C11—C10—C15—C14	-0.4 (3)
C3—C4—C5—S1	0.0 (2)	C11—C12—C13—C14	-1.0 (3)
C3—C6—C7—C8	179.75 (18)	C11—C12—C13—O16	178.9 (2)
C4—C3—C6—C7	1.8 (3)	C12—C13—C14—C15	1.0 (3)
C5—S1—C2—C3	0.11 (18)	C12—C13—O16—C17	0.2 (3)
C6—C3—C4—C5	-179.58 (19)	C13—C14—C15—C10	-0.3 (4)
C6—C7—C8—O9	17.1 (3)	C13—O16—C17—C18	-177.0 (2)
C6—C7—C8—C10	-161.57 (19)	C14—C13—O16—C17	-179.9 (2)

C7—C8—C10—C11	-12.6 (3)	C15—C10—C11—C12	0.4 (3)
C7—C8—C10—C15	167.75 (19)	O16—C13—C14—C15	-178.9 (2)
C8—C10—C11—C12	-179.3 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C2—H2 \cdots O9 ⁱ	0.93	2.47	3.324 (2)	153

Symmetry code: (i) $-x+1, -y+1, -z+2$.**1-(4-Bromophenyl)-3-(thiophen-3-yl)prop-1-en-3-one (4)***Crystal data*C₁₃H₉BrOS $M_r = 293.17$ Monoclinic, $P2_1/c$ $a = 14.1245$ (7) Å $b = 14.2016$ (13) Å $c = 5.8809$ (4) Å $\beta = 98.081$ (6)° $V = 1167.93$ (15) Å³ $Z = 4$ $F(000) = 584$ $D_x = 1.667$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4399 reflections

 $\theta = 2.9$ – 27.3 ° $\mu = 3.67$ mm⁻¹ $T = 293$ K

Plate, yellow

 $0.4 \times 0.4 \times 0.05$ mm*Data collection*

SuperNova, single source at offset/far, Eos diffractometer

Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 15.9631 pixels mm⁻¹ ω scans

Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)

 $T_{\min} = 0.367$, $T_{\max} = 1.000$

12050 measured reflections

2392 independent reflections

1683 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.045$ $\theta_{\max} = 26.4$ °, $\theta_{\min} = 2.9$ ° $h = -17$ → 17 $k = -17$ → 17 $l = -7$ → 7 *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.043$ $wR(F^2) = 0.107$ $S = 1.02$

2392 reflections

158 parameters

20 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.7623P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.40$ e Å⁻³ $\Delta\rho_{\min} = -0.46$ e Å⁻³*Special details*

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1A	0.07255 (13)	0.3898 (2)	0.4329 (4)	0.0574 (6)	0.702 (4)
S1B	0.1418 (5)	0.3457 (7)	0.2749 (13)	0.0574 (6)	0.298 (4)
C2A	0.1532 (7)	0.412 (2)	0.663 (3)	0.048 (3)	0.702 (4)
H2A	0.137355	0.439712	0.795694	0.058*	0.702 (4)
C2B	0.2476 (16)	0.341 (5)	0.438 (6)	0.048 (3)	0.298 (4)
H2B	0.301123	0.310516	0.397088	0.058*	0.298 (4)
C3	0.2452 (2)	0.3858 (2)	0.6388 (5)	0.0417 (8)	
C4A	0.2461 (7)	0.3456 (17)	0.416 (2)	0.048 (3)	0.702 (4)
H4A	0.302496	0.326195	0.365401	0.058*	0.702 (4)
C4B	0.1551 (18)	0.422 (5)	0.664 (6)	0.048 (3)	0.298 (4)
H4B	0.142572	0.455562	0.792513	0.058*	0.298 (4)
C5A	0.1586 (7)	0.3375 (9)	0.2809 (18)	0.053 (3)	0.702 (4)
H5A	0.146845	0.309732	0.136500	0.063*	0.702 (4)
C5B	0.0855 (10)	0.4021 (19)	0.475 (3)	0.053 (3)	0.298 (4)
H5B	0.020804	0.416891	0.461047	0.063*	0.298 (4)
C6	0.3264 (2)	0.3933 (2)	0.8186 (6)	0.0451 (8)	
H6	0.313682	0.414696	0.960622	0.054*	
C7	0.4168 (2)	0.3729 (2)	0.8023 (6)	0.0485 (9)	
H7	0.433284	0.356673	0.659823	0.058*	
C8	0.4916 (3)	0.3752 (2)	1.0027 (6)	0.0460 (8)	
O9	0.4718 (2)	0.3764 (2)	1.1988 (4)	0.0676 (8)	
C10	0.5938 (2)	0.3754 (2)	0.9658 (5)	0.0403 (8)	
C11	0.6238 (2)	0.4075 (2)	0.7646 (6)	0.0451 (8)	
H11	0.578794	0.428136	0.644229	0.054*	
C12	0.7202 (3)	0.4092 (3)	0.7410 (6)	0.0470 (8)	
H12	0.740087	0.432427	0.607649	0.056*	
C13	0.7860 (2)	0.3760 (2)	0.9180 (6)	0.0434 (8)	
C14	0.7584 (3)	0.3438 (3)	1.1203 (6)	0.0491 (9)	
H14	0.803570	0.322082	1.239069	0.059*	
C15	0.6624 (2)	0.3447 (2)	1.1429 (6)	0.0447 (8)	
H15	0.643292	0.324227	1.279603	0.054*	
Br16	0.91660 (3)	0.37156 (4)	0.88024 (8)	0.0730 (2)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0459 (8)	0.0718 (14)	0.0532 (11)	0.0051 (8)	0.0029 (6)	-0.0014 (9)
S1B	0.0459 (8)	0.0718 (14)	0.0532 (11)	0.0051 (8)	0.0029 (6)	-0.0014 (9)
C2A	0.044 (4)	0.056 (10)	0.045 (4)	0.006 (4)	0.010 (3)	-0.004 (4)
C2B	0.044 (4)	0.056 (10)	0.045 (4)	0.006 (4)	0.010 (3)	-0.004 (4)
C3	0.0453 (19)	0.0398 (19)	0.0417 (18)	-0.0020 (15)	0.0115 (15)	0.0021 (15)
C4A	0.052 (4)	0.051 (5)	0.043 (5)	-0.002 (3)	0.013 (3)	-0.006 (5)
C4B	0.052 (4)	0.051 (5)	0.043 (5)	-0.002 (3)	0.013 (3)	-0.006 (5)
C5A	0.058 (6)	0.055 (5)	0.051 (4)	0.015 (4)	0.025 (4)	-0.013 (3)
C5B	0.058 (6)	0.055 (5)	0.051 (4)	0.015 (4)	0.025 (4)	-0.013 (3)

C6	0.045 (2)	0.051 (2)	0.0416 (19)	-0.0044 (16)	0.0107 (15)	-0.0040 (15)
C7	0.047 (2)	0.054 (2)	0.0452 (19)	-0.0018 (17)	0.0105 (16)	-0.0068 (16)
C8	0.0447 (19)	0.050 (2)	0.045 (2)	0.0020 (16)	0.0112 (16)	-0.0036 (16)
O9	0.0531 (16)	0.107 (3)	0.0450 (15)	0.0056 (14)	0.0139 (12)	0.0000 (14)
C10	0.0455 (19)	0.0372 (18)	0.0386 (18)	-0.0011 (15)	0.0075 (15)	-0.0038 (14)
C11	0.049 (2)	0.050 (2)	0.0350 (18)	0.0028 (17)	0.0007 (15)	0.0004 (15)
C12	0.054 (2)	0.051 (2)	0.0366 (18)	-0.0055 (17)	0.0101 (16)	0.0013 (16)
C13	0.0382 (18)	0.045 (2)	0.048 (2)	-0.0061 (15)	0.0075 (15)	-0.0056 (16)
C14	0.051 (2)	0.050 (2)	0.0426 (19)	-0.0004 (17)	-0.0036 (16)	0.0041 (16)
C15	0.050 (2)	0.048 (2)	0.0363 (18)	-0.0041 (17)	0.0062 (15)	0.0021 (15)
Br16	0.0430 (3)	0.0970 (4)	0.0803 (4)	-0.0074 (2)	0.0128 (2)	0.0047 (2)

Geometric parameters (Å, °)

S1A—C2A	1.671 (8)	C6—H6	0.9300
C2A—H2A	0.9300	C6—C7	1.325 (5)
S1B—C2B	1.661 (16)	C7—H7	0.9300
C2B—H2B	0.9300	C7—C8	1.469 (5)
C2A—C3	1.378 (9)	C8—O9	1.224 (4)
C2B—C3	1.347 (16)	C8—C10	1.489 (5)
C4A—H4A	0.9300	C10—C11	1.389 (5)
C4B—H4B	0.9300	C10—C15	1.390 (5)
S1A—C5A	1.770 (8)	C11—H11	0.9300
C4A—C5A	1.378 (11)	C11—C12	1.388 (5)
C5A—H5A	0.9300	C12—H12	0.9300
S1B—C5B	1.708 (15)	C12—C13	1.378 (5)
C4B—C5B	1.407 (16)	C13—C14	1.381 (5)
C5B—H5B	0.9300	C13—Br16	1.890 (3)
C3—C4A	1.431 (8)	C14—H14	0.9300
C3—C4B	1.400 (16)	C14—C15	1.381 (5)
C3—C6	1.451 (5)	C15—H15	0.9300
C4A—C5A—S1A	107.3 (6)	C7—C6—C3	127.0 (3)
C4B—C5B—S1B	107.4 (9)	C7—C6—H6	116.5
S1A—C2A—H2A	122.9	C6—C7—H7	119.0
S1B—C2B—H2B	124.6	C6—C7—C8	121.9 (3)
C5A—C4A—C3	116.0 (7)	C8—C7—H7	119.0
C5A—C4A—H4A	122.0	C7—C8—C10	119.1 (3)
C5B—C4B—H4B	123.6	O9—C8—C7	121.5 (3)
C2A—S1A—C5A	92.8 (4)	O9—C8—C10	119.5 (3)
S1A—C5A—H5A	126.3	C11—C10—C8	122.9 (3)
C4A—C5A—H5A	126.3	C11—C10—C15	118.5 (3)
C2B—S1B—C5B	95.2 (7)	C15—C10—C8	118.6 (3)
C4B—C5B—H5B	126.3	C10—C11—H11	119.6
S1B—C5B—H5B	126.3	C12—C11—C10	120.8 (3)
C2A—C3—C4A	109.4 (5)	C12—C11—H11	119.6
C2B—C3—C4B	113.8 (9)	C11—C12—H12	120.5
C4A—C3—C6	126.0 (4)	C13—C12—C11	119.1 (3)

C4B—C3—C6	122.2 (7)	C13—C12—H12	120.5
C2B—C3—C6	124.0 (7)	C12—C13—C14	121.5 (3)
C2A—C3—C6	124.5 (5)	C12—C13—Br16	119.2 (3)
C3—C6—H6	116.5	C14—C13—Br16	119.3 (3)
C3—C2A—S1A	114.3 (5)	C13—C14—H14	120.7
C3—C2B—S1B	110.7 (9)	C15—C14—C13	118.7 (3)
C3—C2A—H2A	122.9	C15—C14—H14	120.7
C3—C2B—H2B	124.6	C10—C15—H15	119.3
C3—C4A—H4A	122.0	C14—C15—C10	121.5 (3)
C3—C4B—H4B	123.6	C14—C15—H15	119.3
C3—C4B—C5B	112.7 (11)		
C2A—S1A—C5A—C4A	-3 (2)	C6—C3—C4B—C5B	-176 (3)
C2B—S1B—C5B—C4B	4 (5)	C6—C7—C8—O9	15.6 (6)
C5B—S1B—C2B—C3	-4 (5)	C6—C7—C8—C10	-164.9 (3)
C5A—S1A—C2A—C3	2 (2)	C7—C8—C10—C11	24.4 (5)
S1A—C2A—C3—C4A	0 (2)	C7—C8—C10—C15	-157.6 (3)
S1B—C2B—C3—C4B	2 (5)	C8—C10—C11—C12	177.8 (3)
S1B—C2B—C3—C6	179.7 (18)	C8—C10—C15—C14	-179.4 (3)
S1A—C2A—C3—C6	-176.6 (10)	O9—C8—C10—C11	-156.0 (4)
C2A—C3—C6—C7	-177.0 (17)	O9—C8—C10—C15	22.0 (5)
C2B—C3—C6—C7	13 (4)	C10—C11—C12—C13	1.8 (5)
C4A—C3—C6—C7	6.9 (15)	C11—C10—C15—C14	-1.3 (5)
C4B—C3—C6—C7	-170 (4)	C11—C12—C13—C14	-2.0 (5)
C2A—C3—C4A—C5A	-3 (2)	C11—C12—C13—Br16	176.5 (3)
C2B—C3—C4B—C5B	1 (6)	C12—C13—C14—C15	0.5 (5)
C3—C4B—C5B—S1B	-4 (7)	C13—C14—C15—C10	1.2 (5)
C3—C4A—C5A—S1A	4 (2)	C15—C10—C11—C12	-0.2 (5)
C3—C6—C7—C8	-174.3 (3)	Br16—C13—C14—C15	-177.9 (3)
C6—C3—C4A—C5A	173.8 (12)		

Hydrogen-bond geometry (Å, °)

Cg1 and Cg2 are the centroids of the major- and minor-disorder components of the thiophene ring, respectively.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C5A—H5A...Cg1 ⁱ	0.93	2.80	3.493 (14)	132
C5A—H5A...Cg2 ⁱ	0.93	2.85	3.52 (2)	130

Symmetry code: (i) *x*, $-y+1/2$, $z-1/2$.