research paper

STRUCTURAL CHEMISTRY

ISSN 2053-2296

Three closely related 1-(naphthalen-2-yl)prop-2-en-1-ones: pseudosymmetry, disorder and supramoleular assembly mediated by C—H···π and C—Br···π interactions

Marisiddaiah Girisha,[a] Belakavadi K. Sagar,[a] Hemmige S. Yathirajan,[a] Ravindranath S. Rathore[b] and Christopher Glidewell[c]*

[a]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysuru 570 006, India, [b]Centre for Biological Sciences (Bioinformatics), School of Earth, Biological and Environmental Sciences, Central University of South Bihar, Patna 800 014, India, and [c]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland. *Correspondence e-mail: cg@st-andrews.ac.uk

Received 19 January 2017
Accepted 20 January 2017

Edited by A. L. Spek, Utrecht University, The Netherlands

Keywords: chalcones; crystal structure; pseudosymmetry; disorder; molecular conformation; molecular aggregate; hydrogen bonding; C—Br···π bonding; Claisen–Schmidt condensation reaction.

CCDC references: 1528834; 1528833; 1528832

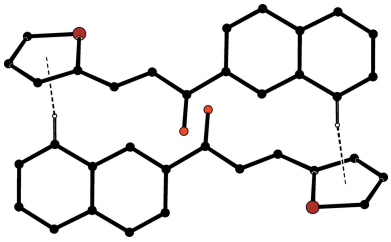
Supporting information: this article has supporting information at journals.iucr.org/c

It has been observed that when electron-rich naphthyl rings are present in chalcones they can participate in π–π stacking interactions, and this can play an important role in orientating inhibitors within the active sites of enzymes, while chalcones containing heterocyclic substituents additionally exhibit fungistatic and fungicidal properties. With these considerations in mind, three new chalcones containing 2-naphthyl substituents were prepared. 3-(4-Fluorophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one, $C_{19}H_{13}FO$, (I), crystallizes with $Z' = 2$ in the space group $P\overline{1}$ and the four molecules in the unit cell adopt an arrangement which resembles that in the space group $P2_1/a$. Although 3-(4-bromophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one, $C_{19}H_{13}BrO$, (II), with $Z' = 1$, is not isostructural with (I), the molecules of (I) and (II) adopt very similar conformations. In 1-(naphthalen-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one, $C_{17}H_{12}OS$, (III), the thiophene unit is disordered over two sets of atomic sites, with occupancies of 0.780 (3) and 0.220 (3), which are related by a near 180° rotation of the thiophene unit about its exocyclic C—C bond. The molecules of compound (I) are linked by three independent C—H···π(arene) hydrogen bonds to form centrosymmetric octamolecular aggregates, whereas the molecules of compound (II) are linked into molecular ladders by a combination of C—H···π(arene) and C—Br···π(arene) interactions, and those of compound (III) are linked into centrosymmetric dimers by C—H···π(thiophene) interactions.

1. Introduction

Chalcones, or 1,3-disubstituted-prop-2-en-1-ones (R^1COCH=CHR^2), exhibit a range of biological activities (Di Carlo *et al.*, 1999; Dimmock *et al.*, 1999), including potential as effective pharmaceutical agents in a number of applications, such as anticancer agents (Lawrence *et al.*, 2006) and as anti-infective and anti-inflammatory agents (Nowakowska, 2007). It has been observed that when electron-rich naphthyl rings are present in chalcones they can participate in π–π stacking interactions, and this can play an important role in orientating inhibitors within the active sites of enzymes (Mascarello *et al.*, 2010), while chalcones containing heterocyclic substituents additionally exhibit fungistatic and fungicidal properties (Opletalová & Sedivý, 1999).

Prompted by these considerations, we have prepared three new chalcones containing 2-naphthyl substituents, namely 3-(4-fluorophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one, (I), 3-(4-bromophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one, (II), and 1-(naphthalen-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one, (III)



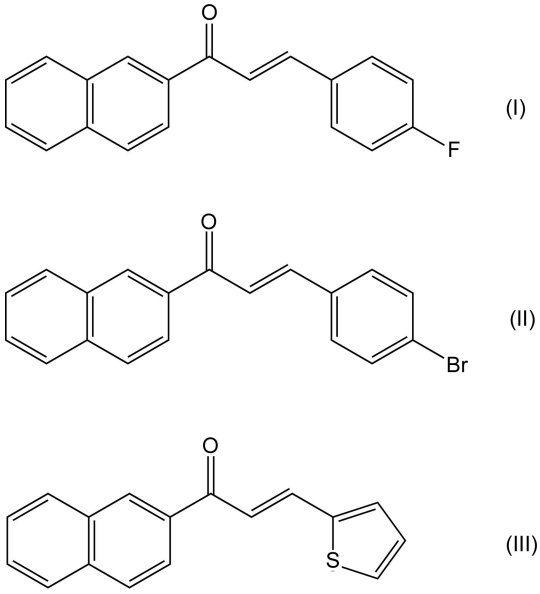
© 2017 International Union of Crystallography

Table 1
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{19}H_{13}FO$	$C_{19}H_{13}BrO$	$C_{17}H_{12}OS$
M_r	276.29	337.19	264.33
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	296	296	296
a, b, c (Å)	7.6678 (4), 11.5007 (5), 15.7874 (8)	5.8714 (5), 7.8616 (7), 15.5954 (13)	5.8271 (3), 7.4489 (4), 15.2068 (9)
α, β, γ (°)	96.249 (3), 96.752 (3), 90.796 (3)	95.007 (5), 90.218 (5), 92.179 (5)	79.745 (3), 84.427 (3), 85.763 (3)
V (Å^3)	1373.84 (12)	716.58 (11)	645.36 (6)
Z	4	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.09	2.86	0.24
Crystal size (mm)	0.41 × 0.32 × 0.30	0.30 × 0.28 × 0.27	0.25 × 0.20 × 0.15
Data collection			
Diffractometer	Bruker Kappa APEXII	Bruker Kappa APEXII	Bruker Kappa APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.960, 0.973	0.451, 0.462	0.933, 0.965
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	25027, 5721, 3046	12745, 2941, 1842	18170, 2694, 2164
R_{int}	0.032	0.054	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629	0.629	0.629
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.055, 0.182, 1.06	0.049, 0.082, 1.05	0.058, 0.140, 1.12
No. of reflections	5721	2941	2694
No. of parameters	379	190	185
No. of restraints	0	0	15
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.19	0.44, −0.47	0.23, −0.41

Computer programs: *APEX2* (Bruker, 2012), *SAINT-Plus* (Bruker, 2012), *SHELXS86* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(Figs. 1–3, and Scheme 1), whose molecular and supramolecular structures we report here. The compounds were all



Scheme 1

prepared by Claisen–Schmidt condensation reactions between 2-acetylnaphthalene and the appropriate aldehyde. The constitutions of compounds (I) and (II) differ only in the identity of the 4-halogeno substituent in the aryl ring, while compound (III) contains a thiophen-2-yl unit instead of a substituted aryl ring.

2. Experimental

2.1. Synthesis and crystallization

For the synthesis of compounds (I)–(III), an aqueous potassium hydroxide solution (50% *w*/*v*, 5 ml) was added to equimolar mixtures (0.58 mmol of each component) of 2-acetylnaphthalene and the appropriate aldehyde [*i.e.* 4-fluorobenzaldehyde for (I), 4-bromobenzaldehyde for (II) and thiophene-2-carbaldehyde for (III)] in methanol (20 ml). The mixtures were then stirred at ambient temperature for 4 h, an excess of ice-cold water was added, and the resulting solid products were collected by filtration and dried in air. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in dichloromethane–ethyl acetate (1:1 *v*/*v*) for (I) and (III) or dimethyl sulfoxide for (II). The yields were 80% for (I), 85% for (II) and 75% for (III).

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. It was apparent from an early stage in the refinement of compound (III) that the thiophene unit was disordered over two sets of atomic sites

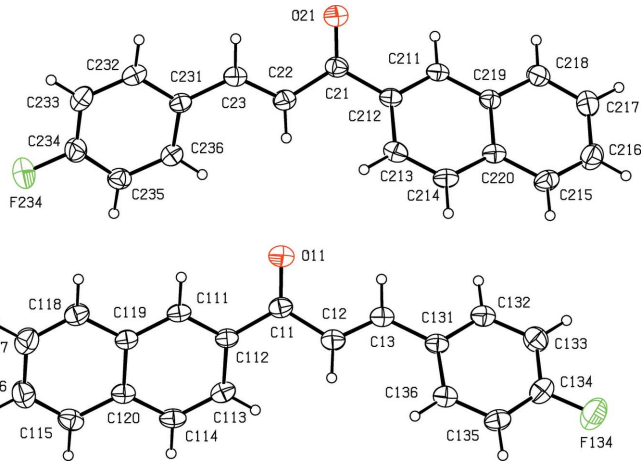


Figure 1
The structures of the two independent molecules in compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

having unequal occupancies. For the minor-disorder component, the bonded distances and 1,3-nonbonded distances were restrained to be the same as the corresponding distances in the major-disorder component, subject to s.u. values of 0.005 and 0.01 Å, respectively. In addition, the anisotropic displacement parameters for pairs of atoms occupying essentially the same volumes of physical space were constrained to be identical. All H atoms, apart from those in the minor-disorder component of compound (III), were located in difference maps, and were then treated as riding atoms in geometrically idealized positions, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). The H atoms in the minor-disorder component of compound (III) were included in calculated positions on the same basis. With these conditions, the occupancies of the disorder components in (III) refined to 0.780 (3) and 0.220 (3). In the final analysis of variance for compound (I), there were negative values of K = mean(F_o^2)/mean(F_c^2) for the two groups of the very weakest reflections: K = −7.211 for the 749 reflections having F_c/F_c(max) in the range 0.000 < F_c/F_c(max) < 0.002, and K = −0.461 for the 649 reflections having F_c/F_c(max) in the range 0.002 < F_c/F_c(max) < 0.004. For compounds (II) and (III), there were values of K = 2.844 and 2.982, respectively, for the 311 reflections having F_c/F_c(max) in the range 0.000 < F_c/F_c(max) < 0.008, and the 323 reflections having F_c/F_c(max) in the range 0.000 < F_c/F_c(max) < 0.006.

3. Results and discussion

Compound (I) crystallizes with Z' = 2 in the space group $P\overline{1}$. The two independent molecules (Fig. 1) were selected to have the same signs for the torsion angles Cx2—Cx1—Cx12—Cx11 and Cx2—Cx3—Cx31—Cx32 (Table 2), where x = 1 for molecule 1, containing atom O11, and x = 2 for molecule 2, containing atom O21, and the values indicate very similar conformations for the two independent molecules. The corresponding values for compound (II) (Fig. 2) indicate a

Table 2
Selected torsion angles (°) for compounds (I)–(III).

Parameter	(I) x = 1	(I) x = 2	(II) x = nil	(III) x = nil
Cx2—Cx1—Cx12—Cx11	155.7 (2)	151.6 (2)	158.6 (3)	158.1 (3)
Cx2—Cx3—Cx31—Cx32	169.7 (2)	167.2 (2)	169.5 (4)	
Cx2—Cx3—Cx32—Cx33				171.1 (5)
Cx2—Cx3—Cx32—Sx41				171.3 (4)

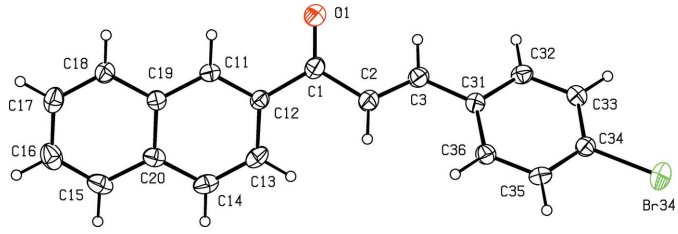


Figure 2
The molecular structure of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

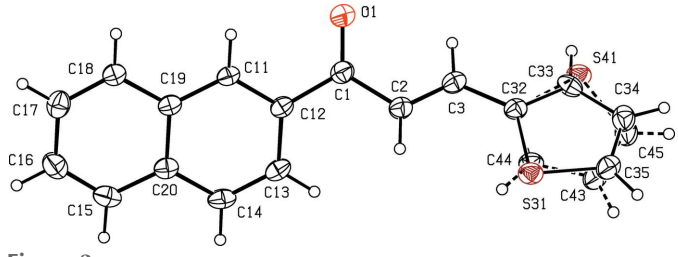


Figure 3
The molecular structure of compound (III), showing the atom-labelling scheme and the disordered thiophene unit; the major component, with occupancy 0.780 (3), is shown with full lines and the minor component, with occupancy 0.220 (3), is shown with broken lines. Displacement ellipsoids are drawn at the 30% probability level.

molecular conformation in (II) very similar to those found in (I).

In compound (III), the thiophene unit is disordered over two sets of atomic sites, with occupancies of 0.780 (3) and 0.220 (3) (Fig. 3). The two disorder components are related by an approximate 180° rotation about the exocyclic C—C bond, so that (III) exhibits conformational disorder, with a dihedral angle between the major and minor forms of the thiophene ring of only 6.0 (8)°. The torsion angle C2—C1—C12—C11 (Table 2) is very similar to the corresponding values in compounds (I) and (II); the torsion angles C2—C3—C32—C33 and C2—C3—C32—S41 are again close to the analogous values in compounds (I) and (II), so that, despite their different compositions, the overall conformations of the molecules in (I)–(III) are all very similar.

In all of the structures reported here, the naphthalene units exhibit strong bond fixation; the bonds Cx11—Cx12, Cx13—Cx14, Cx15—Cx16 and Cx17—Cx18, where x = 1 or 2 in compound (I) and nil in each of (II) and (III) (Figs. 1–3),are always significantly shorter than the other C—C bonds in the naphthalene units, as expected from the bond orders calculated for naphthalene itself (Glidewell & Lloyd, 1984).

Table 3
Hydrogen-bond parameters (Å, °) for compounds (I)–(III).

*Cg*1–*Cg*5 represent the centroids of the C115–C120, C231–C236, C111–C114/C120/C119, C15–C20 and S31/C32–C35 rings, respectively.

	D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)	C132—H132···*Cg*1^{i}	0.93	2.88	3.557 (3)	131
	C135—H135···*Cg*2^{ii}	0.93	2.83	3.503 (3)	130
	C214—H214···*Cg*3^{ii}	0.93	2.87	3.554 (2)	131
(II)	C32—H32···*Cg*4^{iii}	0.93	2.85	3.513 (3)	129
(III)	C18—H18···*Cg*5^{iv}	0.93	2.80	3.572 (4)	141

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

For the two molecules in the selected asymmetric unit of compound (I), comparison of the atomic coordinates for corresponding pairs of atoms in the two molecules shows that they are related by an approximate, although noncrystallographic, 2_1 screw axis along the line $(\frac{1}{4}, y, \frac{1}{2})$; a similar comparison for pairs of atoms in molecule 1 at (x, y, z) and molecule 2 at $(-x+1, -y+1, -z+1)$ shows that these are related by an approximate, but noncrystallographic, *a*-glide plane at $y = \frac{1}{4}$. Hence, although the unit cell for (I) has only triclinic symmetry, the arrangement of the four molecules within it approximately mimics the arrangement that would be found in the space group $P2_1/a$ (Fig. 4).

The supramolecular assemblies in compounds (I)–(III) are determined by C—H···π hydrogen bonds (Table 3), augmented in the case of (II) (Table 4) by a C—Br···π interaction (Matter *et al.*, 2009; Mazik *et al.*, 2010). The C—H···π interactions in (I)–(III) all have fairly long H···*Cg* distances and we have considered as structurally significant only those interactions having H···*Cg* distances less than 2.90 Å (*cf.* Braga *et al.*, 1998; Takahashi *et al.*, 2001). On this basis, there are three significant C—H···π hydrogen bonds in the structure of compound (I) (Table 3) and these combine to link the molecules into aggregates containing four molecules of each of types 1 and 2 (Fig. 5). The type 1 molecules act as double donors and double acceptors, while the type 2 molecules act as single donors and single acceptors. The octamolecular aggregate contains molecules of type 1 at the positions (x, y, z), $(-x, -y+1, -z+1)$, $(x-1, y, z)$ and $(-x+1, -y+1, -z+1)$, and molecules of type 2 at the positions (x, y, z), $(-x, -y+1, -z+1)$, $(x+1, y, z)$ and $(-x-1, -y+1, -z+1)$, so that the whole aggregate is centred at $(0, \frac{1}{2}, \frac{1}{2})$

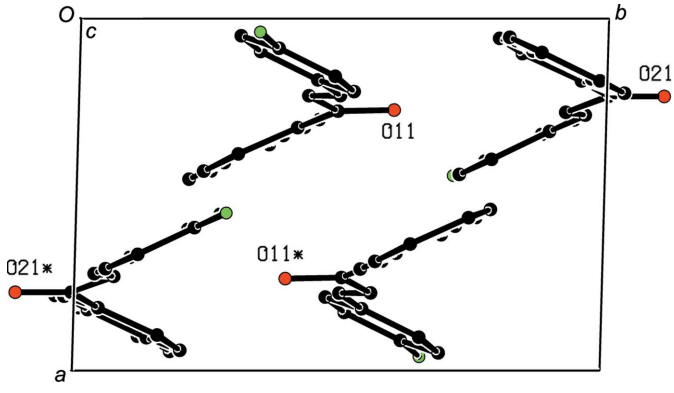


Figure 4
The arrangement of the four molecules in the unit cell of compound (I), showing the resemblance to that expected in the space group $P2_1/a$. For the sake of clarity, H atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(-x+1, -y+1, -z+1)$.

Table 4
Parameters (Å, °) for the C—Br···π(arene) interaction in compound (II).

*Cg*4 represents the centroid of the C15–C20 ring.

C—Br···*Cg*	C—Br	Br···*Cg*	C···*Cg*	C—Br···*Cg*
C34—Br34···*Cg*4^{ii}	1.889 (3)	3.7527 (15)	4.620 (3)	105.13 (10)

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

In the structure of compound (II), a combination of one C—H···π hydrogen bond (Table 3) and one C—Br···π interaction (Table 4) combine to link the molecules into a ribbon, or molecular ladder, running parallel to the $[1\bar{1}0]$ direction (Fig. 6). The ribbon contains two types of rings, both centrosymmetric, with rings containing inversion-related pairs of C—H···π hydrogen bonds centred at $(\frac{1}{2}-n, n, \frac{1}{2})$ and those containing inversion-related pairs of C—Br···π interactions centred at $(-n, \frac{1}{2}+n, \frac{1}{2})$, where *n* represents an integer in each case. It is interesting to note that, despite the presence of three independent aromatic rings within the molecule, both interactions involved in the formation of the ribbon utilize the same ring, one on each face, such that the angle

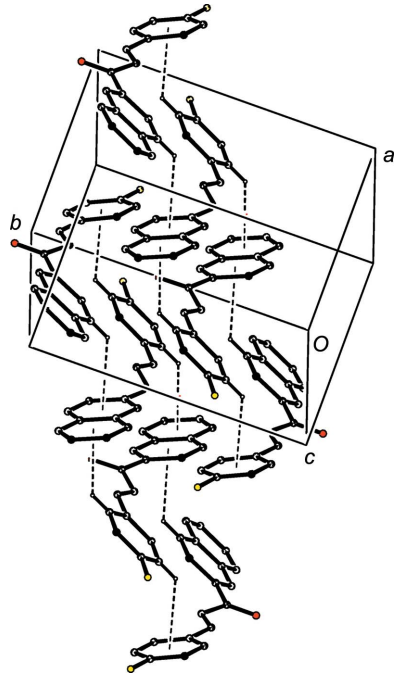


Figure 5
Part of the crystal structure of compound (I), showing the formation of an eight-molecule aggregate centred at $(0, \frac{1}{2}, \frac{1}{2})$ generated by three independent C—H···π hydrogen bonds (shown as dashed lines). For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

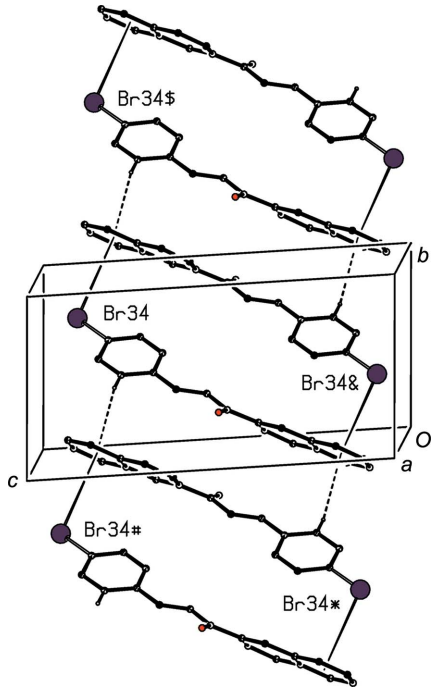
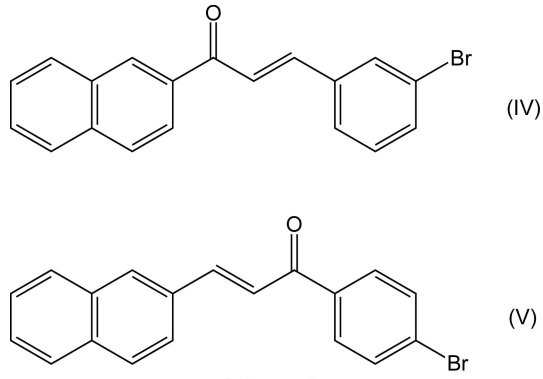


Figure 6
Part of the crystal structure of compound (II), showing the formation of a ribbon or molecular ladder running parallel to [1$\bar{1}$0] and built from a combination of C—H···π and C—Br···π interactions (shown as dashed and tapered lines, respectively). For the sake of clarity, H atoms not involved in the motif shown have been omitted. The Br atoms marked with an asterisk (*), a hash (#), a dollar sign ($) or an ampersand (&) are at the symmetry positions ($-x+1, -y, -z+1$), ($x+1, y-1, z$), ($x-1, y+1, z$) and ($-x, -y+1, -z+1$), respectively,

Br34^{ii}···*Cg*4···H32^{iii} is 165.8° [symmetry codes: (ii) $-x, -y+1, -z+1$; (iii) $-x+1, -y, -z+1$; *Cg*4 represents the centroid of the C15–C20 ring (*cf.* Tables 3 and 4)].

By comparison with the supramolecular aggregation in compounds (I) and (II), that in compound (III) is very simple: a single C—H···π interaction (Table 3) links inversion-related molecules into a centrosymmetric dimer (Fig. 7).

It is of interest briefly to compare the structures of compounds (I)–(III) reported here with those of some related compounds. 3-(3-Bromophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one, (IV) (Scheme 2) (Moorthi *et al.*, 2007), is a positional isomer of compound (II), differing from it only in the location of the Br substituent. While compounds (II) and (IV) crystallize in the same space group, *i.e.* $P\bar{1}$, with fairly similar reduced cell repeat vectors, in (II), the inter-axial angles are all greater than 90°, whereas those in (IV) are all less than 90°, so that these compounds cannot be even approximately isostructural. The supramolecular assembly of (IV) was described (Moorthi *et al.*, 2007) as comprising chains built from C—H···Br hydrogen bonds. However, this description must be questioned on two grounds; firstly, the H···Br distance in question, 2.93 Å, is not significantly shorter than the sum, 2.94 Å, of the van der Waals radii (Rowland & Taylor, 1996), and secondly, it has been convincingly demonstrated that Br atoms bonded to C atoms are extremely poor acceptors even from good hydrogen-bond donors, such as O—H and N—H, and correspondingly worse from a weak donor, such as C—H (Brammer *et al.*, 2001; Thallapally & Nangia, 2001). (*E*)-1-(4-Bromophenyl)-3-(naphthalen-2-yl)-prop-2-en-1-one, (V) (Thanigaimani *et al.*, 2015), is also an isomer of compound (II) but with the locations of the aryl and naphthyl units interchanged. Compound (V) also crystallizes in the space group $P\bar{1}$, with reduced cell repeat vectors also similar to those of compounds (II) and (V), and with the inter-axial angles all greater than 90°, although (V) is not isostructural with (II). The supramolecular assembly in (V) depends upon a single C—Br···π interaction between inversion-related pairs of molecules forming centrosymmetric dimers.



Scheme 2

The structures have also been reported for a number of related chalcones derived from 6-methoxynaphthaldehyde (Yathirajan *et al.*, 2006; Butcher *et al.*, 2007; Jasinski *et al.*, 2009; Nayak *et al.*, 2014; Patel & Chadha, 2015). We also note the structures of a number of chalcones containing halogen-

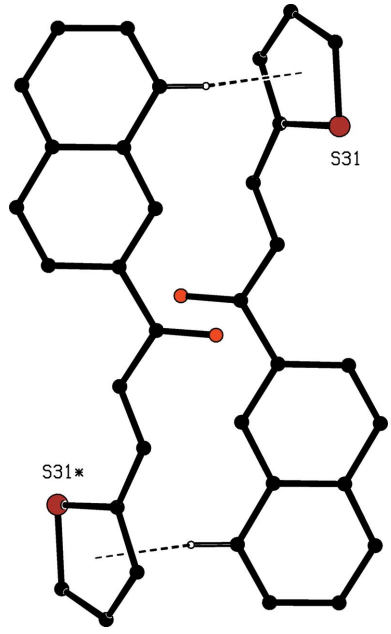


Figure 7
Part of the crystal structure of compound (III), showing the formation of a centrosymmetric dimer built from C—H···π hydrogen bonds (shown as dashed lines). For the sake of clarity, the unit-cell outline, the minor component of the disordered thiophene unit and H atoms not involved in the motif shown have all been omitted. The S atom marked with an asterisk (*) is at the symmetry position ($-x+2, -y+1, -z+1$).

substituted thiophene units (Butcher *et al.*, 2007; Naik *et al.*, 2015; Girisha *et al.*, 2016).

Acknowledgements

MG and BKS each thank the UGC (India) for the award of a Rajeev Gandhi fellowship.

References

Braga, D., Grepioni, F. & Tedesco, E. (1998). *Organometallics*, **17**, 2669–2672.
Brammer, L., Bruton, E. A. & Sherwood, P. (2001). *Cryst. Growth Des.* **1**, 277–290.
Bruker (2012). *APEX2*, *SADABS* and *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
Butcher, R. J., Yathirajan, H. S., Ashalatha, B. V., Narayana, B. & Sarojini, B. K. (2007). *Acta Cryst.* E**63**, o1430–o1431.
Di Carlo, G., Mascolo, N., Izzo, A. A. & Capasso, F. (1999). *Life Sci.* **65**, 337–353.
Dimmock, J. R., Elias, D. W., Beazely, M. A. & Kandepu, N. M. (1999). *Curr. Med. Chem.* **6**, 1125–1149.
Girisha, M., Yathirajan, H. S., Jasinski, J. P. & Glidewell, C. (2016). *Acta Cryst.* E**72**, 1153–1158.
Glidewell, C. & Lloyd, D. (1984). *Tetrahedron*, **40**, 4455–4472.
Jasinski, J. P., Butcher, R. J., Mayekar, A. N., Yathirajan, H. S. & Narayana, B. (2009). *J. Chem. Crystallogr.* **39**, 157–162.
Lawrence, N. J., Patterson, R. P., Ooi, L.-L., Cook, D. & Ducki, S. (2006). *Bioorg. Med. Chem. Lett.* **16**, 5844–5848.
Mascarello, A., Chiaradia, L. D., Vernal, J., Villarino, A., Guido, R. V. C., Perizzolo, P., Poirier, V., Wong, D., Martins, P. G. A., Nunes, R. J., Yunes, R. A., Andricopulo, A. D., Av-Gay, Y. & Terenzi, H. (2010). *Bioorg. Med. Chem.* **18**, 3783–3789.
Matter, H., Nazaré, M., Güssregen, S., Will, D. W., Schreuder, H., Bauer, A., Urmann, M., Ritter, K., Wagner, M. & Wehner, V. (2009). *Angew. Chem. Int. Ed.* **48**, 2911–2916.
Mazik, M., Buthe, A. C. & Jones, P. G. (2010). *Tetrahedron*, **66**, 385–389.
Moorthi, S. S., Chinnakali, K., Nanjundan, S., Radhakrishnanan, S. & Fun, H.-K. (2007). *Acta Cryst.* E**63**, o692–o694.
Naik, V. S., Yathirajan, H. S., Jasinski, J. P., Smolenski, V. A. & Glidewell, C. (2015). *Acta Cryst.* E**71**, 1093–1099.
Nayak, P. S., Narayana, B., Yathirajan, H. S., Hosten, E. C., Betz, R. & Glidewell, C. (2014). *Acta Cryst.* C**70**, 1011–1016.
Nowakowska, Z. (2007). *Eur. J. Med. Chem.* **42**, 125–137.
Opletalová, V. & Sedivý, D. (1999). *Ceska Slov. Farm.* **48**, 252–255.
Patel, P. N. & Chadha, A. (2015). *Acta Cryst.* E**71**, o884–o885.
Rowland, R. S. & Taylor, R. (1996). *J. Phys. Chem.* **100**, 7384–7391.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Takahashi, O., Kohno, Y., Iwasaki, S., Saito, K., Iwaoka, M., Tomoda, S., Umezawa, Y., Tauboyama, S. & Nishio, M. (2001). *Bull. Chem. Soc. Jpn*, **74**, 2421–2430.
Thallapally, P. K. & Nangia, A. (2001). *CrystEngComm*, **27**, 1–6.
Thanigaimani, K., Arshad, S., Khalib, N. C., Razak, I. A., Arunagiri, C., Subashini, A., Sulaiman, S. F., Hashim, N. S. & Ooi, K. L. (2015). *Spectrochim. Acta Part A*, **149**, 90–102.
Yathirajan, H. S., Sarojini, B. K., Bindya, S., Narayana, B. & Bolte, M. (2006). *Acta Cryst.* E**62**, o4046–o4047.

supporting information

Acta Cryst. (2017). C**73**, 115-120 [https://doi.org/10.1107/S205322961700105X]

Three closely related 1-(naphthalen-2-yl)prop-2-en-1-ones: pseudosymmetry, disorder and supramoleular assembly mediated by C—H···π and C—Br···π interactions

Marisiddaiah Girisha, Belakavadi K. Sagar, Hemmige S. Yathirajan, Ravindranath S. Rathore and Christopher Glidewell

Computing details

For all compounds, data collection: *APEX2* (Bruker, 2012); cell refinement: *APEX2* (Bruker, 2012); data reduction: *SAINT-Plus* (Bruker, 2012); program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(I) 3-(4-Fluorophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one

Crystal data

$C_{19}H_{13}FO$
M_r = 276.29
Triclinic, $P\overline{1}$
a = 7.6678 (4) Å
b = 11.5007 (5) Å
c = 15.7874 (8) Å
α = 96.249 (3)°
β = 96.752 (3)°
γ = 90.796 (3)°
V = 1373.84 (12) Å^3
Z = 4
$F(000)$ = 576
D_x = 1.336 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6071 reflections
θ = 1.3–27.2°
μ = 0.09 mm^{-1}
T = 296 K
Block, colourless
0.41 × 0.32 × 0.30 mm

Data collection

Bruker Kappa APEXII diffractometer
Radiation source: fine focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.960, T_{max} = 0.973
25027 measured reflections
5721 independent reflections
3046 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 26.6°, θ_{min} = 1.8°
h = −9→9
k = −14→14
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.182
S = 1.06
5721 reflections
379 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.5614P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C11	0.2636 (3)	0.49245 (19)	0.46846 (15)	0.0455 (6)
O11	0.2606 (3)	0.59900 (14)	0.46856 (12)	0.0680 (6)
C12	0.2197 (3)	0.4364 (2)	0.54308 (16)	0.0538 (7)
H12	0.1913	0.3568	0.5362	0.065*
C13	0.2193 (3)	0.49594 (19)	0.61931 (16)	0.0458 (6)
H13	0.2513	0.5749	0.6237	0.055*
C111	0.2880 (3)	0.45775 (18)	0.31358 (15)	0.0407 (5)
H111	0.2416	0.5313	0.3088	0.049*
C112	0.3106 (3)	0.41720 (18)	0.39246 (15)	0.0421 (5)
C113	0.3852 (3)	0.30607 (19)	0.39978 (16)	0.0473 (6)
H113	0.4006	0.2773	0.4529	0.057*
C114	0.4342 (3)	0.24143 (19)	0.32976 (17)	0.0488 (6)
H114	0.4852	0.1695	0.3361	0.059*
C115	0.4574 (3)	0.2146 (2)	0.17426 (18)	0.0532 (6)
H115	0.5056	0.1414	0.1788	0.064*
C116	0.4343 (4)	0.2558 (2)	0.09649 (19)	0.0648 (8)
H116	0.4684	0.2113	0.0487	0.078*
C117	0.3594 (4)	0.3650 (2)	0.08786 (18)	0.0643 (7)
H117	0.3434	0.3926	0.0343	0.077*
C118	0.3100 (3)	0.4308 (2)	0.15763 (16)	0.0530 (6)
H118	0.2602	0.5031	0.1512	0.064*
C119	0.3332 (3)	0.39099 (18)	0.23947 (15)	0.0397 (5)
C120	0.4100 (3)	0.28039 (18)	0.24811 (15)	0.0425 (6)
C131	0.1745 (3)	0.45205 (18)	0.69754 (15)	0.0425 (6)
C132	0.2077 (3)	0.5214 (2)	0.77522 (16)	0.0508 (6)
H132	0.2608	0.5949	0.7768	0.061*
C133	0.1641 (4)	0.4845 (2)	0.85047 (17)	0.0594 (7)
H133	0.1883	0.5316	0.9024	0.071*
C134	0.0843 (4)	0.3767 (2)	0.84632 (18)	0.0589 (7)
F134	0.0381 (3)	0.34032 (16)	0.91995 (11)	0.0930 (6)
C135	0.0490 (3)	0.3046 (2)	0.77151 (17)	0.0536 (6)
H135	−0.0044	0.2313	0.7708	0.064*
C136	0.0938 (3)	0.3424 (2)	0.69728 (17)	0.0488 (6)
H136	0.0701	0.2941	0.6459	0.059*
C21	0.2219 (3)	1.00381 (19)	0.54713 (15)	0.0453 (6)
O21	0.2219 (3)	1.10970 (14)	0.54542 (11)	0.0664 (5)

C22	0.2670 (3)	0.9241 (2)	0.47339 (15)	0.0508 (6)
H22	0.2901	0.8464	0.4804	0.061*
C23	0.2750 (3)	0.96147 (19)	0.39753 (15)	0.0455 (6)
H23	0.2476	1.0393	0.3932	0.055*
C211	0.2104 (3)	1.01657 (17)	0.70294 (14)	0.0398 (5)
H211	0.2589	1.0918	0.7067	0.048*
C212	0.1777 (3)	0.95200 (18)	0.62421 (15)	0.0413 (5)
C213	0.1001 (3)	0.83855 (19)	0.61895 (16)	0.0470 (6)
H213	0.0788	0.7937	0.5659	0.056*
C214	0.0566 (3)	0.79453 (19)	0.69011 (16)	0.0471 (6)
H214	0.0022	0.7209	0.6849	0.057*
C215	0.0533 (3)	0.8133 (2)	0.84710 (17)	0.0533 (7)
H215	−0.0028	0.7404	0.8432	0.064*
C216	0.0963 (4)	0.8748 (2)	0.92514 (18)	0.0639 (7)
H216	0.0723	0.8430	0.9744	0.077*
C217	0.1770 (4)	0.9862 (2)	0.93198 (17)	0.0619 (7)
H217	0.2065	1.0280	0.9857	0.074*
C218	0.2122 (3)	1.0334 (2)	0.86059 (16)	0.0508 (6)
H218	0.2636	1.1079	0.8659	0.061*
C219	0.1722 (3)	0.97130 (18)	0.77841 (14)	0.0391 (5)
C220	0.0922 (3)	0.85821 (18)	0.77167 (15)	0.0406 (5)
C231	0.3216 (3)	0.89484 (18)	0.32013 (14)	0.0407 (5)
C232	0.2893 (3)	0.9409 (2)	0.24220 (16)	0.0497 (6)
H232	0.2392	1.0139	0.2407	0.060*
C233	0.3301 (3)	0.8805 (2)	0.16714 (17)	0.0578 (7)
H233	0.3064	0.9113	0.1150	0.069*
C234	0.4060 (4)	0.7744 (2)	0.17121 (17)	0.0576 (7)
F234	0.4474 (3)	0.71518 (15)	0.09738 (11)	0.0926 (6)
C235	0.4438 (3)	0.7263 (2)	0.24616 (16)	0.0500 (6)
H235	0.4976	0.6544	0.2469	0.060*
C236	0.4009 (3)	0.78637 (19)	0.32071 (16)	0.0466 (6)
H236	0.4250	0.7542	0.3723	0.056*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0406 (14)	0.0416 (13)	0.0530 (15)	−0.0046 (10)	−0.0008 (11)	0.0067 (11)
O11	0.0959 (15)	0.0433 (10)	0.0657 (12)	−0.0017 (9)	0.0145 (10)	0.0047 (8)
C12	0.0609 (17)	0.0413 (13)	0.0590 (17)	−0.0063 (11)	0.0085 (13)	0.0034 (12)
C13	0.0413 (14)	0.0379 (12)	0.0581 (16)	0.0012 (10)	0.0048 (11)	0.0066 (11)
C111	0.0335 (12)	0.0328 (11)	0.0558 (15)	0.0008 (9)	0.0003 (10)	0.0104 (10)
C112	0.0386 (13)	0.0355 (11)	0.0513 (15)	−0.0050 (10)	0.0021 (11)	0.0053 (10)
C113	0.0405 (14)	0.0455 (13)	0.0557 (15)	−0.0025 (11)	−0.0029 (11)	0.0145 (11)
C114	0.0389 (14)	0.0366 (12)	0.0710 (18)	0.0057 (10)	0.0021 (12)	0.0104 (12)
C115	0.0421 (14)	0.0449 (13)	0.0724 (19)	0.0025 (11)	0.0119 (13)	0.0004 (13)
C116	0.0635 (19)	0.0711 (18)	0.0595 (19)	−0.0033 (15)	0.0158 (14)	−0.0033 (15)
C117	0.0679 (19)	0.0714 (18)	0.0541 (17)	−0.0033 (15)	0.0041 (14)	0.0133 (14)
C118	0.0516 (16)	0.0502 (14)	0.0574 (17)	0.0018 (12)	0.0006 (13)	0.0126 (12)

C119	0.0297 (12)	0.0379 (11)	0.0510 (14)	−0.0041 (9)	0.0013 (10)	0.0069 (10)
C120	0.0305 (12)	0.0387 (12)	0.0581 (16)	−0.0024 (9)	0.0029 (11)	0.0065 (11)
C131	0.0348 (13)	0.0371 (11)	0.0553 (15)	0.0043 (10)	0.0028 (11)	0.0056 (11)
C132	0.0472 (15)	0.0426 (13)	0.0625 (17)	0.0002 (11)	0.0069 (12)	0.0053 (12)
C133	0.0583 (17)	0.0612 (16)	0.0557 (17)	−0.0013 (13)	0.0004 (13)	0.0008 (13)
C134	0.0538 (17)	0.0670 (17)	0.0590 (18)	0.0017 (13)	0.0061 (13)	0.0217 (14)
F134	0.1102 (15)	0.1042 (13)	0.0695 (12)	−0.0169 (11)	0.0150 (10)	0.0292 (10)
C135	0.0449 (15)	0.0462 (13)	0.0703 (19)	−0.0034 (11)	0.0042 (13)	0.0128 (13)
C136	0.0424 (14)	0.0442 (12)	0.0592 (16)	0.0009 (11)	0.0046 (12)	0.0053 (11)
C21	0.0457 (14)	0.0402 (12)	0.0480 (15)	0.0016 (10)	−0.0018 (11)	0.0038 (11)
O21	0.0979 (16)	0.0444 (10)	0.0589 (12)	0.0038 (9)	0.0149 (10)	0.0084 (8)
C22	0.0614 (17)	0.0417 (13)	0.0499 (15)	0.0062 (11)	0.0045 (12)	0.0096 (11)
C23	0.0435 (14)	0.0382 (12)	0.0553 (16)	0.0015 (10)	0.0063 (11)	0.0066 (11)
C211	0.0363 (12)	0.0297 (10)	0.0533 (15)	0.0004 (9)	0.0065 (10)	0.0030 (10)
C212	0.0388 (13)	0.0346 (11)	0.0501 (15)	0.0049 (9)	0.0030 (11)	0.0054 (10)
C213	0.0427 (14)	0.0411 (12)	0.0535 (15)	0.0038 (10)	−0.0037 (11)	−0.0011 (11)
C214	0.0413 (14)	0.0353 (11)	0.0640 (17)	−0.0023 (10)	0.0022 (12)	0.0070 (11)
C215	0.0467 (15)	0.0468 (13)	0.0702 (19)	−0.0008 (11)	0.0149 (13)	0.0156 (13)
C216	0.0690 (19)	0.0683 (17)	0.0595 (18)	0.0029 (15)	0.0199 (15)	0.0162 (14)
C217	0.0699 (19)	0.0642 (17)	0.0516 (17)	0.0005 (14)	0.0126 (14)	0.0015 (13)
C218	0.0526 (15)	0.0428 (13)	0.0567 (16)	0.0008 (11)	0.0100 (12)	0.0004 (11)
C219	0.0329 (12)	0.0373 (11)	0.0476 (14)	0.0059 (9)	0.0066 (10)	0.0038 (10)
C220	0.0296 (12)	0.0363 (11)	0.0559 (15)	0.0025 (9)	0.0031 (10)	0.0069 (10)
C231	0.0369 (13)	0.0377 (11)	0.0480 (14)	−0.0023 (10)	0.0037 (10)	0.0083 (10)
C232	0.0457 (15)	0.0450 (13)	0.0607 (17)	0.0005 (11)	0.0063 (12)	0.0167 (12)
C233	0.0624 (18)	0.0638 (16)	0.0497 (16)	−0.0073 (14)	0.0067 (13)	0.0189 (13)
C234	0.0617 (18)	0.0574 (16)	0.0555 (17)	−0.0070 (13)	0.0192 (14)	0.0020 (13)
F234	0.1272 (16)	0.0911 (12)	0.0633 (11)	0.0034 (11)	0.0390 (11)	−0.0037 (9)
C235	0.0463 (15)	0.0433 (13)	0.0622 (17)	0.0019 (11)	0.0129 (12)	0.0065 (12)
C236	0.0459 (14)	0.0461 (13)	0.0486 (15)	−0.0011 (11)	0.0052 (11)	0.0098 (11)

Geometric parameters (Å, º)

C11—O11	1.226 (3)	C21—O21	1.221 (2)
C11—C12	1.473 (3)	C21—C22	1.478 (3)
C11—C112	1.484 (3)	C21—C212	1.483 (3)
C12—C13	1.318 (3)	C22—C23	1.323 (3)
C12—H12	0.9300	C22—H22	0.9300
C13—C131	1.459 (3)	C23—C231	1.454 (3)
C13—H13	0.9300	C23—H23	0.9300
C111—C112	1.370 (3)	C211—C212	1.371 (3)
C111—C119	1.409 (3)	C211—C219	1.411 (3)
C111—H111	0.9300	C211—H211	0.9300
C112—C113	1.418 (3)	C212—C213	1.416 (3)
C113—C114	1.357 (3)	C213—C214	1.355 (3)
C113—H113	0.9300	C213—H213	0.9300
C114—C120	1.403 (3)	C214—C220	1.406 (3)
C114—H114	0.9300	C214—H214	0.9300

C115—C116	1.357 (4)	C215—C216	1.354 (3)
C115—C120	1.407 (3)	C215—C220	1.409 (3)
C115—H115	0.9300	C215—H215	0.9300
C116—C117	1.402 (4)	C216—C217	1.403 (4)
C116—H116	0.9300	C216—H216	0.9300
C117—C118	1.362 (3)	C217—C218	1.355 (3)
C117—H117	0.9300	C217—H217	0.9300
C118—C119	1.410 (3)	C218—C219	1.409 (3)
C118—H118	0.9300	C218—H218	0.9300
C119—C120	1.422 (3)	C219—C220	1.418 (3)
C131—C132	1.383 (3)	C231—C232	1.389 (3)
C131—C136	1.396 (3)	C231—C236	1.396 (3)
C132—C133	1.379 (3)	C232—C233	1.378 (3)
C132—H132	0.9300	C232—H232	0.9300
C133—C134	1.368 (4)	C233—C234	1.364 (4)
C133—H133	0.9300	C233—H233	0.9300
C134—F134	1.360 (3)	C234—F234	1.358 (3)
C134—C135	1.364 (4)	C234—C235	1.363 (3)
C135—C136	1.372 (3)	C235—C236	1.375 (3)
C135—H135	0.9300	C235—H235	0.9300
C136—H136	0.9300	C236—H236	0.9300

O11—C11—C12	120.9 (2)	O21—C21—C22	121.3 (2)
O11—C11—C112	120.4 (2)	O21—C21—C212	120.4 (2)
C12—C11—C112	118.7 (2)	C22—C21—C212	118.30 (19)
C13—C12—C11	121.9 (2)	C23—C22—C21	121.2 (2)
C13—C12—H12	119.1	C23—C22—H22	119.4
C11—C12—H12	119.1	C21—C22—H22	119.4
C12—C13—C131	127.4 (2)	C22—C23—C231	127.5 (2)
C12—C13—H13	116.3	C22—C23—H23	116.3
C131—C13—H13	116.3	C231—C23—H23	116.3
C112—C111—C119	121.8 (2)	C212—C211—C219	121.39 (19)
C112—C111—H111	119.1	C212—C211—H211	119.3
C119—C111—H111	119.1	C219—C211—H211	119.3
C111—C112—C113	118.9 (2)	C211—C212—C213	118.9 (2)
C111—C112—C11	119.3 (2)	C211—C212—C21	119.02 (19)
C113—C112—C11	121.7 (2)	C213—C212—C21	122.1 (2)
C114—C113—C112	120.4 (2)	C214—C213—C212	120.9 (2)
C114—C113—H113	119.8	C214—C213—H213	119.6
C112—C113—H113	119.8	C212—C213—H213	119.6
C113—C114—C120	121.7 (2)	C213—C214—C220	121.2 (2)
C113—C114—H114	119.2	C213—C214—H214	119.4
C120—C114—H114	119.2	C220—C214—H214	119.4
C116—C115—C120	121.3 (2)	C216—C215—C220	121.0 (2)
C116—C115—H115	119.4	C216—C215—H215	119.5
C120—C115—H115	119.4	C220—C215—H215	119.5
C115—C116—C117	120.3 (3)	C215—C216—C217	120.2 (2)
C115—C116—H116	119.8	C215—C216—H216	119.9

C117—C116—H116	119.8	C217—C216—H216	119.9
C118—C117—C116	120.2 (3)	C218—C217—C216	120.3 (2)
C118—C117—H117	119.9	C218—C217—H217	119.8
C116—C117—H117	119.9	C216—C217—H217	119.8
C117—C118—C119	120.9 (2)	C217—C218—C219	121.1 (2)
C117—C118—H118	119.5	C217—C218—H218	119.4
C119—C118—H118	119.5	C219—C218—H218	119.4
C111—C119—C118	122.7 (2)	C218—C219—C211	122.6 (2)
C111—C119—C120	118.6 (2)	C218—C219—C220	118.5 (2)
C118—C119—C120	118.7 (2)	C211—C219—C220	118.9 (2)
C114—C120—C115	122.9 (2)	C214—C220—C215	122.5 (2)
C114—C120—C119	118.6 (2)	C214—C220—C219	118.8 (2)
C115—C120—C119	118.5 (2)	C215—C220—C219	118.8 (2)
C132—C131—C136	117.8 (2)	C232—C231—C236	118.0 (2)
C132—C131—C13	119.8 (2)	C232—C231—C23	119.3 (2)
C136—C131—C13	122.4 (2)	C236—C231—C23	122.7 (2)
C133—C132—C131	121.8 (2)	C233—C232—C231	121.3 (2)
C133—C132—H132	119.1	C233—C232—H232	119.4
C131—C132—H132	119.1	C231—C232—H232	119.4
C134—C133—C132	118.0 (2)	C234—C233—C232	118.3 (2)
C134—C133—H133	121.0	C234—C233—H233	120.9
C132—C133—H133	121.0	C232—C233—H233	120.9
F134—C134—C135	118.9 (2)	F234—C234—C235	118.8 (2)
F134—C134—C133	118.4 (3)	F234—C234—C233	118.3 (2)
C135—C134—C133	122.7 (3)	C235—C234—C233	122.8 (2)
C134—C135—C136	118.6 (2)	C234—C235—C236	118.6 (2)
C134—C135—H135	120.7	C234—C235—H235	120.7
C136—C135—H135	120.7	C236—C235—H235	120.7
C135—C136—C131	121.2 (2)	C235—C236—C231	121.0 (2)
C135—C136—H136	119.4	C235—C236—H236	119.5
C131—C136—H136	119.4	C231—C236—H236	119.5

O11—C11—C12—C13	−17.5 (4)	O21—C21—C22—C23	−13.5 (4)
C112—C11—C12—C13	162.9 (2)	C212—C21—C22—C23	167.1 (2)
C11—C12—C13—C131	178.4 (2)	C21—C22—C23—C231	178.3 (2)
C119—C111—C112—C113	1.4 (3)	C219—C211—C212—C213	1.4 (3)
C119—C111—C112—C11	178.89 (19)	C219—C211—C212—C21	−179.5 (2)
O11—C11—C112—C111	−23.9 (3)	O21—C21—C212—C211	−27.9 (3)
C12—C11—C112—C111	155.7 (2)	C22—C21—C212—C211	151.6 (2)
O11—C11—C112—C113	153.6 (2)	O21—C21—C212—C213	151.1 (2)
C12—C11—C112—C113	−26.8 (3)	C22—C21—C212—C213	−29.4 (3)
C111—C112—C113—C114	0.4 (3)	C211—C212—C213—C214	0.8 (3)
C11—C112—C113—C114	−177.0 (2)	C21—C212—C213—C214	−178.2 (2)
C112—C113—C114—C120	−1.4 (3)	C212—C213—C214—C220	−2.1 (3)
C120—C115—C116—C117	−1.0 (4)	C220—C215—C216—C217	1.6 (4)
C115—C116—C117—C118	0.3 (4)	C215—C216—C217—C218	0.1 (4)
C116—C117—C118—C119	0.1 (4)	C216—C217—C218—C219	−1.2 (4)
C112—C111—C119—C118	179.6 (2)	C217—C218—C219—C211	−178.6 (2)

C112—C111—C119—C120	−2.2 (3)	C217—C218—C219—C220	0.6 (3)
C117—C118—C119—C111	178.3 (2)	C212—C211—C219—C218	176.8 (2)
C117—C118—C119—C120	0.2 (3)	C212—C211—C219—C220	−2.3 (3)
C113—C114—C120—C115	−179.1 (2)	C213—C214—C220—C215	−178.1 (2)
C113—C114—C120—C119	0.5 (3)	C213—C214—C220—C219	1.2 (3)
C116—C115—C120—C114	−179.1 (2)	C216—C215—C220—C214	177.0 (2)
C116—C115—C120—C119	1.3 (3)	C216—C215—C220—C219	−2.2 (3)
C111—C119—C120—C114	1.2 (3)	C218—C219—C220—C214	−178.2 (2)
C118—C119—C120—C114	179.5 (2)	C211—C219—C220—C214	1.0 (3)
C111—C119—C120—C115	−179.1 (2)	C218—C219—C220—C215	1.1 (3)
C118—C119—C120—C115	−0.8 (3)	C211—C219—C220—C215	−179.7 (2)
C12—C13—C131—C132	169.7 (2)	C22—C23—C231—C232	167.2 (2)
C12—C13—C131—C136	−12.1 (4)	C22—C23—C231—C236	−13.9 (4)
C136—C131—C132—C133	0.3 (4)	C236—C231—C232—C233	1.5 (3)
C13—C131—C132—C133	178.6 (2)	C23—C231—C232—C233	−179.5 (2)
C131—C132—C133—C134	−0.8 (4)	C231—C232—C233—C234	−1.0 (4)
C132—C133—C134—F134	−178.9 (2)	C232—C233—C234—F234	−179.5 (2)
C132—C133—C134—C135	0.9 (4)	C232—C233—C234—C235	−0.4 (4)
F134—C134—C135—C136	179.2 (2)	F234—C234—C235—C236	−179.7 (2)
C133—C134—C135—C136	−0.7 (4)	C233—C234—C235—C236	1.2 (4)
C134—C135—C136—C131	0.2 (4)	C234—C235—C236—C231	−0.6 (4)
C132—C131—C136—C135	0.0 (3)	C232—C231—C236—C235	−0.7 (3)
C13—C131—C136—C135	−178.3 (2)	C23—C231—C236—C235	−179.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C132—H132···*Cg*1^{i}	0.93	2.88	3.557 (3)	131
C135—H135···*Cg*2^{ii}	0.93	2.83	3.503 (3)	130
C214—H214···*Cg*3^{ii}	0.93	2.87	3.554 (2)	131

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

(II) 3-(4-Bromophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one

Crystal data

$C_{19}H_{13}BrO$
M_r = 337.19
Triclinic, $P\overline{1}$
a = 5.8714 (5) Å
b = 7.8616 (7) Å
c = 15.5954 (13) Å
α = 95.007 (5)°
β = 90.218 (5)°
γ = 92.179 (5)°
V = 716.58 (11) Å^3

Z = 2
F(000) = 340
D_x = 1.563 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3816 reflections
θ = 1.3–29.8°
μ = 2.86 mm^{-1}
T = 296 K
Block, colourless
0.30 × 0.28 × 0.27 mm

Data collection

Bruker Kappa APEXII diffractometer
Radiation source: fine focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.451, T_{max} = 0.462
12745 measured reflections
2941 independent reflections
1842 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 26.6°, θ_{min} = 2.6°
h = −7→7
k = −9→9
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.082
S = 1.05
2941 reflections
190 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0164P)^2 + 0.6492P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5068 (6)	0.2375 (4)	0.4774 (2)	0.0415 (9)
O1	0.7111 (4)	0.2606 (3)	0.49106 (15)	0.0566 (7)
C2	0.3387 (6)	0.3049 (5)	0.5402 (2)	0.0460 (9)
H2	0.1897	0.3177	0.5220	0.055*
C3	0.3935 (6)	0.3475 (4)	0.6212 (2)	0.0407 (9)
H3	0.5422	0.3268	0.6369	0.049*
C11	0.5650 (5)	0.1296 (4)	0.3265 (2)	0.0341 (8)
H11	0.7103	0.1809	0.3314	0.041*
C12	0.4239 (5)	0.1438 (4)	0.3957 (2)	0.0348 (8)
C13	0.2048 (6)	0.0631 (5)	0.3885 (2)	0.0444 (9)
H13	0.1055	0.0734	0.4348	0.053*
C14	0.1380 (6)	−0.0292 (4)	0.3146 (2)	0.0454 (9)
H14	−0.0052	−0.0841	0.3118	0.055*
C15	0.2162 (6)	−0.1383 (4)	0.1639 (3)	0.0479 (10)
H15	0.0738	−0.1944	0.1591	0.057*
C16	0.3583 (7)	−0.1484 (5)	0.0960 (3)	0.0541 (11)
H16	0.3127	−0.2109	0.0451	0.065*
C17	0.5745 (7)	−0.0651 (5)	0.1018 (2)	0.0493 (10)
H17	0.6712	−0.0727	0.0546	0.059*
C18	0.6430 (6)	0.0265 (4)	0.1759 (2)	0.0403 (9)
H18	0.7867	0.0808	0.1792	0.048*
C19	0.4979 (5)	0.0400 (4)	0.2482 (2)	0.0334 (8)

C20	0.2798 (6)	−0.0440 (4)	0.2422 (2)	0.0376 (8)
C31	0.2512 (5)	0.4230 (4)	0.6895 (2)	0.0345 (8)
C32	0.3290 (5)	0.4328 (4)	0.7740 (2)	0.0398 (9)
H32	0.4694	0.3885	0.7856	0.048*
C33	0.2048 (5)	0.5061 (4)	0.8411 (2)	0.0402 (9)
H33	0.2596	0.5107	0.8973	0.048*
C34	−0.0016 (5)	0.5723 (4)	0.8236 (2)	0.0360 (8)
Br34	−0.17324 (8)	0.67362 (6)	0.91526 (3)	0.06341 (18)
C35	−0.0841 (5)	0.5658 (4)	0.7406 (2)	0.0387 (9)
H35	−0.2238	0.6115	0.7295	0.046*
C36	0.0415 (5)	0.4912 (4)	0.6743 (2)	0.0397 (9)
H36	−0.0149	0.4863	0.6183	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.041 (2)	0.049 (2)	0.036 (2)	0.0112 (19)	0.0044 (17)	0.0078 (18)
O1	0.0424 (16)	0.083 (2)	0.0434 (15)	0.0066 (14)	0.0013 (12)	−0.0014 (14)
C2	0.040 (2)	0.057 (3)	0.040 (2)	0.0099 (18)	0.0015 (17)	−0.0001 (19)
C3	0.036 (2)	0.048 (2)	0.039 (2)	0.0035 (17)	0.0025 (17)	0.0061 (18)
C11	0.0291 (18)	0.032 (2)	0.041 (2)	0.0014 (15)	0.0002 (16)	0.0047 (17)
C12	0.0329 (19)	0.039 (2)	0.033 (2)	0.0062 (16)	0.0026 (16)	0.0052 (17)
C13	0.038 (2)	0.050 (2)	0.047 (2)	0.0055 (18)	0.0140 (18)	0.013 (2)
C14	0.033 (2)	0.039 (2)	0.064 (3)	0.0000 (17)	0.0039 (19)	0.008 (2)
C15	0.042 (2)	0.035 (2)	0.066 (3)	0.0021 (17)	−0.017 (2)	−0.001 (2)
C16	0.069 (3)	0.044 (2)	0.049 (3)	0.014 (2)	−0.012 (2)	−0.005 (2)
C17	0.058 (3)	0.051 (2)	0.040 (2)	0.018 (2)	0.0063 (19)	0.003 (2)
C18	0.040 (2)	0.041 (2)	0.040 (2)	0.0052 (17)	0.0028 (17)	0.0006 (18)
C19	0.036 (2)	0.0306 (19)	0.034 (2)	0.0073 (16)	−0.0022 (16)	0.0044 (16)
C20	0.035 (2)	0.030 (2)	0.049 (2)	0.0027 (16)	−0.0056 (17)	0.0034 (18)
C31	0.0363 (19)	0.032 (2)	0.035 (2)	0.0004 (16)	0.0020 (16)	0.0039 (16)
C32	0.0321 (19)	0.045 (2)	0.043 (2)	0.0045 (16)	−0.0002 (17)	0.0077 (18)
C33	0.041 (2)	0.044 (2)	0.036 (2)	0.0011 (17)	−0.0035 (17)	0.0019 (17)
C34	0.0357 (19)	0.031 (2)	0.041 (2)	−0.0026 (16)	0.0027 (16)	0.0002 (17)
Br34	0.0648 (3)	0.0676 (3)	0.0562 (3)	0.0145 (2)	0.0149 (2)	−0.0101 (2)
C35	0.0284 (18)	0.041 (2)	0.048 (2)	0.0027 (16)	0.0000 (17)	0.0092 (18)
C36	0.038 (2)	0.046 (2)	0.035 (2)	0.0016 (17)	−0.0016 (16)	0.0061 (18)

Geometric parameters (Å, º)

C1—O1	1.221 (4)	C16—C17	1.405 (5)
C1—C2	1.473 (5)	C16—H16	0.9300
C1—C12	1.485 (5)	C17—C18	1.359 (5)
C2—C3	1.314 (4)	C17—H17	0.9300
C2—H2	0.9300	C18—C19	1.414 (4)
C3—C31	1.456 (4)	C18—H18	0.9300
C3—H3	0.9300	C19—C20	1.417 (4)
C11—C12	1.363 (4)	C31—C32	1.388 (4)

C11—C19	1.403 (4)	C31—C36	1.389 (4)
C11—H11	0.9300	C32—C33	1.375 (4)
C12—C13	1.413 (5)	C32—H32	0.9300
C13—C14	1.355 (5)	C33—C34	1.373 (4)
C13—H13	0.9300	C33—H33	0.9300
C14—C20	1.405 (5)	C34—C35	1.375 (4)
C14—H14	0.9300	C34—Br34	1.889 (3)
C15—C16	1.349 (5)	C35—C36	1.375 (4)
C15—C20	1.413 (5)	C35—H35	0.9300
C15—H15	0.9300	C36—H36	0.9300

O1—C1—C2	121.0 (3)	C16—C17—H17	119.9
O1—C1—C12	120.2 (3)	C17—C18—C19	120.6 (3)
C2—C1—C12	118.9 (3)	C17—C18—H18	119.7
C3—C2—C1	121.8 (3)	C19—C18—H18	119.7
C3—C2—H2	119.1	C11—C19—C18	122.3 (3)
C1—C2—H2	119.1	C11—C19—C20	118.7 (3)
C2—C3—C31	128.3 (3)	C18—C19—C20	119.0 (3)
C2—C3—H3	115.9	C14—C20—C15	123.4 (3)
C31—C3—H3	115.9	C14—C20—C19	118.2 (3)
C12—C11—C19	122.1 (3)	C15—C20—C19	118.4 (3)
C12—C11—H11	118.9	C32—C31—C36	117.6 (3)
C19—C11—H11	118.9	C32—C31—C3	119.3 (3)
C11—C12—C13	118.7 (3)	C36—C31—C3	123.1 (3)
C11—C12—C1	119.5 (3)	C33—C32—C31	122.0 (3)
C13—C12—C1	121.8 (3)	C33—C32—H32	119.0
C14—C13—C12	120.6 (3)	C31—C32—H32	119.0
C14—C13—H13	119.7	C34—C33—C32	118.8 (3)
C12—C13—H13	119.7	C34—C33—H33	120.6
C13—C14—C20	121.7 (3)	C32—C33—H33	120.6
C13—C14—H14	119.2	C33—C34—C35	121.0 (3)
C20—C14—H14	119.2	C33—C34—Br34	119.1 (3)
C16—C15—C20	121.2 (4)	C35—C34—Br34	119.9 (2)
C16—C15—H15	119.4	C36—C35—C34	119.5 (3)
C20—C15—H15	119.4	C36—C35—H35	120.2
C15—C16—C17	120.5 (4)	C34—C35—H35	120.2
C15—C16—H16	119.8	C35—C36—C31	121.1 (3)
C17—C16—H16	119.8	C35—C36—H36	119.4
C18—C17—C16	120.3 (3)	C31—C36—H36	119.4
C18—C17—H17	119.9		

O1—C1—C2—C3	−19.8 (6)	C13—C14—C20—C19	0.3 (5)
C12—C1—C2—C3	161.6 (3)	C16—C15—C20—C14	179.8 (3)
C1—C2—C3—C31	176.8 (3)	C16—C15—C20—C19	−0.2 (5)
C19—C11—C12—C13	1.1 (5)	C11—C19—C20—C14	1.9 (4)
C19—C11—C12—C1	178.8 (3)	C18—C19—C20—C14	180.0 (3)
O1—C1—C12—C11	−19.9 (5)	C11—C19—C20—C15	−178.1 (3)
C2—C1—C12—C11	158.6 (3)	C18—C19—C20—C15	0.0 (5)

O1—C1—C12—C13	157.7 (3)	C2—C3—C31—C32	169.5 (4)
C2—C1—C12—C13	−23.7 (5)	C2—C3—C31—C36	−12.4 (6)
C11—C12—C13—C14	1.2 (5)	C36—C31—C32—C33	0.3 (5)
C1—C12—C13—C14	−176.4 (3)	C3—C31—C32—C33	178.5 (3)
C12—C13—C14—C20	−1.9 (5)	C31—C32—C33—C34	−0.4 (5)
C20—C15—C16—C17	0.1 (5)	C32—C33—C34—C35	0.1 (5)
C15—C16—C17—C18	0.1 (5)	C32—C33—C34—Br34	−179.9 (2)
C16—C17—C18—C19	−0.3 (5)	C33—C34—C35—C36	0.3 (5)
C12—C11—C19—C18	179.3 (3)	Br34—C34—C35—C36	−179.7 (2)
C12—C11—C19—C20	−2.6 (5)	C34—C35—C36—C31	−0.4 (5)
C17—C18—C19—C11	178.3 (3)	C32—C31—C36—C35	0.0 (5)
C17—C18—C19—C20	0.2 (5)	C3—C31—C36—C35	−178.0 (3)
C13—C14—C20—C15	−179.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C32—H32···*Cg*4^{i}	0.93	2.85	3.513 (3)	129

Symmetry code: (i) −*x*+1, −*y*, −*z*+1.

(III) 1-(Naphthalen-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one

Crystal data

$C_{17}H_{12}OS$
M_r = 264.33
Triclinic, $P\overline{1}$
a = 5.8271 (3) Å
b = 7.4489 (4) Å
c = 15.2068 (9) Å
α = 79.745 (3)°
β = 84.427 (3)°
γ = 85.763 (3)°
V = 645.36 (6) Å^3

Z = 2
F(000) = 276
D_x = 1.360 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4247 reflections
θ = 1.4–32.6°
μ = 0.24 mm^{-1}
T = 296 K
Block, colourless
0.25 × 0.20 × 0.15 mm

Data collection

Bruker Kappa APEXII diffractometer
Radiation source: fine focus sealed tube
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.933, T_{max} = 0.965
18170 measured reflections

2694 independent reflections
2164 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26.6°, θ_{min} = 2.7°
h = −7→7
k = −9→9
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.140
S = 1.12
2694 reflections

185 parameters
15 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 0.8674P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.9718 (5)	0.2608 (4)	0.50120 (19)	0.0423 (7)	
O1	1.1716 (4)	0.2701 (4)	0.51604 (15)	0.0639 (7)	
C11	1.0710 (4)	0.3045 (3)	0.33771 (18)	0.0349 (6)	
H11	1.2055	0.3543	0.3481	0.042*	
C12	0.9152 (4)	0.2463 (3)	0.40887 (18)	0.0363 (6)	
C13	0.7111 (5)	0.1705 (4)	0.3930 (2)	0.0430 (7)	
H13	0.6026	0.1340	0.4407	0.052*	
C14	0.6715 (5)	0.1503 (4)	0.3085 (2)	0.0450 (7)	
H14	0.5383	0.0965	0.2997	0.054*	
C15	0.7924 (5)	0.1952 (4)	0.1456 (2)	0.0477 (7)	
H15	0.6608	0.1423	0.1345	0.057*	
C16	0.9447 (6)	0.2570 (5)	0.0761 (2)	0.0549 (8)	
H16	0.9177	0.2450	0.0182	0.066*	
C17	1.1423 (6)	0.3388 (4)	0.0905 (2)	0.0507 (8)	
H17	1.2449	0.3829	0.0420	0.061*	
C18	1.1860 (5)	0.3546 (4)	0.17489 (19)	0.0395 (6)	
H18	1.3192	0.4084	0.1836	0.047*	
C19	1.0318 (4)	0.2905 (3)	0.24950 (18)	0.0343 (6)	
C20	0.8275 (4)	0.2088 (3)	0.23435 (19)	0.0372 (6)	
C2	0.7821 (5)	0.2641 (4)	0.57262 (19)	0.0426 (7)	0.780 (3)
H2	0.6316	0.2953	0.5577	0.051*	0.780 (3)
C3	0.8251 (5)	0.2231 (4)	0.65741 (19)	0.0406 (6)	0.780 (3)
H3	0.9761	0.1831	0.6687	0.049*	0.780 (3)
C32	0.6634 (4)	0.2334 (3)	0.73499 (18)	0.0368 (6)	0.780 (3)
S31	0.3958 (2)	0.33969 (18)	0.72951 (8)	0.0440 (3)	0.780 (3)
C33	0.7012 (11)	0.1705 (10)	0.8224 (4)	0.0429 (13)	0.780 (3)
H33	0.8370	0.1056	0.8395	0.051*	0.780 (3)
C34	0.5200 (8)	0.2109 (16)	0.8849 (3)	0.0480 (13)	0.780 (3)
H34	0.5217	0.1784	0.9468	0.058*	0.780 (3)
C35	0.3430 (8)	0.3035 (10)	0.8425 (3)	0.0471 (12)	0.780 (3)
H35	0.2067	0.3431	0.8721	0.057*	0.780 (3)
C2A	0.7821 (5)	0.2641 (4)	0.57262 (19)	0.0426 (7)	0.220 (3)
H2A	0.6316	0.2953	0.5577	0.051*	0.220 (3)
C3A	0.8251 (5)	0.2231 (4)	0.65741 (19)	0.0406 (6)	0.220 (3)
H3A	0.9761	0.1831	0.6687	0.049*	0.220 (3)
C42	0.6634 (4)	0.2334 (3)	0.73499 (18)	0.0368 (6)	0.220 (3)

S41	0.7334 (11)	0.1491 (11)	0.8392 (3)	0.0440 (3)	0.220 (3)
C43	0.4393 (19)	0.299 (3)	0.7365 (8)	0.0440 (3)	0.220 (3)
H43	0.3681	0.3486	0.6847	0.053*	0.220 (3)
C44	0.324 (2)	0.287 (4)	0.8227 (9)	0.0471 (12)	0.220 (3)
H44	0.1725	0.3285	0.8352	0.057*	0.220 (3)
C45	0.467 (3)	0.205 (6)	0.8851 (8)	0.0480 (13)	0.220 (3)
H45	0.4230	0.1821	0.9463	0.058*	0.220 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0371 (15)	0.0436 (16)	0.0433 (16)	−0.0010 (12)	−0.0020 (12)	−0.0009 (12)
O1	0.0390 (12)	0.103 (2)	0.0475 (13)	−0.0079 (12)	−0.0034 (10)	−0.0067 (13)
C11	0.0281 (12)	0.0329 (13)	0.0441 (15)	−0.0036 (10)	−0.0003 (11)	−0.0084 (11)
C12	0.0326 (13)	0.0319 (13)	0.0430 (15)	0.0007 (10)	−0.0003 (11)	−0.0051 (11)
C13	0.0337 (14)	0.0387 (15)	0.0532 (18)	−0.0063 (11)	0.0049 (12)	−0.0018 (13)
C14	0.0321 (14)	0.0387 (15)	0.067 (2)	−0.0068 (12)	−0.0059 (13)	−0.0144 (14)
C15	0.0404 (16)	0.0443 (17)	0.064 (2)	0.0020 (13)	−0.0166 (14)	−0.0191 (15)
C16	0.058 (2)	0.060 (2)	0.0501 (19)	0.0077 (16)	−0.0144 (15)	−0.0189 (16)
C17	0.0536 (18)	0.0493 (18)	0.0467 (18)	0.0039 (14)	0.0027 (14)	−0.0077 (14)
C18	0.0372 (14)	0.0366 (14)	0.0443 (16)	−0.0015 (11)	−0.0028 (12)	−0.0065 (12)
C19	0.0299 (12)	0.0274 (12)	0.0444 (15)	0.0034 (10)	−0.0032 (11)	−0.0051 (11)
C20	0.0331 (13)	0.0291 (13)	0.0497 (16)	0.0041 (10)	−0.0043 (11)	−0.0095 (11)
C2	0.0357 (14)	0.0468 (16)	0.0445 (16)	0.0018 (12)	−0.0035 (12)	−0.0072 (13)
C3	0.0340 (14)	0.0381 (15)	0.0494 (17)	−0.0011 (11)	0.0004 (12)	−0.0096 (12)
C32	0.0355 (14)	0.0326 (13)	0.0439 (15)	−0.0065 (11)	−0.0021 (11)	−0.0096 (11)
S31	0.0347 (6)	0.0467 (8)	0.0509 (6)	0.0066 (4)	−0.0080 (4)	−0.0109 (4)
C33	0.044 (3)	0.041 (3)	0.043 (3)	0.005 (2)	−0.020 (2)	0.000 (2)
C34	0.057 (3)	0.047 (2)	0.0410 (17)	−0.015 (4)	0.0007 (17)	−0.0066 (14)
C35	0.0433 (18)	0.047 (2)	0.056 (3)	−0.0085 (17)	0.0066 (18)	−0.025 (2)
C2A	0.0357 (14)	0.0468 (16)	0.0445 (16)	0.0018 (12)	−0.0035 (12)	−0.0072 (13)
C3A	0.0340 (14)	0.0381 (15)	0.0494 (17)	−0.0011 (11)	0.0004 (12)	−0.0096 (12)
C42	0.0355 (14)	0.0326 (13)	0.0439 (15)	−0.0065 (11)	−0.0021 (11)	−0.0096 (11)
S41	0.0347 (6)	0.0467 (8)	0.0509 (6)	0.0066 (4)	−0.0080 (4)	−0.0109 (4)
C43	0.0347 (6)	0.0467 (8)	0.0509 (6)	0.0066 (4)	−0.0080 (4)	−0.0109 (4)
C44	0.0433 (18)	0.047 (2)	0.056 (3)	−0.0085 (17)	0.0066 (18)	−0.025 (2)
C45	0.057 (3)	0.047 (2)	0.0410 (17)	−0.015 (4)	0.0007 (17)	−0.0066 (14)

Geometric parameters (Å, º)

C1—O1	1.217 (3)	C19—C20	1.432 (4)
C1—C2	1.474 (4)	C2—C3	1.315 (4)
C1—C12	1.497 (4)	C2—H2	0.9300
C11—C12	1.372 (4)	C3—C32	1.446 (4)
C11—C19	1.405 (4)	C3—H3	0.9300
C11—H11	0.9300	C32—C33	1.363 (6)
C12—C13	1.413 (4)	C32—S31	1.698 (2)
C13—C14	1.364 (4)	S31—C35	1.692 (5)

C13—H13	0.9300	C33—C34	1.403 (7)
C14—C20	1.404 (4)	C33—H33	0.9300
C14—H14	0.9300	C34—C35	1.350 (5)
C15—C16	1.350 (5)	C34—H34	0.9300
C15—C20	1.407 (4)	C35—H35	0.9300
C15—H15	0.9300	S41—C45	1.695 (7)
C16—C17	1.395 (5)	C43—C44	1.404 (9)
C16—H16	0.9300	C43—H43	0.9300
C17—C18	1.359 (4)	C44—C45	1.352 (7)
C17—H17	0.9300	C44—H44	0.9300
C18—C19	1.413 (4)	C45—H45	0.9300
C18—H18	0.9300		

O1—C1—C2	121.2 (3)	C14—C20—C15	123.7 (3)
O1—C1—C12	119.8 (3)	C14—C20—C19	118.3 (3)
C2—C1—C12	118.9 (2)	C15—C20—C19	118.0 (3)
C12—C11—C19	121.5 (2)	C3—C2—C1	120.2 (3)
C12—C11—H11	119.3	C3—C2—H2	119.9
C19—C11—H11	119.3	C1—C2—H2	119.9
C11—C12—C13	119.2 (3)	C2—C3—C32	127.2 (2)
C11—C12—C1	118.5 (2)	C2—C3—H3	116.4
C13—C12—C1	122.3 (2)	C32—C3—H3	116.4
C14—C13—C12	120.6 (3)	C33—C32—C3	126.9 (3)
C14—C13—H13	119.7	C33—C32—S31	109.4 (3)
C12—C13—H13	119.7	C3—C32—S31	123.67 (19)
C13—C14—C20	121.4 (3)	C35—S31—C32	92.48 (19)
C13—C14—H14	119.3	C32—C33—C34	115.0 (5)
C20—C14—H14	119.3	C32—C33—H33	122.5
C16—C15—C20	121.7 (3)	C34—C33—H33	122.5
C16—C15—H15	119.1	C35—C34—C33	110.4 (5)
C20—C15—H15	119.1	C35—C34—H34	124.8
C15—C16—C17	120.4 (3)	C33—C34—H34	124.8
C15—C16—H16	119.8	C34—C35—S31	112.7 (4)
C17—C16—H16	119.8	C34—C35—H35	123.6
C18—C17—C16	120.4 (3)	S31—C35—H35	123.6
C18—C17—H17	119.8	C44—C43—H43	122.8
C16—C17—H17	119.8	C45—C44—C43	110.2 (8)
C17—C18—C19	120.9 (3)	C45—C44—H44	124.9
C17—C18—H18	119.6	C43—C44—H44	124.9
C19—C18—H18	119.6	C44—C45—S41	112.5 (7)
C11—C19—C18	122.5 (2)	C44—C45—H45	123.8
C11—C19—C20	118.9 (2)	S41—C45—H45	123.8
C18—C19—C20	118.6 (3)		

C19—C11—C12—C13	0.1 (4)	C16—C15—C20—C19	−0.2 (4)
C19—C11—C12—C1	178.3 (2)	C11—C19—C20—C14	1.6 (4)
O1—C1—C12—C11	−21.7 (4)	C18—C19—C20—C14	−178.1 (2)
C2—C1—C12—C11	158.1 (3)	C11—C19—C20—C15	−179.6 (2)

O1—C1—C12—C13	156.5 (3)	C18—C19—C20—C15	0.6 (4)
C2—C1—C12—C13	−23.7 (4)	O1—C1—C2—C3	−20.2 (5)
C11—C12—C13—C14	1.8 (4)	C12—C1—C2—C3	160.0 (3)
C1—C12—C13—C14	−176.3 (3)	C1—C2—C3—C32	174.8 (3)
C12—C13—C14—C20	−2.0 (4)	C2—C3—C32—C33	171.5 (5)
C20—C15—C16—C17	−0.7 (5)	C2—C3—C32—S31	−12.1 (4)
C15—C16—C17—C18	1.1 (5)	C33—C32—S31—C35	1.4 (4)
C16—C17—C18—C19	−0.6 (4)	C3—C32—S31—C35	−175.6 (3)
C12—C11—C19—C18	178.0 (2)	C3—C32—C33—C34	175.3 (6)
C12—C11—C19—C20	−1.8 (4)	S31—C32—C33—C34	−1.5 (8)
C17—C18—C19—C11	−180.0 (3)	C32—C33—C34—C35	0.9 (11)
C17—C18—C19—C20	−0.2 (4)	C33—C34—C35—S31	0.2 (10)
C13—C14—C20—C15	−178.4 (3)	C32—S31—C35—C34	−0.9 (6)
C13—C14—C20—C19	0.3 (4)	C43—C44—C45—S41	1 (4)
C16—C15—C20—C14	178.5 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H18···*Cg*5^{i}	0.93	2.80	3.572 (4)	141

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.