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Four closely related *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)benzamides: order *versus* disorder, and similar molecular conformations but different modes of supramolecular aggregation, with a new disordered refinement of 2-amino-3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophene

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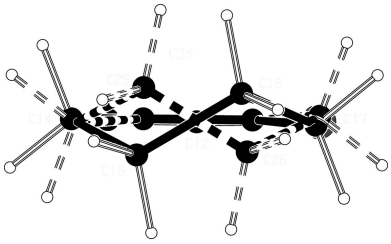
Four closely related *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)benzamides, bearing different substituents on the benzamide ring, have been synthesized and structurally characterized. In each of *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-3-fluorobenzamide, $C_{22}H_{18}FNO_2S$, (I), *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-4-chlorobenzamide, $C_{22}H_{18}ClNO_2S$, (II), *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-2,6-difluorobenzamide, $C_{22}H_{17}F_2NO_2S$, (III), and *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-2-methoxybenzamide, $C_{23}H_{21}NO_3S$, (IV), the last of which crystallizes with $Z' = 2$ in the space group $P\overline{1}$, the fused six-membered ring adopts a half-chair conformation. In each of (I)–(III), this ring is disordered over two sets of atomic sites having occupancies of 0.811 (6) and 0.189 (6) in (I), 0.645 (7) and 0.355 (7) in (II), and 0.784 (6) and 0.216 (6) in (III), such that the two disorder components of the ring are almost enantiomeric. Molecules of (I) are linked into chains by π–π stacking interactions, and those of (II) are linked into chains by a C—H$\cdots\pi$ hydrogen bond. A combination of two C—H$\cdots$O hydrogen bonds and two C—H$\cdots\pi$ hydrogen bonds links the molecules of (III) into complex sheets, but the molecules of (IV) are linked by a combination of two hydrogen bonds, one each of the C—H$\cdots$O and C—H$\cdots\pi$ types, to form centrosymmetric tetramers. The structures of (I)–(IV) are compared with that of the unsubstituted analogue *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)benzamide and a new refinement of the parent amine 2-amino-3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophene, using the original data set, has found that here too the fused six-membered ring exhibits conformational disorder, with occupancies of 0.887 (9) and 0.113 (9). Comparisons are made with some related compounds.

1. Introduction

Thiophene derivatives exhibit a wide range of biological properties, including analgesic activity (Cannito *et al.*, 1990; Molvi *et al.*, 2007), anti-inflammatory activity (Cannito *et al.*, 1990; Ashalatha *et al.*, 2007; Molvi *et al.*, 2007), antimicrobial activity (Ashalatha *et al.*, 2007; Rai *et al.*, 2008) and NO-scavenging activity (Molvi *et al.*, 2007); in addition, 2-amino-3-benzoylthiophenes can act as allosteric enhancers at the A_1 adenosine receptor (Aurelio *et al.*, 2010). With this in mind, we synthesized and characterized a series of four variously sub-



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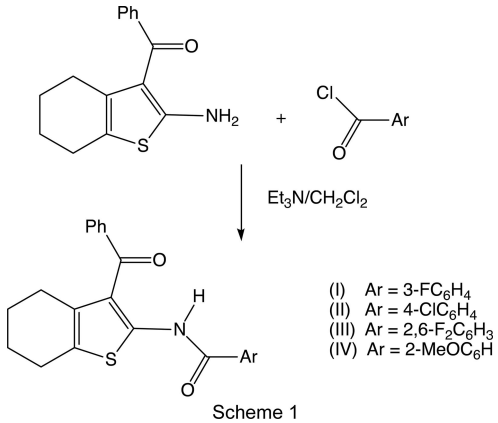
stituted *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-benzamides (Scheme 1), whose structures we report here, namely *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-3-fluorobenzamide, (I), *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo-[*b*]thiophen-2-yl)-4-chlorobenzamide, (II), *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-2,6-difluorobenzamide, (III), and *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-2-methoxybenzamide, (IV), and we compare the structures of (I)–(IV) with those of the recently reported analogue *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)benzamide, (V) (Scheme 2) (Kaur, Jasinski, Yathirajan *et al.*, 2014), and the parent amine 2-amino-3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophene, (VI) (Kubicki *et al.*, 2012).



Scheme 1

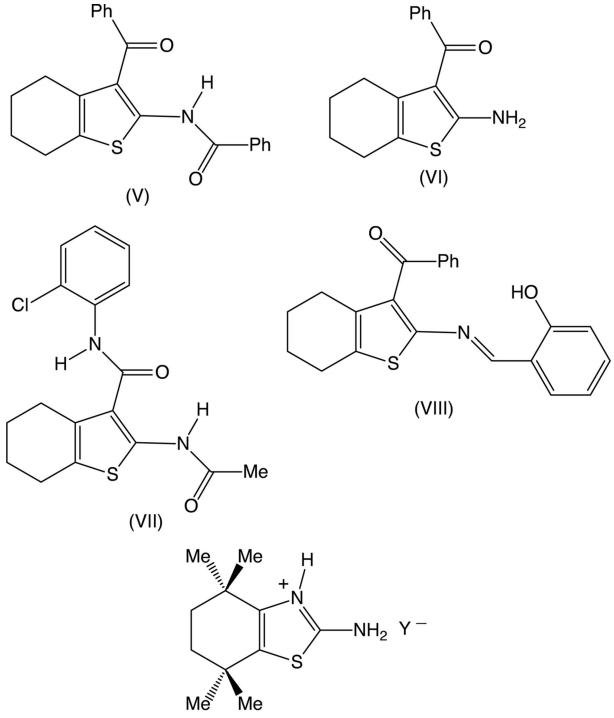
2. Experimental

2.1. Synthesis and crystallization

2-Amino-3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophene was a gift from RL Fine Chem Pvt Ltd, Bengaluru, India. All other reagents are available commercially and were used as received. For the synthesis of compounds (I)–(IV), the appropriately substituted benzoyl chloride (0.78 mmol), *viz.* 3-fluorobenzoyl chloride for (I), 4-chlorobenzoyl chloride for (II), 2,6-difluorobenzoyl chloride for (III) and 2-methoxybenzoyl chloride for (IV), was added dropwise with stirring to a mixture of 2-amino-3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophene (0.78 mmol) and triethylamine (2.34 mmol) in dichloromethane (20 ml), and the resulting solutions were stirred at 273 K for 6 h. Each solution was poured into ice-cold aqueous hydrochloric acid (1 *M*, 100 ml), and the aqueous mixtures were then exhaustively extracted with dichloromethane. The organic extracts were washed first with a saturated solution of sodium hydrogen carbonate and then with brine, before removal of the solvent under reduced pressure. 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 dimethylformamide.

2.2. Analytical and spectroscopic data

Compound (I): yield 85%, m.p. 390–391 K; IR (KBr): 3070 (NH_2), 2929, 1668 (C═O, keto), 1595 (C═O, amide), 1585, 1445, 1354, 1081; ^{1}H NMR (DMSO-d_6): δ 11.20 (*s*, NH), 7.64–7.23 (*m*, 9H), 2.68 (*dd*, *J* = 6.4, 12.0 Hz, 2H), 2.23 (*dd*, *J* = 6.0, 11.6 Hz, 2H), 1.75–1.72 (*m*, 2H), 1.60–1.57 (*m*, 2H). Compound (II): yield 87%, m.p. 428 K; IR (KBr): 3290 (NH_2), 2932, 1663 (C═O, keto), 1592 (C═O, amide), 1527, 1442, 1025, 750; ^{1}H NMR (DMSO-d_6): δ 11.31 (*s*, NH), 7.63–7.44 (*m*, 9H), 2.68 (*dd*, *J* = 5.6, 11.6 Hz, 2H), 2.18 (*dd*, *J* = 5.6, 11.6 Hz, 2H), 1.75–1.72 (*m*, 2*H*), 1.59–1.56 (*m*, 2H). Compound (III): yield 81%, m.p. 445 K; IR (KBr): 3220 (NH_2), 2930, 1682 (C═O, keto), 1623 (C═O, amide), 1528, 1462, 1251, 1051; ^{1}H NMR (DMSO-d_6): δ 11.97 (*s*, NH), 7.57–7.40 (*m*, 8H), 2.71 (*dd*, *J* = 6.4, 12.8 Hz, 2H), 1.97 (*dd*, *J* = 6.0, 12.0 Hz, 2H), 1.82–1.76 (*m*, 2*H*), 1.56–1.50 (*m*, 2H). Compound (IV): yield 88%, m.p. 447 K; IR (KBr): 3223 (NH_2), 2945, 1645 (C═O, keto), 1623 (C═O, amide), 1598, 1466, 1331, 1249, 1045; ^{1}H NMR (DMSO-d_6): δ 12.94 (*s*, NH), 7.62 (*d*, *J* = 7.6 Hz, 2H), 7.53–7.48 (*m*, 2H), 7.47–7.41 (*m*, 2H), 7.09 (*dd*, *J* = 8, 15.2 Hz, 2H), 7.01 (*d*, *J* = 8.1 Hz, 1H), 4.17 (*s*, 3H, OMe), 2.71 (*dd*, *J* = 6.4, 12.4 Hz, 2H), 1.94 (*dd*, *J* = 6, 12 Hz, 2H), 1.79–1.74 (*m*, 2H), 1.55–1.51 (*m*, 2H).



Scheme 2

2.3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. It was apparent from an early stage in the refinements of each of compounds (I)–(III) that the atoms of the –$(CH_2)_4$– fragments were disordered over two sets of sites having unequal occupancies. For the minor-disorder forms, the bonded distances and the 1,3 nonbonded distances were restrained to be the same as those in the major forms, subject to s.u. values of 0.005 and 0.01 Å, respectively. In addition, the anisotropic displacement parameters for the corresponding pairs of atoms in the two

Table 1
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{22}H_{18}FNO_2S$	$C_{22}H_{18}ClNO_2S$	$C_{22}H_{17}F_2NO_2S$
M_r	379.43	395.88	397.43
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$
Temperature (K)	296	296	296
a, b, c (Å)	15.9489 (11), 7.2739 (4), 15.9222 (10)	11.6776 (6), 14.2461 (8), 12.2957 (7)	7.6718 (3), 9.8833 (3), 13.5925 (5)
α, β, γ (°)	90, 95.813 (4), 90	90, 109.336 (2), 90	104.925 (4), 95.476 (2), 103.633 (3)
V (Å^3)	1837.6 (2)	1930.13 (18)	954.32 (6)
Z	4	4	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.20	0.32	0.21
Crystal size (mm)	0.15 × 0.10 × 0.10	0.25 × 0.20 × 0.20	0.20 × 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}	0.895, 0.980	0.882, 0.937	0.942, 0.970
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21151, 3809, 2343	28361, 3743, 2127	21940, 4375, 2814
R_{int}	0.052	0.047	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629	0.615	0.650
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.122, 1.02	0.047, 0.142, 1.03	0.041, 0.108, 1.02
No. of reflections	3809	3743	4375
No. of parameters	258	257	267
No. of restraints	12	12	12
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.20	0.30, −0.28	0.23, −0.25

	(IV)	(VI)
Crystal data		
Chemical formula	$C_{23}H_{21}NO_3S$	$C_{15}H_{15}NOS$
M_r	391.47	257.34
Crystal system, space group	Triclinic, $P\overline{1}$	Orthorhombic, $Pna2_1$
Temperature (K)	296	295
a, b, c (Å)	8.5129 (9), 12.9230 (14), 19.031 (2)	9.2080 (4), 14.0485 (7), 10.3826 (6)
α, β, γ (°)	96.811 (5), 102.665 (6), 105.321 (5)	90, 90, 90
V (Å^3)	1935.1 (4)	1343.08 (12)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.19	0.23
Crystal size (mm)	0.20 × 0.15 × 0.15	0.30 × 0.15 × 0.15
Data collection		
Diffractometer	Bruker Kappa APEXII	Agilent Eos Gemini
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*CrysAlis RED*; Agilent, 2012)
T_{min}, T_{max}	0.935, 0.972	0.948, 0.966
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37373, 8015, 4159	3556, 1984, 1720
R_{int}	0.072	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.628	0.664
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.151, 0.97	0.036, 0.086, 1.04
No. of reflections	8015	1984
No. of parameters	511	176
No. of restraints	0	13
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.36	0.13, −0.23
Absolute structure	–	Flack x determined using 422 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	–	−0.03 (6)

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

Table 2
Selected geometric parameters (Å, °) for compounds (I)–(VI).

For the major- and minor-disorder components in compounds (I)–(III) and (VI), the ring-puckering parameters are calculated for the atom sequences C13*A*–C14–C15–C16–C17–C17*A* and C13*A*–C24–C25–C26–C27–C17*A*, respectively. For molecules 1 and 2 in compound (IV), the parameters are calculated for the atom sequences C13*A*–C114–C115–C116–C117–C17*A* and C23*A*–C24–C25–C26–C27–C27*A*, respectively. The parameters for compound (V) were calculated using the published atomic coordinates (Kaur, Jasinski, Yathirajan *et al.*, 2014), but with the atom labels and the enantiomorph adjusted to match those of the major-disorder components in compounds (I)–(III) and (VI). For compounds (I)–(V), τ_1, τ_2, τ_3 and τ_4 represent, respectively, the torsion angles S*x*11—C*x*12—N*x*1—C*x*7, C*x*12—N*x*1—C*x*7—C*x*1, N*x*1—C*x*7—C*x*1—C*x*2 and C*x*13—C*x*37—C*x*31—C*x*32, where *x* is nul for (I)–(III) and (V), and *x* = 1 or 2 for molecules 1 and 2 in compound (IV). For compound (VI), τ_4 represents the torsion angle C3—C37—C31—C32.

(*a*) Ring-puckering parameters

	Q	θ	φ
(I), major	0.516 (5)	49.0 (6)	145.5 (8)
(I), minor	0.50 (2)	139 (2)	332 (4)
(II), major	0.514 (9)	48.3 (11)	150.4 (17)
(II), minor	0.538 (18)	134 (2)	330 (3)
(III), major	0.507 (5)	50.0 (6)	138.7 (8)
(III), minor	0.489 (19)	143 (2)	327 (4)
(IV), molecule 1	0.467 (3)	50.7 (4)	150.5 (5)
(IV), molecule 2	0.486 (4)	51.5 (4)	143.5 (5)
(V)	0.5098 (19)	53.3 (2)	142.2 (2)
(VI), major	0.517 (6)	49.7 (7)	148.9 (9)
(VI), minor	0.51 (3)	135 (3)	331 (6)

(*b*) Torsion angles

	τ_1	τ_2	τ_3	τ_4
(I)	0.0 (3)	179.60 (18)	−158.70 (19)	−132.4 (2)
(II)	6.6 (4)	−175.9 (3)	−170.5 (3)	−110.7 (4)
(III)	−7.3 (3)	178.76 (18)	−134.5 (2)	−147.78 (19)
(IV), molecule 1	−1.9 (3)	−176.7 (2)	5.6 (4)	−141.1 (3)
(IV), molecule 2	−2.5 (3)	−178.2 (2)	5.0 (4)	−150.8 (2)
(V)	−2.1 (2)	−179.62 (14)	−172.35 (14)	−148.30 (15)
(VI)				−137.6 (3)

disorder forms were constrained to be identical. All H atoms were treated as riding atoms in geometrically idealized positions, with C—H = 0.93 (aromatic), 0.96 (CH_3) or 0.97 Å (CH_2) and N—H = 0.86 Å, and with U_{iso}(H) = kU_{eq}(carrier), where k = 1.5 for the methyl groups in (IV), which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. Refinement of the atomic coordinates for the H atoms bonded to N atoms resulted in N—H distances ranging from 0.74 (4) to 0.90 (3) Å; hence, the riding model was preferred. Several low-angle reflections which had been attenuated by the beam stop were omitted from the refinements, *i.e.* 100 for (I), 001 for (III), and 010 and 001 for (IV). Subject to these conditions, the refined occupancies for the two disorder forms were 0.811 (6) and 0.189 (6) in (I), 0.645 (7) and 0.355 (7) in (II), and 0.784 (6) and 0.216 (6) in (III). In the final analysis of variance for (I), there was a negative value, −1.093, of K = [mean(F_o^2)/mean(F_c^2)] for the group of 443 very weak reflections having F_c/F_c(max) in the range 0.000 < F_c/F_c(max) < 0.005. In (II)–(IV), the corresponding values were −10.734 for 430 reflections in the range 0.000 < F_c/F_c(max) < 0.003, −3.962 for 481 reflections in the range 0.000 < F_c/F_c(max) < 0.005 and −6.754 for 992 reflections in the range 0.000 < F_c/F_c(max) < 0.004, respectively. The disorder in compound (VI), which was refined using the original data set (Kubicki *et al.*, 2012), was handled exactly as in compounds (I)–(III), giving refined occupancies for the two disorder components of 0.887 (9) and 0.113 (9). The correct orientation of the structure of compound (VI) with respect to the polar-axis direction was established using the Flack x parameter (Flack, 1983), calculated (Parsons *et al.*, 2013) as x = −0.03 (6) by the use of 422 quotients of the type [(I^+) − (I^-)]/[(I^+) + (I^-)].

3. Results and discussion

The reference molecules were all chosen to have the same sign for the torsion angle τ_4 (Table 2), defining the orientation of the 3-benzoyl group relative to the thiophene ring. Compound (IV) crystallizes with Z' = 2 in the space group $P\overline{1}$, but a search for possible additional crystallographic symmetry found none; it will be convenient to refer to the molecules of (IV) containing atoms S111 and S211 as molecules of types 1 and 2, respectively.

In each of (I)–(III), the –$(CH_2)_4$– portion of the fused six-membered ring exhibits disorder over two sets of sites with occupancies of 0.811 (6) and 0.189 (6) in (I), 0.645 (7) and 0.355 (7) in (II), and 0.784 (6) and 0.216 (6) in (III). This ring adopts a half-chair conformation in each of (I)–(IV) (Figs. 1–4), as shown by the ring-puckering angles (Cremer & Pople, 1975) in Table 2, as calculated using *PLATON* (Spek, 2009); for an idealized half-chair conformation, the values of the puckering angles are θ = 50.8° and φ = (60k + 30)°, where k represents an integer (Boeyens, 1978). Thus, in each of (I)–(III), the conformations of the two disorder components of this ring are nearly enantiomeric, as shown by the puckering angles, which for genuine enantiomers are related by θ' = (180 − θ) and φ' = (180 + φ) (Fig. 5). The energies of the two conformations in each compound must be very similar, and it

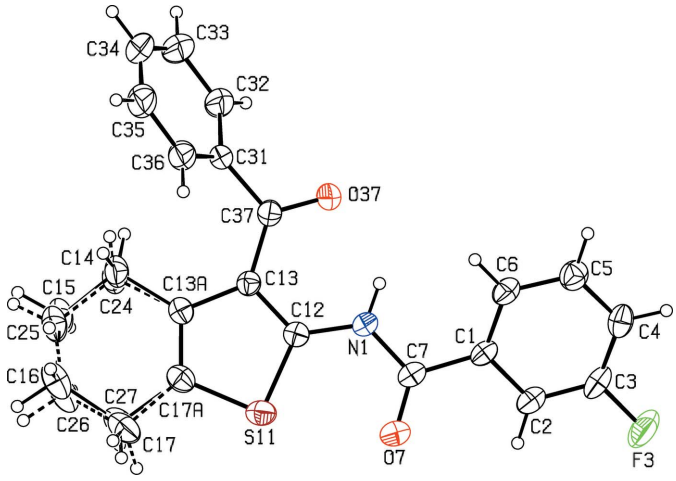


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme and the disorder in the fused six-membered ring. The major conformer is drawn with full lines, the minor conformer with broken lines and displacement ellipsoids at the 30% probability level.

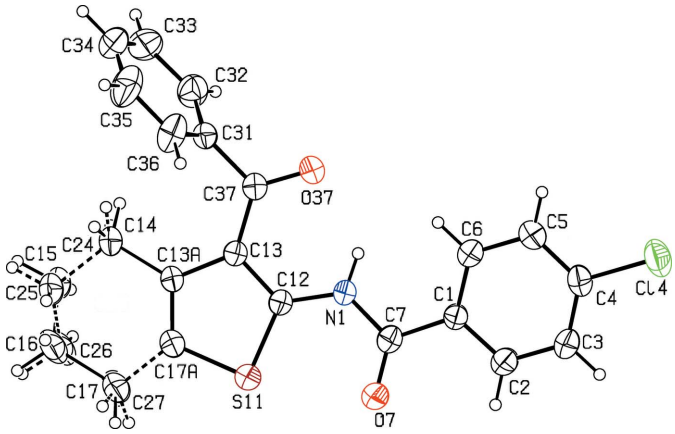


Figure 2
The molecular structure of compound (II), showing the atom-labelling scheme and the disorder in the fused six-membered ring. The major conformer is drawn with full lines, the minor conformer with broken lines and displacement ellipsoids at the 30% probability level.

is quite possible that the exact ratios of the two forms may vary slightly from one crystal to another.

Apart from the absence of disorder in compound (IV), the overall conformations of the molecules in (I)–(IV) are fairly similar (Table 2). The relative orientations of the benzoyl and thiophene units are very similar, and this may be due, at least in part, to the presence in each case of a short intramolecular N—H···O contact (Table 3). The orientations of the substituted aryl rings differ between compounds (I) and (IV), in terms of the location of the substituents (Figs. 1 and 4), which, in the case of (IV) may be controlled by a second intramolecular N—H···O contact involving the methoxy O atoms (Table 3).

The molecules of the unsubstituted analogue *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)benzamide, (V), adopt a conformation which is very similar to that found here in compounds (I)–(IV) (Table 2); although the conformation

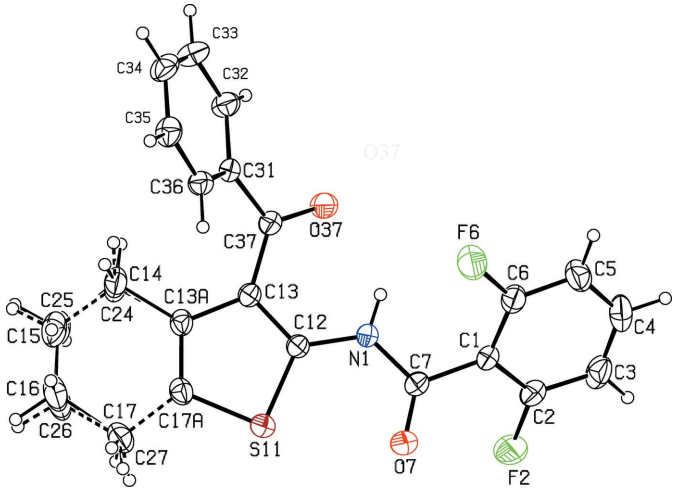


Figure 3
The molecular structure of compound (III), showing the atom-labelling scheme and the disorder in the fused six-membered ring. The major conformer is drawn with full lines, the minor conformer with broken lines and displacement ellipsoids at the 30% probability level.

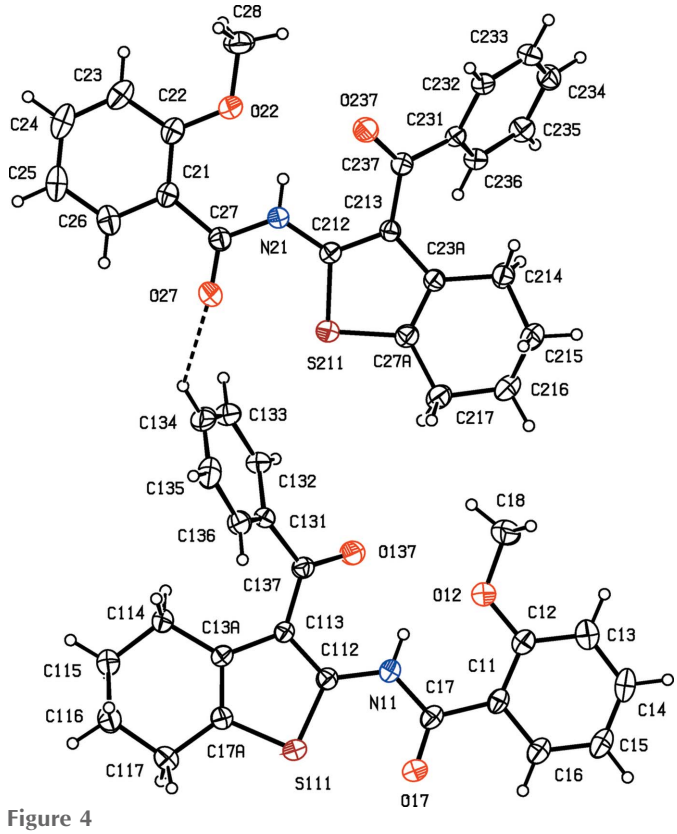


Figure 4
The structures of the two independent molecules of compound (IV), showing the atom-labelling scheme and the C—H···O hydrogen bond (dashed line) linking the two molecules within the selected asymmetric unit. Displacement ellipsoids are drawn at the 30% probability level.

adopted by the fused six-membered ring is described variously as 'envelope' and 'half-chair' in the original report (Kaur, Jasinski, Yathirajan *et al.*, 2014), the conformation is undoubtedly a half-chair form; there is no indication of any disorder in this ring.

The structures reported here all exhibit different modes of supramolecular assembly and no two exhibit the same selection of direction-specific intermolecular interactions. The

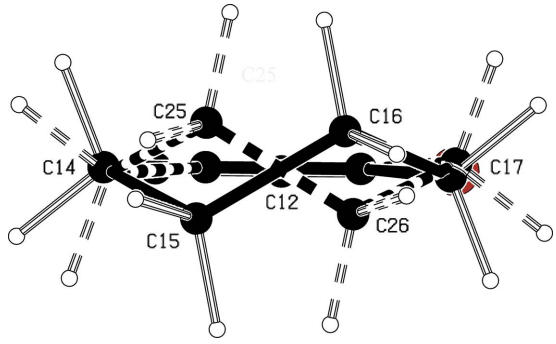


Figure 5
Part of the crystal structure of compound (II), showing the nearly enantiomeric nature of the two disorder components in the fused six-membered ring. Atoms not forming part of the benzothiophene unit have been omitted for the sake of clarity, as have many of the atom labels. The major component is drawn with full lines and the minor component is drawn with broken lines.

Table 3
Hydrogen bonds and short intramolecular contacts (Å, °) for compounds (I)–(IV) and (VI).

*Cg*1, *Cg*2 and *Cg*3 represent the centroids of the C31–C36, S11/C12/C13/C13*A*/C17*A* and S211/C212/C213/C23*A*/C27*A* rings, respectively.

Compound	*D*—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)	N1—H1···O37	0.86	2.01	2.650 (3)	130
(II)	N1—H1···O37	0.86	1.99	2.641 (3)	132
	C3—H3···*Cg*1^{i}	0.93	2.76	3.676 (4)	169
	C14—H14*A*···*Cg*1	0.97	2.96	3.475 (9)	114
	C24—H24*A*···*Cg*1	0.97	2.73	3.530 (17)	140
(III)	N1—H1···O37	0.86	2.08	2.718 (2)	130
	C3—H3···O37^{ii}	0.93	2.50	3.326 (3)	148
	C17—H17*A*···O7^{iii}	0.97	2.59	3.494 (12)	156
	C4—H4···*Cg*1^{iv}	0.93	2.85	3.635 (2)	143
	C35—H35···*Cg*2^{v}	0.93	2.75	3.520 (2)	141
(IV)	N11—H11···O12	0.86	1.95	2.630 (3)	136
	N11—H11···O137	0.86	2.11	2.731 (3)	128
	N21—H21···O22	0.86	1.93	2.616 (3)	136
	N21—H21···O237	0.86	2.13	2.748 (3)	129
	C134—H134···O27	0.93	2.49	3.232 (4)	137
	C235—H235···*Cg*3^{vi}	0.93	2.95	3.869 (3)	170
(VI)	N2—H2*A*···O37	0.86	2.07	2.666 (3)	125
	N2—H2*B*···O37^{vii}	0.86	2.03	2.831 (3)	156

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

supramolecular assembly in compound (I) depends upon two π–π stacking interactions. The thiophene ring in the molecule at (x, y, z) makes dihedral angles of 1.36 (11)° with the substituted C1–C6 phenyl rings in the two molecules at ($-x+1$, $y-\frac{1}{2}$, $-z+\frac{1}{2}$) and ($-x+1$, $y+\frac{1}{2}$, $-z+\frac{1}{2}$); the ring-centroid separations are 3.6043 (13) and 3.8362 (13) Å, respectively, and the shortest perpendicular distances from the plane of one ring in each pair to the centroid of the other are 3.5740 (9) and 3.5254 (9) Å, respectively, corresponding to ring-centroid offsets of *ca* 0.47 and 1.51 Å. The co-operative effect of these two interactions leads to the formation of a π-stacked chain running parallel to the [010] direction (Fig. 6). The supramolecular assembly in compound (II) depends on a single C—H···π(arene) hydrogen bond (Table 3), which links molecules related by the 2_1 screw axis along ($\frac{1}{4}$, y, $\frac{1}{4}$) to form a chain running parallel to [010] (Fig. 7).

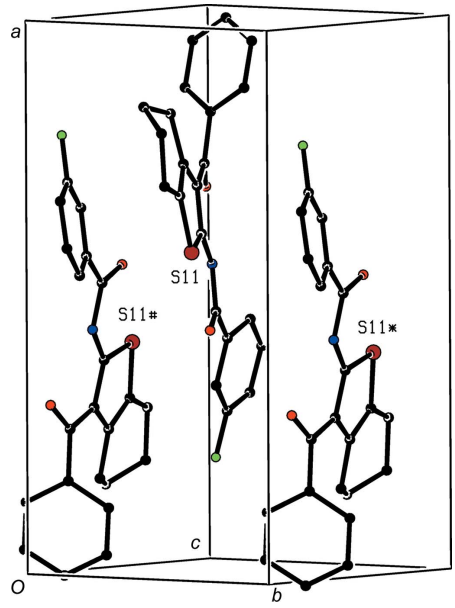


Figure 6
Part of the crystal structure of compound (I), showing the formation of a π-stacked chain parallel to [010]. The minor-disorder component and all H atoms have been omitted for the sake of clarity. S atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$) and ($-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$), respectively.

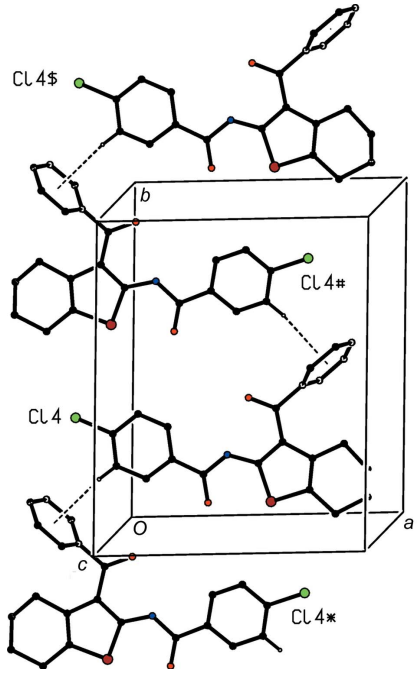


Figure 7
Part of the crystal structure of compound (II), showing the formation of a chain along [010] built from C—H···π(arene) hydrogen bonds, shown as dashed lines. The minor-disorder component and H atoms not involved in the motif shown have been omitted for the sake of clarity. Cl atoms marked with an asterisk (*), a hash (#) or a dollar sign ($) are at the symmetry positions ($-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$), ($-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$) and ($x$, $y+1$, z), respectively.

A combination of two C—H···O hydrogen bonds and two C—H···π hydrogen bonds (Table 3) links the molecules of compound (III) into complex sheets, whose formation can be analysed readily in terms of two one-dimensional substructures (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000). The two C—H···O hydrogen bonds link the molecules into a chain of centrosymmetric rings running parallel to the [001] direction, in which $R_2^2(16)$ (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) rings centred at ($\frac{1}{2}$, 0, n) alternate with $R_2^2(20)$ rings centred at ($\frac{1}{2}$, 0, $\frac{1}{2}+n$), where n represents an integer in each case (Fig. 8). In the second substructure, the two C—H···π hydrogen bonds combine to form a chain of centrosymmetric rings, in which two types of ring alternate, running parallel to the [100] direction. Rings containing two hydrogen bonds and centred at (n, 0, $\frac{1}{2}$) alternate with rings centred at ($\frac{1}{2}+n$, 0, $\frac{1}{2}$), where n represents an integer in each case (Fig. 9). The combination of these two chain motifs along [100] and [001] generates a complex sheet lying parallel to (010), but there are no direction-specific interactions between adjacent sheets.

The supramolecular assembly in compound (IV), by contrast, is very simple. A single C—H···π hydrogen bond

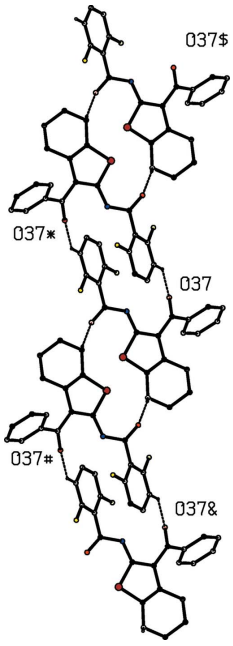


Figure 8
Part of the crystal structure of compound (III), showing the formation of a chain of centrosymmetric rings built from C—H···O hydrogen bonds (dashed lines) and running parallel to [001]. The unit-cell outline and H atoms not involved in the motifs shown have been omitted for the sake of clarity. O atoms marked with an asterisk (*), a hash (#), a dollar sign ($) or an ampersand (&) are at the symmetry positions $(-x+1, -y, -z+1)$, $(-x+1, -y, -z)$, $(x, y, z+1)$ and $(x, y, z-1)$, respectively.

links inversion related pairs of type 2 molecules into a cyclic centrosymmetric dimer, from which two inversion-related type 1 molecules are pendent, so forming a finite zero-dimensional four-molecule aggregate (Fig. 10). There are no direction-specific interactions between adjacent tetramers.

Although the original report on the structure of compound (V) (Kaur, Jasinski, Yathirajan *et al.*, 2014) noted the formation of π-stacked chains running parallel to the [010] direction, there is also, in fact, a C—H···π(arene) hydrogen bond which links inversion-related molecules into centrosymmetric dimers (Fig. 11), so that the supramolecular assembly takes the form of a chain of π-stacked hydrogen-bonded dimers along [010].

Although compounds (I)–(V) all have very similar constitutions and very similar molecular conformations, they all exhibit different patterns of intermolecular hydrogen bonds. The single donor in (II) forms part of the aryl substituent (*cf.* Scheme 1), as does one of the donors in (III), while the 3-benzoyl substituent provides donors in each of (III)–(V). The connection between molecular structure and supramolecular aggregation remains unpredictable and the details of the connection remain elusive.

It is of interest briefly to consider the structures of some related compounds. The structure of parent amine (VI) (see Scheme 2), namely 2-amino-3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophene, has been reported recently (Kubicki *et al.*, 2012) and the anisotropic displacement parameters suggest that there is disorder in the fused six-membered ring which was not accounted for in the original refinement. Accordingly, we undertook a new refinement of this structure, using the original data set, and found that there is indeed disorder in this ring, exactly comparable to the disorder found in compounds (I)–(III), with the two conformations having occupancies of 0.887 (9) and 0.113 (9) (Fig. 12 and Table 2).

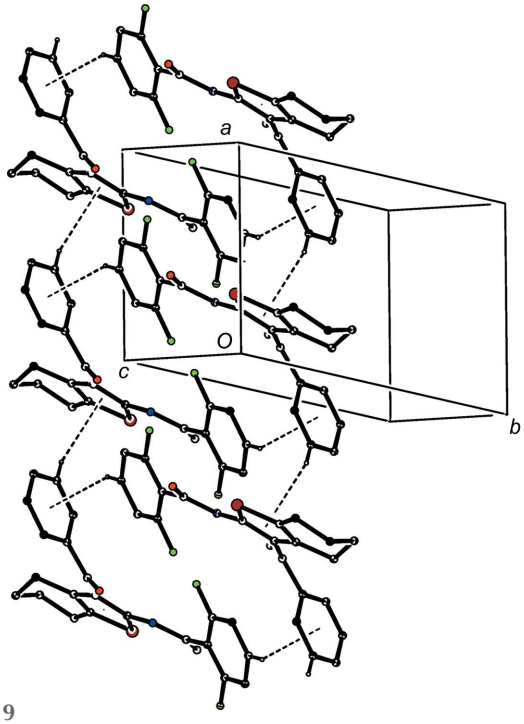


Figure 9
Part of the crystal structure of compound (III), showing the formation of a chain of centrosymmetric rings built from C—H···π hydrogen bonds (dashed lines) and running parallel to [100]. H atoms not involved in the motifs shown have been omitted for the sake of clarity.

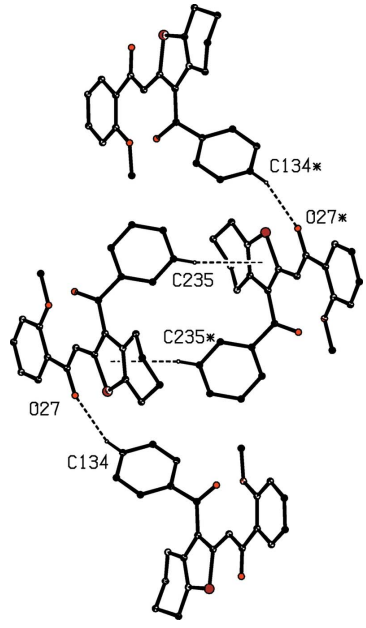


Figure 10
Part of the crystal structure of compound (IV), showing the formation of a centrosymmetric tetramer built from C—H···π and C—H···O hydrogen bonds (dashed lines) and running parallel to [100]. The unit-cell outline and H atoms not involved in the motifs shown have been omitted for the sake of clarity.

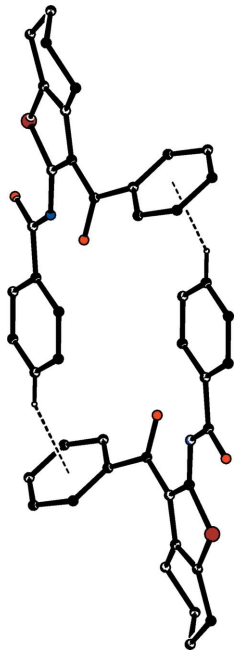


Figure 11
Part of the crystal structure of compound (V), showing the formation of a hydrogen-bonded dimer (dashed lines) containing the molecules at (x, y, z) and ($-x+2$, $-y$, $-z+1$). The original atomic coordinates (Kaur, Jasinski, Yathirajan *et al.*, 2014) have been used and, for the sake of clarity, the unit-cell outline and H atoms not involved in the motif shown have been omitted.

Crystal structures have also been described for compounds (VII) (Kumar *et al.*, 2005) and (VIII) (Kaur, Jasinski, Kavitha *et al.*, 2014) (Scheme 2). In each of these compounds, the fused six-membered ring adopts a half-chair conformation, as in (I)–(VI) above, but with no evidence for conformational disorder in either of them. The orientation of the 3-benzoyl group in (VIII), however, differs from that in (I)–(VI) by a rotation of approximately 180° about the exocyclic C—C bond, supporting the idea that the intramolecular N—H···O interaction in (I)–(VI) has a role in determining the orientation of this substituent. In each of salts (IX) and (X), the fused six-membered ring again adopts a half-chair conformation (Sagar *et al.*, 2017). Both half-chair rings exhibit conformational disorder, but with different proportions for the two conformers, *i.e.* 0.721 (5) and 0.279 (5) in (IX), and 0.575 (4) and 0.425 (4) in (X).

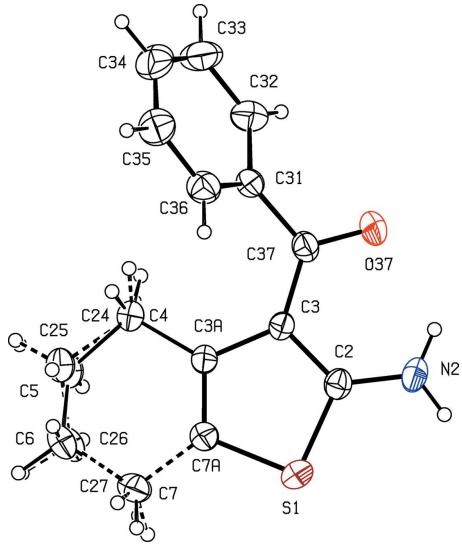


Figure 12
The molecular structure of compound (VI), showing the atom-labelling scheme and the disorder in the fused six-membered ring; the structure was refined using the original data set (Kubicki *et al.*, 2012). The major conformer is drawn with full lines, the minor conformer with broken lines and displacement ellipsoids at the 30% probability level.

It is somewhat disappointing to note that structures with unmodelled disorder, such as compound (VI) (Kubicki *et al.*, 2012), are still being published and thus incorporated into the Cambridge Structural Database (Groom *et al.*, 2016). In this connection, we have recently noted (Rajam *et al.*, 2017) an example of unmodelled conformational disorder involving a simple thiophene group (Rajam *et al.*, 2015). While enantiomeric and positional disorder are usually easy to detect, although not always straightforward to unravel in cases of multiple disorder across special positions (Zakaria *et al.*, 2003; Cobo *et al.*, 2009), the detection of conformational disorder usually requires careful inspection of the refined bond distances and anisotropic displacement parameters, as well as attention to any unexpected features in the difference maps.

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supporting information

Acta Cryst. (2018). C**74**, 45-53 [https://doi.org/10.1107/S2053229617017326]

Four closely related *N*-(3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)benzamides: order *versus* disorder, and similar molecular conformations but different modes of supramolecular aggregation, with a new disordered refinement of 2-amino-3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophene

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

Computing details

Data collection: *APEX2* (Bruker, 2012) for (I), (II), (III), (IV); *CrysAlis PRO* (Agilent, 2012) for (VI). Cell refinement: *APEX2* (Bruker, 2012) for (I), (II), (III), (IV); *CrysAlis PRO* (Agilent, 2012) for (VI). Data reduction: *SAINT-Plus* (Bruker, 2012) for (I), (II), (III), (IV); *CrysAlis RED* (Agilent, 2012) for (VI). For all structures, 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).

N-(3-Benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-3-fluorobenzamide (I)

Crystal data

$C_{22}H_{18}FNO_2S$
M_r = 379.43
Monoclinic, $P2_1/c$
a = 15.9489 (11) Å
b = 7.2739 (4) Å
c = 15.9222 (10) Å
β = 95.813 (4)°
V = 1837.6 (2) Å^3
Z = 4
$F(000)$ = 792
D_x = 1.371 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5442 reflections
θ = 1.3–30.3°
μ = 0.20 mm^{-1}
T = 296 K
Block, colourless
0.15 × 0.10 × 0.10 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} = 0.895, T_{max} = 0.980
21151 measured reflections
3809 independent reflections
2343 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 26.6°, θ_{min} = 2.6°
h = −20→20
k = −9→9
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.122$
$S = 1.02$
3809 reflections
258 parameters
12 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.4981P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0080 (9)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C7	0.47949 (13)	0.6101 (3)	0.23133 (15)	0.0465 (6)	
O7	0.44771 (10)	0.6398 (3)	0.15993 (11)	0.0692 (5)	
C1	0.42894 (13)	0.6016 (3)	0.30528 (14)	0.0429 (5)	
C2	0.34324 (14)	0.5665 (3)	0.28845 (16)	0.0532 (6)	
H2	0.3192	0.5467	0.2335	0.064*	
C3	0.29523 (14)	0.5618 (3)	0.35488 (19)	0.0583 (7)	
F3	0.21131 (9)	0.5261 (3)	0.33825 (12)	0.0938 (6)	
C4	0.32656 (16)	0.5938 (3)	0.43705 (18)	0.0589 (7)	
H4	0.2920	0.5897	0.4806	0.071*	
C5	0.41083 (15)	0.6321 (3)	0.45229 (17)	0.0587 (7)	
H5	0.4341	0.6563	0.5071	0.070*	
C6	0.46162 (14)	0.6352 (3)	0.38704 (15)	0.0514 (6)	
H6	0.5188	0.6603	0.3986	0.062*	
N1	0.56415 (10)	0.5827 (2)	0.24788 (11)	0.0451 (5)	
H1	0.5837	0.5629	0.2994	0.054*	
S11	0.59028 (4)	0.62223 (8)	0.08302 (4)	0.0539 (2)	
C12	0.62066 (14)	0.5844 (3)	0.18772 (13)	0.0409 (5)	
C13	0.70640 (13)	0.5546 (3)	0.20427 (13)	0.0395 (5)	
C13A	0.74750 (14)	0.5566 (3)	0.12712 (14)	0.0434 (5)	0.811 (6)
C14	0.8387 (2)	0.5147 (13)	0.1187 (2)	0.0586 (11)	0.811 (6)
H14A	0.8575	0.4166	0.1574	0.070*	0.811 (6)
H14B	0.8726	0.6227	0.1339	0.070*	0.811 (6)
C15	0.8513 (2)	0.4569 (6)	0.0284 (2)	0.0694 (12)	0.811 (6)
H15A	0.9110	0.4503	0.0218	0.083*	0.811 (6)
H15B	0.8269	0.3362	0.0168	0.083*	0.811 (6)
C16	0.8092 (3)	0.5961 (6)	−0.0331 (2)	0.0761 (13)	0.811 (6)
H16A	0.8227	0.5677	−0.0897	0.091*	0.811 (6)
H16B	0.8307	0.7179	−0.0186	0.091*	0.811 (6)

C17	0.7135 (3)	0.5940 (16)	−0.0311 (2)	0.0714 (10)	0.811 (6)
H17A	0.6889	0.7017	−0.0599	0.086*	0.811 (6)
H17B	0.6899	0.4857	−0.0601	0.086*	0.811 (6)
C17A	0.69285 (15)	0.5931 (3)	0.05883 (14)	0.0506 (6)	0.811 (6)
C23A	0.74750 (14)	0.5566 (3)	0.12712 (14)	0.0434 (5)	0.189 (6)
C24	0.8348 (9)	0.495 (6)	0.1092 (7)	0.0586 (11)	0.189 (6)
H24A	0.8384	0.3624	0.1134	0.070*	0.189 (6)
H24B	0.8762	0.5469	0.1516	0.070*	0.189 (6)
C25	0.8558 (7)	0.555 (3)	0.0216 (8)	0.0694 (12)	0.189 (6)
H25A	0.8645	0.6867	0.0213	0.083*	0.189 (6)
H25B	0.9078	0.4961	0.0093	0.083*	0.189 (6)
C26	0.7861 (8)	0.505 (2)	−0.0465 (7)	0.0761 (13)	0.189 (6)
H26A	0.7748	0.3736	−0.0447	0.091*	0.189 (6)
H26B	0.8034	0.5337	−0.1017	0.091*	0.189 (6)
C27	0.7063 (13)	0.613 (7)	−0.0321 (6)	0.0714 (10)	0.189 (6)
H27A	0.7132	0.7411	−0.0461	0.086*	0.189 (6)
H27B	0.6583	0.5642	−0.0675	0.086*	0.189 (6)
C27A	0.69285 (15)	0.5931 (3)	0.05883 (14)	0.0506 (6)	0.189 (6)
C37	0.74366 (14)	0.5196 (3)	0.29015 (14)	0.0456 (5)	
O37	0.69952 (10)	0.4827 (3)	0.34774 (10)	0.0662 (5)	
C31	0.83655 (14)	0.5287 (3)	0.31342 (14)	0.0490 (6)	
C32	0.87614 (17)	0.3885 (4)	0.36015 (17)	0.0771 (9)	
H32	0.8457	0.2854	0.3735	0.093*	
C33	0.9610 (2)	0.4007 (6)	0.3872 (2)	0.1091 (13)	
H33	0.9878	0.3052	0.4182	0.131*	
C34	1.0054 (2)	0.5528 (7)	0.3686 (2)	0.1112 (14)	
H34	1.0625	0.5604	0.3869	0.133*	
C35	0.96709 (19)	0.6936 (6)	0.3235 (2)	0.0901 (11)	
H35	0.9978	0.7974	0.3116	0.108*	
C36	0.88298 (16)	0.6819 (4)	0.29559 (16)	0.0633 (7)	
H36	0.8569	0.7779	0.2644	0.076*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C7	0.0376 (13)	0.0436 (13)	0.0566 (15)	0.0011 (10)	−0.0027 (11)	0.0005 (11)
O7	0.0486 (10)	0.0989 (14)	0.0582 (11)	0.0068 (10)	−0.0046 (8)	0.0119 (10)
C1	0.0333 (12)	0.0363 (12)	0.0578 (14)	0.0029 (9)	−0.0017 (10)	−0.0025 (10)
C2	0.0380 (13)	0.0541 (14)	0.0658 (16)	0.0034 (11)	−0.0026 (12)	−0.0075 (12)
C3	0.0309 (13)	0.0537 (15)	0.091 (2)	0.0032 (11)	0.0084 (13)	−0.0069 (14)
F3	0.0362 (8)	0.1247 (14)	0.1210 (14)	−0.0057 (9)	0.0103 (9)	−0.0185 (11)
C4	0.0507 (15)	0.0548 (15)	0.0740 (18)	0.0080 (12)	0.0201 (14)	−0.0036 (13)
C5	0.0536 (16)	0.0597 (16)	0.0629 (16)	0.0043 (13)	0.0067 (13)	−0.0094 (12)
C6	0.0373 (13)	0.0534 (14)	0.0625 (16)	−0.0019 (11)	0.0002 (11)	−0.0063 (12)
N1	0.0354 (10)	0.0538 (11)	0.0451 (11)	−0.0001 (8)	−0.0001 (8)	−0.0001 (9)
S11	0.0551 (4)	0.0550 (4)	0.0494 (4)	−0.0035 (3)	−0.0051 (3)	0.0073 (3)
C12	0.0440 (13)	0.0354 (11)	0.0428 (12)	−0.0034 (9)	0.0029 (10)	−0.0009 (9)
C13	0.0385 (12)	0.0385 (12)	0.0415 (12)	−0.0032 (9)	0.0043 (10)	−0.0021 (9)

C13A	0.0491 (14)	0.0389 (12)	0.0430 (13)	−0.0039 (10)	0.0083 (11)	−0.0010 (10)
C14	0.0567 (17)	0.069 (2)	0.0538 (17)	0.0025 (14)	0.0213 (14)	−0.0051 (17)
C15	0.072 (2)	0.081 (3)	0.0596 (19)	−0.006 (2)	0.0288 (16)	−0.013 (2)
C16	0.105 (3)	0.073 (3)	0.056 (2)	−0.006 (2)	0.033 (2)	0.008 (2)
C17	0.091 (2)	0.079 (3)	0.0439 (15)	−0.0127 (19)	0.0082 (14)	0.0074 (14)
C17A	0.0610 (15)	0.0470 (13)	0.0438 (13)	−0.0063 (11)	0.0054 (11)	0.0013 (11)
C23A	0.0491 (14)	0.0389 (12)	0.0430 (13)	−0.0039 (10)	0.0083 (11)	−0.0010 (10)
C24	0.0567 (17)	0.069 (2)	0.0538 (17)	0.0025 (14)	0.0213 (14)	−0.0051 (17)
C25	0.072 (2)	0.081 (3)	0.0596 (19)	−0.006 (2)	0.0288 (16)	−0.013 (2)
C26	0.105 (3)	0.073 (3)	0.056 (2)	−0.006 (2)	0.033 (2)	0.008 (2)
C27	0.091 (2)	0.079 (3)	0.0439 (15)	−0.0127 (19)	0.0082 (14)	0.0074 (14)
C27A	0.0610 (15)	0.0470 (13)	0.0438 (13)	−0.0063 (11)	0.0054 (11)	0.0013 (11)
C37	0.0417 (13)	0.0507 (13)	0.0449 (13)	−0.0004 (11)	0.0069 (11)	−0.0020 (11)
O37	0.0436 (10)	0.1099 (15)	0.0458 (10)	−0.0053 (9)	0.0081 (8)	0.0106 (9)
C31	0.0402 (13)	0.0706 (16)	0.0364 (12)	0.0043 (12)	0.0056 (10)	0.0010 (11)
C32	0.0546 (17)	0.109 (2)	0.0678 (18)	0.0135 (17)	0.0090 (14)	0.0293 (17)
C33	0.060 (2)	0.183 (4)	0.084 (2)	0.036 (2)	0.0046 (18)	0.044 (3)
C34	0.0412 (18)	0.220 (5)	0.072 (2)	0.003 (2)	0.0046 (16)	0.001 (3)
C35	0.0554 (19)	0.147 (3)	0.068 (2)	−0.031 (2)	0.0091 (16)	−0.020 (2)
C36	0.0523 (16)	0.0833 (19)	0.0546 (15)	−0.0114 (14)	0.0066 (12)	−0.0084 (14)

Geometric parameters (Å, º)

C7—O7	1.216 (3)	C16—H16A	0.9700
C7—N1	1.364 (3)	C16—H16B	0.9700
C7—C1	1.494 (3)	C17—C17A	1.502 (3)
C1—C6	1.374 (3)	C17—H17A	0.9700
C1—C2	1.390 (3)	C17—H17B	0.9700
C2—C3	1.368 (3)	C24—C25	1.529 (7)
C2—H2	0.9300	C24—H24A	0.9700
C3—F3	1.363 (3)	C24—H24B	0.9700
C3—C4	1.372 (4)	C25—C26	1.517 (6)
C4—C5	1.370 (3)	C25—H25A	0.9700
C4—H4	0.9300	C25—H25B	0.9700
C5—C6	1.381 (3)	C26—C27	1.532 (8)
C5—H5	0.9300	C26—H26A	0.9700
C6—H6	0.9300	C26—H26B	0.9700
N1—C12	1.380 (3)	C27—H27A	0.9700
N1—H1	0.8600	C27—H27B	0.9700
S11—C12	1.711 (2)	C37—O37	1.241 (2)
S11—C17A	1.731 (2)	C37—C31	1.492 (3)
C12—C13	1.383 (3)	C31—C32	1.377 (3)
C13—C13A	1.450 (3)	C31—C36	1.383 (3)
C13—C37	1.457 (3)	C32—C33	1.380 (4)
C13A—C17A	1.349 (3)	C32—H32	0.9300
C13A—C14	1.505 (3)	C33—C34	1.363 (5)
C14—C15	1.531 (5)	C33—H33	0.9300
C14—H14A	0.9700	C34—C35	1.360 (5)

C14—H14B	0.9700	C34—H34	0.9300
C15—C16	1.517 (5)	C35—C36	1.372 (4)
C15—H15A	0.9700	C35—H35	0.9300
C15—H15B	0.9700	C36—H36	0.9300
C16—C17	1.530 (7)		

O7—C7—N1	121.2 (2)	C17—C16—H16B	109.5
O7—C7—C1	122.5 (2)	H16A—C16—H16B	108.0
N1—C7—C1	116.4 (2)	C17A—C17—C16	109.6 (4)
C6—C1—C2	119.1 (2)	C17A—C17—H17A	109.8
C6—C1—C7	123.8 (2)	C16—C17—H17A	109.8
C2—C1—C7	117.0 (2)	C17A—C17—H17B	109.8
C3—C2—C1	118.2 (2)	C16—C17—H17B	109.8
C3—C2—H2	120.9	H17A—C17—H17B	108.2
C1—C2—H2	120.9	C13A—C17A—C17	125.7 (2)
F3—C3—C2	118.0 (2)	C13A—C17A—S11	113.12 (17)
F3—C3—C4	118.2 (2)	C17—C17A—S11	121.0 (2)
C2—C3—C4	123.7 (2)	C25—C24—H24A	109.2
C5—C4—C3	117.3 (2)	C25—C24—H24B	109.2
C5—C4—H4	121.3	H24A—C24—H24B	107.9
C3—C4—H4	121.3	C26—C25—C24	111.9 (8)
C4—C5—C6	120.6 (2)	C26—C25—H25A	109.2
C4—C5—H5	119.7	C24—C25—H25A	109.2
C6—C5—H5	119.7	C26—C25—H25B	109.2
C1—C6—C5	121.0 (2)	C24—C25—H25B	109.2
C1—C6—H6	119.5	H25A—C25—H25B	107.9
C5—C6—H6	119.5	C25—C26—C27	109.2 (9)
C7—N1—C12	124.59 (19)	C25—C26—H26A	109.8
C7—N1—H1	117.7	C27—C26—H26A	109.8
C12—N1—H1	117.7	C25—C26—H26B	109.8
C12—S11—C17A	90.94 (11)	C27—C26—H26B	109.8
N1—C12—C13	124.67 (19)	H26A—C26—H26B	108.3
N1—C12—S11	122.51 (17)	C26—C27—H27A	110.2
C13—C12—S11	112.82 (16)	C26—C27—H27B	110.2
C12—C13—C13A	111.11 (19)	H27A—C27—H27B	108.5
C12—C13—C37	120.50 (18)	O37—C37—C13	121.6 (2)
C13A—C13—C37	128.3 (2)	O37—C37—C31	116.7 (2)
C17A—C13A—C13	112.0 (2)	C13—C37—C31	121.72 (19)
C17A—C13A—C14	121.3 (2)	C32—C31—C36	118.8 (2)
C13—C13A—C14	126.7 (2)	C32—C31—C37	119.4 (2)
C13A—C14—C15	111.0 (3)	C36—C31—C37	121.6 (2)
C13A—C14—H14A	109.4	C31—C32—C33	120.1 (3)
C15—C14—H14A	109.4	C31—C32—H32	119.9
C13A—C14—H14B	109.4	C33—C32—H32	119.9
C15—C14—H14B	109.4	C34—C33—C32	119.9 (3)
H14A—C14—H14B	108.0	C34—C33—H33	120.0
C16—C15—C14	109.4 (4)	C32—C33—H33	120.0
C16—C15—H15A	109.8	C35—C34—C33	120.7 (3)

C14—C15—H15A	109.8	C35—C34—H34	119.6
C16—C15—H15B	109.8	C33—C34—H34	119.6
C14—C15—H15B	109.8	C34—C35—C36	119.7 (3)
H15A—C15—H15B	108.2	C34—C35—H35	120.1
C15—C16—C17	110.9 (5)	C36—C35—H35	120.1
C15—C16—H16A	109.5	C35—C36—C31	120.6 (3)
C17—C16—H16A	109.5	C35—C36—H36	119.7
C15—C16—H16B	109.5	C31—C36—H36	119.7

O7—C7—C1—C6	−155.4 (2)	C13—C13A—C14—C15	157.6 (4)
N1—C7—C1—C6	24.6 (3)	C13A—C14—C15—C16	50.1 (9)
O7—C7—C1—C2	21.3 (3)	C14—C15—C16—C17	−65.8 (9)
N1—C7—C1—C2	−158.70 (19)	C15—C16—C17—C17A	45.8 (10)
C6—C1—C2—C3	−1.8 (3)	C13—C13A—C17A—C17	−176.0 (6)
C7—C1—C2—C3	−178.6 (2)	C14—C13A—C17A—C17	1.1 (9)
C1—C2—C3—F3	−179.54 (19)	C13—C13A—C17A—S11	−1.1 (2)
C1—C2—C3—C4	1.5 (4)	C14—C13A—C17A—S11	176.0 (5)
F3—C3—C4—C5	−179.1 (2)	C16—C17—C17A—C13A	−14.3 (11)
C2—C3—C4—C5	−0.1 (4)	C16—C17—C17A—S11	171.1 (4)
C3—C4—C5—C6	−0.9 (4)	C12—S11—C17A—C13A	−0.08 (18)
C2—C1—C6—C5	0.8 (3)	C12—S11—C17A—C17	175.1 (6)
C7—C1—C6—C5	177.4 (2)	C24—C25—C26—C27	64.7 (15)
C4—C5—C6—C1	0.6 (4)	C12—C13—C37—O37	13.2 (3)
O7—C7—N1—C12	−0.4 (3)	C13A—C13—C37—O37	−164.1 (2)
C1—C7—N1—C12	179.60 (18)	C12—C13—C37—C31	−166.0 (2)
C7—N1—C12—C13	−179.27 (19)	C13A—C13—C37—C31	16.7 (3)
C7—N1—C12—S11	0.0 (3)	O37—C37—C31—C32	48.4 (3)
C17A—S11—C12—N1	−178.05 (17)	C13—C37—C31—C32	−132.4 (2)
C17A—S11—C12—C13	1.32 (16)	O37—C37—C31—C36	−125.9 (2)
N1—C12—C13—C13A	177.20 (17)	C13—C37—C31—C36	53.4 (3)
S11—C12—C13—C13A	−2.2 (2)	C36—C31—C32—C33	−1.0 (4)
N1—C12—C13—C37	−0.5 (3)	C37—C31—C32—C33	−175.5 (3)
S11—C12—C13—C37	−179.90 (16)	C31—C32—C33—C34	0.8 (5)
C12—C13—C13A—C17A	2.1 (3)	C32—C33—C34—C35	0.1 (6)
C37—C13—C13A—C17A	179.6 (2)	C33—C34—C35—C36	−0.7 (5)
C12—C13—C13A—C14	−174.8 (5)	C34—C35—C36—C31	0.5 (4)
C37—C13—C13A—C14	2.7 (6)	C32—C31—C36—C35	0.4 (4)
C17A—C13A—C14—C15	−19.0 (9)	C37—C31—C36—C35	174.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O37	0.86	2.01	2.650 (3)	130

N-(3-Benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-4-chlorobenzamide (II)

Crystal data

$C_{22}H_{18}ClNO_2S$
$M_r = 395.88$
Monoclinic, $P2_1/n$
$a = 11.6776$ (6) Å
$b = 14.2461$ (8) Å
$c = 12.2957$ (7) Å
$\beta = 109.336$ (2)°
$V = 1930.13$ (18) Å^3
$Z = 4$
$F(000) = 824$
$D_x = 1.362$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3743 reflections
$\theta = 2.3$–$25.9°$
$\mu = 0.32$ mm^{-1}
$T = 296$ K
Block, colourless
0.25 × 0.20 × 0.20 mm

Data collection

Bruker Kappa APEXII diffractometer
Radiation source: fine focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
$T_{min} = 0.882$, $T_{max} = 0.937$
28361 measured reflections
3743 independent reflections
2127 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 25.9°$, $\theta_{min} = 2.3°$
$h = -13 \rightarrow 14$
$k = -17 \rightarrow 17$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.142$
$S = 1.03$
3743 reflections
257 parameters
12 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 1.4106P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ 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)
C7	0.3052 (3)	0.1590 (2)	0.3054 (3)	0.0537 (8)	
O7	0.32402 (18)	0.07451 (16)	0.3102 (2)	0.0725 (7)	
C1	0.1822 (2)	0.1999 (2)	0.2546 (2)	0.0489 (7)	
C2	0.0837 (3)	0.1403 (2)	0.2274 (3)	0.0650 (9)	
H2	0.0960	0.0763	0.2420	0.078*	
C3	−0.0329 (3)	0.1740 (2)	0.1790 (3)	0.0677 (9)	
H3	−0.0988	0.1333	0.1613	0.081*	
C4	−0.0506 (3)	0.2678 (2)	0.1575 (3)	0.0594 (8)	
Cl4	−0.19694 (8)	0.31045 (8)	0.09347 (9)	0.0892 (4)	
C5	0.0452 (3)	0.3286 (2)	0.1820 (3)	0.0653 (9)	

H5	0.0321	0.3924	0.1663	0.078*	
C6	0.1611 (3)	0.2943 (2)	0.2301 (3)	0.0623 (9)	
H6	0.2265	0.3354	0.2466	0.075*	
N1	0.39838 (19)	0.22082 (17)	0.3469 (2)	0.0533 (6)	
H1	0.3796	0.2793	0.3453	0.064*	
S11	0.57075 (7)	0.08612 (6)	0.41343 (8)	0.0635 (3)	
C12	0.5191 (2)	0.1988 (2)	0.3909 (2)	0.0490 (7)	
C13	0.6120 (2)	0.2646 (2)	0.4221 (3)	0.0494 (7)	
C13A	0.7289 (2)	0.2195 (2)	0.4669 (3)	0.0508 (7)	0.645 (7)
C14	0.8512 (4)	0.2670 (4)	0.512 (2)	0.0704 (12)	0.645 (7)
H14A	0.8458	0.3215	0.5569	0.084*	0.645 (7)
H14B	0.8748	0.2883	0.4470	0.084*	0.645 (7)
C15	0.9492 (4)	0.1985 (4)	0.5870 (6)	0.073 (2)	0.645 (7)
H15A	1.0291	0.2258	0.6030	0.088*	0.645 (7)
H15B	0.9359	0.1881	0.6599	0.088*	0.645 (7)
C16	0.9424 (5)	0.1075 (5)	0.5256 (8)	0.098 (4)	0.645 (7)
H16A	1.0097	0.0678	0.5684	0.117*	0.645 (7)
H16B	0.9487	0.1187	0.4500	0.117*	0.645 (7)
C17	0.8221 (5)	0.0579 (3)	0.513 (2)	0.0762 (16)	0.645 (7)
H17A	0.8114	0.0051	0.4606	0.091*	0.645 (7)
H17B	0.8232	0.0341	0.5871	0.091*	0.645 (7)
C17A	0.7193 (3)	0.1256 (2)	0.4670 (3)	0.0577 (8)	0.645 (7)
C23A	0.7289 (2)	0.2195 (2)	0.4669 (3)	0.0508 (7)	0.355 (7)
C24	0.8530 (6)	0.2643 (5)	0.517 (4)	0.0704 (12)	0.355 (7)
H24B	0.8638	0.2872	0.5941	0.084*	0.355 (7)
H24A	0.8594	0.3173	0.4699	0.084*	0.355 (7)
C25	0.9537 (7)	0.1911 (8)	0.5217 (11)	0.073 (2)	0.355 (7)
H25A	0.9514	0.1761	0.4441	0.088*	0.355 (7)
H25B	1.0328	0.2174	0.5631	0.088*	0.355 (7)
C26	0.9341 (8)	0.1042 (9)	0.5808 (13)	0.098 (4)	0.355 (7)
H26A	0.9248	0.1201	0.6541	0.117*	0.355 (7)
H26B	1.0036	0.0628	0.5954	0.117*	0.355 (7)
C27	0.8192 (8)	0.0548 (5)	0.503 (4)	0.0762 (16)	0.355 (7)
H27A	0.8335	0.0287	0.4359	0.091*	0.355 (7)
H27B	0.7971	0.0039	0.5448	0.091*	0.355 (7)
C27A	0.7193 (3)	0.1256 (2)	0.4670 (3)	0.0577 (8)	0.355 (7)
C37	0.5843 (3)	0.3642 (2)	0.4080 (3)	0.0572 (8)	
O37	0.4800 (2)	0.39474 (16)	0.3860 (3)	0.0874 (8)	
C31	0.6826 (2)	0.4328 (2)	0.4160 (3)	0.0512 (7)	
C32	0.7229 (3)	0.4937 (3)	0.5060 (3)	0.0753 (10)	
H32	0.6887	0.4934	0.5644	0.090*	
C33	0.8159 (4)	0.5564 (3)	0.5100 (5)	0.1018 (15)	
H33	0.8455	0.5969	0.5724	0.122*	
C34	0.8628 (4)	0.5585 (3)	0.4238 (5)	0.0999 (15)	
H34	0.9237	0.6013	0.4261	0.120*	
C35	0.8222 (4)	0.4993 (4)	0.3349 (4)	0.1031 (15)	
H35	0.8550	0.5011	0.2756	0.124*	
C36	0.7326 (3)	0.4361 (3)	0.3309 (3)	0.0810 (11)	

H36	0.7056	0.3948	0.2690	0.097*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C7	0.0484 (17)	0.058 (2)	0.0569 (19)	−0.0049 (15)	0.0201 (14)	−0.0003 (15)
O7	0.0522 (13)	0.0532 (15)	0.1014 (18)	−0.0018 (10)	0.0109 (12)	0.0012 (12)
C1	0.0429 (16)	0.0499 (18)	0.0536 (17)	−0.0011 (13)	0.0156 (13)	0.0025 (14)
C2	0.0530 (19)	0.057 (2)	0.079 (2)	−0.0055 (15)	0.0133 (16)	0.0044 (17)
C3	0.0455 (18)	0.068 (2)	0.082 (2)	−0.0083 (16)	0.0109 (16)	−0.0026 (19)
C4	0.0462 (17)	0.073 (2)	0.0562 (19)	0.0082 (16)	0.0132 (14)	−0.0036 (17)
Cl4	0.0557 (5)	0.1037 (8)	0.0976 (7)	0.0217 (5)	0.0110 (5)	−0.0074 (6)
C5	0.057 (2)	0.060 (2)	0.075 (2)	0.0078 (16)	0.0181 (17)	0.0029 (17)
C6	0.0493 (18)	0.057 (2)	0.077 (2)	−0.0060 (15)	0.0171 (16)	0.0007 (17)
N1	0.0385 (13)	0.0463 (14)	0.0746 (17)	−0.0016 (11)	0.0180 (12)	0.0005 (12)
S11	0.0506 (5)	0.0498 (5)	0.0864 (6)	0.0004 (4)	0.0176 (4)	−0.0018 (4)
C12	0.0434 (15)	0.0482 (17)	0.0576 (18)	−0.0018 (13)	0.0198 (13)	−0.0001 (14)
C13	0.0435 (15)	0.0469 (17)	0.0601 (18)	−0.0009 (13)	0.0204 (13)	0.0003 (14)
C13A	0.0399 (15)	0.0500 (19)	0.0626 (19)	0.0030 (13)	0.0171 (13)	−0.0024 (14)
C14	0.0395 (16)	0.063 (2)	0.100 (3)	0.0026 (15)	0.0122 (17)	−0.005 (2)
C15	0.042 (2)	0.075 (3)	0.093 (6)	0.005 (2)	0.009 (3)	−0.008 (4)
C16	0.058 (3)	0.083 (3)	0.124 (9)	0.028 (2)	−0.007 (4)	−0.031 (5)
C17	0.056 (2)	0.065 (2)	0.097 (4)	0.0137 (17)	0.011 (2)	−0.004 (2)
C17A	0.0476 (17)	0.056 (2)	0.068 (2)	0.0058 (14)	0.0169 (15)	−0.0026 (16)
C23A	0.0399 (15)	0.0500 (19)	0.0626 (19)	0.0030 (13)	0.0171 (13)	−0.0024 (14)
C24	0.0395 (16)	0.063 (2)	0.100 (3)	0.0026 (15)	0.0122 (17)	−0.005 (2)
C25	0.042 (2)	0.075 (3)	0.093 (6)	0.005 (2)	0.009 (3)	−0.008 (4)
C26	0.058 (3)	0.083 (3)	0.124 (9)	0.028 (2)	−0.007 (4)	−0.031 (5)
C27	0.056 (2)	0.065 (2)	0.097 (4)	0.0137 (17)	0.011 (2)	−0.004 (2)
C27A	0.0476 (17)	0.056 (2)	0.068 (2)	0.0058 (14)	0.0169 (15)	−0.0026 (16)
C37	0.0455 (17)	0.0541 (19)	0.071 (2)	0.0009 (14)	0.0181 (15)	−0.0027 (16)
O37	0.0502 (13)	0.0564 (14)	0.155 (2)	0.0055 (11)	0.0335 (14)	−0.0010 (15)
C31	0.0432 (16)	0.0512 (19)	0.0553 (18)	0.0026 (13)	0.0110 (14)	−0.0002 (15)
C32	0.067 (2)	0.083 (3)	0.072 (2)	−0.004 (2)	0.0176 (18)	−0.014 (2)
C33	0.082 (3)	0.077 (3)	0.122 (4)	−0.011 (2)	0.002 (3)	−0.040 (3)
C34	0.061 (3)	0.073 (3)	0.154 (5)	−0.014 (2)	0.020 (3)	0.013 (3)
C35	0.074 (3)	0.133 (4)	0.105 (3)	−0.025 (3)	0.032 (2)	0.024 (3)
C36	0.062 (2)	0.116 (3)	0.067 (2)	−0.022 (2)	0.0238 (19)	−0.009 (2)

Geometric parameters (Å, º)

C7—O7	1.222 (3)	C16—H16A	0.9700
C7—N1	1.361 (4)	C16—H16B	0.9700
C7—C1	1.483 (4)	C17—C17A	1.497 (5)
C1—C2	1.380 (4)	C17—H17A	0.9700
C1—C6	1.382 (4)	C17—H17B	0.9700
C2—C3	1.379 (4)	C24—C25	1.557 (11)
C2—H2	0.9300	C24—H24B	0.9700

C3—C4	1.364 (4)	C24—H24A	0.9700
C3—H3	0.9300	C25—C26	1.491 (8)
C4—C5	1.367 (4)	C25—H25A	0.9700
C4—Cl4	1.738 (3)	C25—H25B	0.9700
C5—C6	1.375 (4)	C26—C27	1.537 (12)
C5—H5	0.9300	C26—H26A	0.9700
C6—H6	0.9300	C26—H26B	0.9700
N1—C12	1.369 (3)	C27—H27A	0.9700
N1—H1	0.8600	C27—H27B	0.9700
S11—C12	1.705 (3)	C37—O37	1.235 (3)
S11—C17A	1.732 (3)	C37—C31	1.487 (4)
C12—C13	1.388 (4)	C31—C36	1.357 (4)
C13—C13A	1.444 (4)	C31—C32	1.362 (4)
C13—C37	1.452 (4)	C32—C33	1.394 (6)
C13A—C17A	1.343 (4)	C32—H32	0.9300
C13A—C14	1.511 (4)	C33—C34	1.345 (6)
C14—C15	1.556 (11)	C33—H33	0.9300
C14—H14A	0.9700	C34—C35	1.337 (6)
C14—H14B	0.9700	C34—H34	0.9300
C15—C16	1.489 (8)	C35—C36	1.369 (5)
C15—H15A	0.9700	C35—H35	0.9300
C15—H15B	0.9700	C36—H36	0.9300
C16—C17	1.534 (11)		

O7—C7—N1	120.7 (3)	C17—C16—H16B	109.6
O7—C7—C1	122.8 (3)	H16A—C16—H16B	108.1
N1—C7—C1	116.5 (3)	C17A—C17—C16	109.5 (6)
C2—C1—C6	118.2 (3)	C17A—C17—H17A	109.8
C2—C1—C7	118.3 (3)	C16—C17—H17A	109.8
C6—C1—C7	123.5 (3)	C17A—C17—H17B	109.8
C3—C2—C1	121.0 (3)	C16—C17—H17B	109.8
C3—C2—H2	119.5	H17A—C17—H17B	108.2
C1—C2—H2	119.5	C13A—C17A—C17	125.8 (3)
C4—C3—C2	119.3 (3)	C13A—C17A—S11	113.4 (2)
C4—C3—H3	120.3	C17—C17A—S11	120.8 (3)
C2—C3—H3	120.3	C25—C24—H24B	109.7
C3—C4—C5	121.2 (3)	C25—C24—H24A	109.7
C3—C4—Cl4	119.5 (3)	H24B—C24—H24A	108.2
C5—C4—Cl4	119.2 (3)	C26—C25—C24	109.8 (11)
C4—C5—C6	119.1 (3)	C26—C25—H25A	109.7
C4—C5—H5	120.5	C24—C25—H25A	109.7
C6—C5—H5	120.5	C26—C25—H25B	109.7
C5—C6—C1	121.3 (3)	C24—C25—H25B	109.7
C5—C6—H6	119.4	H25A—C25—H25B	108.2
C1—C6—H6	119.4	C25—C26—C27	108.7 (12)
C7—N1—C12	126.3 (3)	C25—C26—H26A	109.9
C7—N1—H1	116.9	C27—C26—H26A	109.9
C12—N1—H1	116.9	C25—C26—H26B	109.9

C12—S11—C17A	90.68 (14)	C27—C26—H26B	109.9
N1—C12—C13	124.2 (3)	H26A—C26—H26B	108.3
N1—C12—S11	122.8 (2)	C26—C27—H27A	110.1
C13—C12—S11	112.9 (2)	C26—C27—H27B	110.1
C12—C13—C13A	111.0 (3)	H27A—C27—H27B	108.4
C12—C13—C37	120.3 (2)	O37—C37—C13	122.3 (3)
C13A—C13—C37	128.8 (3)	O37—C37—C31	117.8 (3)
C17A—C13A—C13	112.1 (3)	C13—C37—C31	119.8 (2)
C17A—C13A—C14	121.1 (3)	C36—C31—C32	118.9 (3)
C13—C13A—C14	126.9 (3)	C36—C31—C37	119.5 (3)
C13A—C14—C15	110.8 (6)	C32—C31—C37	121.5 (3)
C13A—C14—H14A	109.5	C31—C32—C33	119.5 (4)
C15—C14—H14A	109.5	C31—C32—H32	120.3
C13A—C14—H14B	109.5	C33—C32—H32	120.3
C15—C14—H14B	109.5	C34—C33—C32	120.2 (4)
H14A—C14—H14B	108.1	C34—C33—H33	119.9
C16—C15—C14	109.8 (9)	C32—C33—H33	119.9
C16—C15—H15A	109.7	C35—C34—C33	120.2 (4)
C14—C15—H15A	109.7	C35—C34—H34	119.9
C16—C15—H15B	109.7	C33—C34—H34	119.9
C14—C15—H15B	109.7	C34—C35—C36	120.3 (4)
H15A—C15—H15B	108.2	C34—C35—H35	119.9
C15—C16—C17	110.3 (10)	C36—C35—H35	119.9
C15—C16—H16A	109.6	C31—C36—C35	120.9 (4)
C17—C16—H16A	109.6	C31—C36—H36	119.5
C15—C16—H16B	109.6	C35—C36—H36	119.5

O7—C7—C1—C2	9.9 (5)	C13—C13A—C14—C15	161.7 (8)
N1—C7—C1—C2	−170.5 (3)	C13A—C14—C15—C16	49 (2)
O7—C7—C1—C6	−168.4 (3)	C14—C15—C16—C17	−66.2 (18)
N1—C7—C1—C6	11.3 (4)	C15—C16—C17—C17A	49 (2)
C6—C1—C2—C3	−1.1 (5)	C13—C13A—C17A—C17	−176.9 (14)
C7—C1—C2—C3	−179.4 (3)	C14—C13A—C17A—C17	2 (2)
C1—C2—C3—C4	0.2 (5)	C13—C13A—C17A—S11	0.1 (4)
C2—C3—C4—C5	0.6 (5)	C14—C13A—C17A—S11	179.4 (13)
C2—C3—C4—Cl4	178.5 (3)	C16—C17—C17A—C13A	−17 (2)
C3—C4—C5—C6	−0.6 (5)	C16—C17—C17A—S11	165.8 (10)
Cl4—C4—C5—C6	−178.5 (2)	C12—S11—C17A—C13A	−0.4 (3)
C4—C5—C6—C1	−0.4 (5)	C12—S11—C17A—C17	176.8 (13)
C2—C1—C6—C5	1.1 (5)	C24—C25—C26—C27	69.1 (18)
C7—C1—C6—C5	179.4 (3)	C12—C13—C37—O37	11.6 (5)
O7—C7—N1—C12	3.7 (5)	C13A—C13—C37—O37	−168.1 (3)
C1—C7—N1—C12	−175.9 (3)	C12—C13—C37—C31	−166.2 (3)
C7—N1—C12—C13	173.6 (3)	C13A—C13—C37—C31	14.2 (5)
C7—N1—C12—S11	−6.6 (4)	O37—C37—C31—C36	−107.6 (4)
C17A—S11—C12—N1	−179.3 (3)	C13—C37—C31—C36	70.2 (4)
C17A—S11—C12—C13	0.6 (2)	O37—C37—C31—C32	71.4 (4)
N1—C12—C13—C13A	179.2 (3)	C13—C37—C31—C32	−110.7 (4)

S11—C12—C13—C13A	−0.6 (3)	C36—C31—C32—C33	−1.3 (5)
N1—C12—C13—C37	−0.5 (5)	C37—C31—C32—C33	179.6 (3)
S11—C12—C13—C37	179.7 (2)	C31—C32—C33—C34	1.9 (6)
C12—C13—C13A—C17A	0.3 (4)	C32—C33—C34—C35	−1.3 (7)
C37—C13—C13A—C17A	180.0 (3)	C33—C34—C35—C36	0.0 (7)
C12—C13—C13A—C14	−179.0 (14)	C32—C31—C36—C35	0.1 (5)
C37—C13—C13A—C14	0.7 (14)	C37—C31—C36—C35	179.1 (3)
C17A—C13A—C14—C15	−17 (2)	C34—C35—C36—C31	0.6 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O37	0.86	1.99	2.641 (3)	132
C3—H3···*Cg*1^{i}	0.93	2.76	3.676 (4)	169
C14—H14*A*···*Cg*1	0.97	2.96	3.475 (9)	114
C24—H24*A*···*Cg*1	0.97	2.73	3.530 (17)	140

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

N-(3-Benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-2,6-difluorobenzamide (III)

Crystal data

$C_{22}H_{17}F_2NO_2S$
M_r = 397.43
Triclinic, $P\overline{1}$
a = 7.6718 (3) Å
b = 9.8833 (3) Å
c = 13.5925 (5) Å
α = 104.925 (4)°
β = 95.476 (2)°
γ = 103.633 (3)°
V = 954.32 (6) Å^3
Z = 2
F(000) = 412
D_x = 1.383 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4414 reflections
θ = 1.6–27.6°
μ = 0.21 mm^{-1}
T = 296 K
Block, colourless
0.20 × 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} = 0.942, T_{max} = 0.970
21940 measured reflections
4375 independent reflections
2814 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.5°, θ_{min} = 2.2°
h = −9→9
k = −12→12
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.108
S = 1.01
4375 reflections
267 parameters
12 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.3345P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0131 (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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C7	0.2985 (3)	−0.1093 (2)	0.25624 (16)	0.0428 (5)	
O7	0.3232 (2)	−0.18513 (16)	0.17654 (11)	0.0596 (4)	
C1	0.3154 (3)	−0.1458 (2)	0.35650 (15)	0.0413 (5)	
C2	0.4588 (3)	−0.1956 (2)	0.38729 (17)	0.0513 (5)	
F2	0.5870 (2)	−0.20460 (18)	0.32721 (11)	0.0828 (5)	
C3	0.4795 (4)	−0.2323 (2)	0.47761 (19)	0.0631 (7)	
H3	0.5779	−0.2661	0.4953	0.076*	
C4	0.3530 (4)	−0.2184 (2)	0.54089 (18)	0.0645 (7)	
H4	0.3657	−0.2426	0.6024	0.077*	
C5	0.2073 (4)	−0.1690 (3)	0.51490 (18)	0.0620 (6)	
H5	0.1212	−0.1591	0.5581	0.074*	
C6	0.1921 (3)	−0.1348 (2)	0.42373 (17)	0.0513 (5)	
F6	0.04692 (19)	−0.08836 (18)	0.39741 (12)	0.0821 (5)	
N1	0.2570 (2)	0.01864 (17)	0.26251 (12)	0.0430 (4)	
H1	0.2418	0.0668	0.3220	0.052*	
S11	0.28668 (7)	0.00737 (6)	0.06263 (4)	0.04521 (16)	
C12	0.2370 (2)	0.0786 (2)	0.18196 (14)	0.0376 (4)	
C13	0.1764 (3)	0.2008 (2)	0.18835 (14)	0.0384 (4)	
C13A	0.1798 (3)	0.2408 (2)	0.09357 (15)	0.0408 (5)	0.784 (6)
C14	0.1373 (9)	0.3748 (4)	0.0747 (3)	0.0538 (13)	0.784 (6)
H14A	0.1786	0.4556	0.1373	0.065*	0.784 (6)
H14B	0.0069	0.3567	0.0560	0.065*	0.784 (6)
C15	0.2319 (6)	0.4131 (4)	−0.0120 (3)	0.0719 (13)	0.784 (6)
H15A	0.1923	0.4911	−0.0301	0.086*	0.784 (6)
H15B	0.3624	0.4467	0.0111	0.086*	0.784 (6)
C16	0.1877 (6)	0.2814 (4)	−0.1058 (3)	0.0721 (12)	0.784 (6)
H16A	0.2378	0.3097	−0.1626	0.087*	0.784 (6)
H16B	0.0567	0.2449	−0.1262	0.087*	0.784 (6)
C17	0.2639 (16)	0.1612 (6)	−0.0845 (3)	0.0596 (8)	0.784 (6)
H17B	0.2036	0.0698	−0.1365	0.071*	0.784 (6)
H17A	0.3928	0.1833	−0.0881	0.071*	0.784 (6)
C17A	0.2358 (3)	0.1471 (2)	0.02047 (15)	0.0430 (5)	0.784 (6)
C23A	0.1798 (3)	0.2408 (2)	0.09357 (15)	0.0408 (5)	0.216 (6)
C24	0.178 (4)	0.3878 (13)	0.0789 (10)	0.0538 (13)	0.216 (6)
H24A	0.2917	0.4590	0.1140	0.065*	0.216 (6)
H24B	0.0798	0.4197	0.1091	0.065*	0.216 (6)
C25	0.1530 (18)	0.3780 (16)	−0.0355 (10)	0.0719 (13)	0.216 (6)
H25A	0.0303	0.3209	−0.0678	0.086*	0.216 (6)
H25B	0.1685	0.4749	−0.0431	0.086*	0.216 (6)

C26	0.2875 (19)	0.3092 (14)	−0.0896 (10)	0.0721 (12)	0.216 (6)
H26A	0.4104	0.3637	−0.0558	0.087*	0.216 (6)
H26B	0.2748	0.3122	−0.1606	0.087*	0.216 (6)
C27	0.255 (6)	0.152 (2)	−0.0873 (11)	0.0596 (8)	0.216 (6)
H27A	0.1448	0.0920	−0.1346	0.071*	0.216 (6)
H27B	0.3558	0.1150	−0.1084	0.071*	0.216 (6)
C27A	0.2358 (3)	0.1471 (2)	0.02047 (15)	0.0430 (5)	0.216 (6)
C37	0.1167 (3)	0.2722 (2)	0.28286 (15)	0.0421 (5)	
O37	0.1794 (2)	0.26502 (18)	0.36795 (11)	0.0648 (5)	
C31	−0.0315 (3)	0.34488 (19)	0.27706 (14)	0.0371 (4)	
C32	−0.0286 (3)	0.4664 (2)	0.35635 (16)	0.0496 (5)	
H32	0.0702	0.5062	0.4098	0.060*	
C33	−0.1713 (3)	0.5282 (2)	0.35617 (19)	0.0594 (6)	
H33	−0.1680	0.6106	0.4090	0.071*	
C34	−0.3193 (3)	0.4684 (2)	0.27803 (19)	0.0564 (6)	
H34	−0.4159	0.5103	0.2784	0.068*	
C35	−0.3245 (3)	0.3475 (2)	0.19989 (17)	0.0506 (5)	
H35	−0.4252	0.3066	0.1476	0.061*	
C36	−0.1803 (3)	0.2864 (2)	0.19885 (15)	0.0436 (5)	
H36	−0.1833	0.2052	0.1451	0.052*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C7	0.0445 (12)	0.0452 (11)	0.0463 (12)	0.0214 (9)	0.0107 (9)	0.0168 (9)
O7	0.0837 (12)	0.0613 (9)	0.0483 (9)	0.0418 (9)	0.0203 (8)	0.0172 (7)
C1	0.0490 (12)	0.0376 (10)	0.0434 (11)	0.0190 (9)	0.0079 (9)	0.0157 (9)
C2	0.0582 (14)	0.0505 (12)	0.0540 (13)	0.0283 (11)	0.0099 (11)	0.0175 (10)
F2	0.0810 (10)	0.1194 (12)	0.0805 (10)	0.0712 (10)	0.0281 (8)	0.0394 (9)
C3	0.0720 (17)	0.0602 (14)	0.0618 (15)	0.0279 (13)	−0.0077 (13)	0.0234 (12)
C4	0.0820 (19)	0.0572 (14)	0.0531 (14)	0.0079 (13)	−0.0050 (13)	0.0299 (12)
C5	0.0734 (17)	0.0640 (15)	0.0552 (14)	0.0138 (13)	0.0207 (12)	0.0295 (12)
C6	0.0539 (13)	0.0525 (12)	0.0583 (14)	0.0219 (11)	0.0134 (11)	0.0263 (11)
F6	0.0696 (10)	0.1271 (13)	0.0931 (11)	0.0595 (9)	0.0414 (8)	0.0669 (10)
N1	0.0573 (11)	0.0476 (9)	0.0354 (9)	0.0285 (8)	0.0115 (8)	0.0170 (7)
S11	0.0528 (3)	0.0506 (3)	0.0391 (3)	0.0232 (2)	0.0131 (2)	0.0146 (2)
C12	0.0373 (11)	0.0435 (10)	0.0367 (10)	0.0169 (9)	0.0073 (8)	0.0141 (8)
C13	0.0380 (11)	0.0447 (10)	0.0382 (10)	0.0184 (9)	0.0061 (8)	0.0149 (8)
C13A	0.0361 (11)	0.0501 (11)	0.0436 (11)	0.0159 (9)	0.0073 (9)	0.0219 (9)
C14	0.055 (3)	0.0614 (15)	0.0632 (15)	0.0265 (17)	0.0151 (16)	0.0373 (13)
C15	0.065 (3)	0.084 (3)	0.105 (3)	0.036 (2)	0.044 (2)	0.068 (2)
C16	0.073 (3)	0.104 (3)	0.064 (2)	0.032 (3)	0.022 (2)	0.056 (2)
C17	0.0620 (18)	0.0813 (19)	0.0489 (13)	0.0252 (15)	0.0207 (12)	0.0327 (13)
C17A	0.0385 (11)	0.0563 (12)	0.0405 (11)	0.0157 (9)	0.0079 (9)	0.0219 (10)
C23A	0.0361 (11)	0.0501 (11)	0.0436 (11)	0.0159 (9)	0.0073 (9)	0.0219 (9)
C24	0.055 (3)	0.0614 (15)	0.0632 (15)	0.0265 (17)	0.0151 (16)	0.0373 (13)
C25	0.065 (3)	0.084 (3)	0.105 (3)	0.036 (2)	0.044 (2)	0.068 (2)
C26	0.073 (3)	0.104 (3)	0.064 (2)	0.032 (3)	0.022 (2)	0.056 (2)

C27	0.0620 (18)	0.0813 (19)	0.0489 (13)	0.0252 (15)	0.0207 (12)	0.0327 (13)
C27A	0.0385 (11)	0.0563 (12)	0.0405 (11)	0.0157 (9)	0.0079 (9)	0.0219 (10)
C37	0.0500 (12)	0.0421 (10)	0.0392 (11)	0.0202 (9)	0.0072 (9)	0.0134 (9)
O37	0.0888 (12)	0.0849 (11)	0.0370 (8)	0.0588 (10)	0.0047 (8)	0.0159 (8)
C31	0.0431 (11)	0.0380 (10)	0.0362 (10)	0.0169 (9)	0.0086 (9)	0.0152 (8)
C32	0.0544 (13)	0.0453 (11)	0.0471 (12)	0.0201 (10)	0.0033 (10)	0.0059 (9)
C33	0.0646 (15)	0.0453 (12)	0.0690 (16)	0.0276 (11)	0.0132 (13)	0.0052 (11)
C34	0.0506 (14)	0.0554 (13)	0.0777 (17)	0.0304 (11)	0.0192 (12)	0.0267 (12)
C35	0.0402 (12)	0.0583 (13)	0.0568 (13)	0.0155 (10)	0.0026 (10)	0.0226 (11)
C36	0.0492 (12)	0.0427 (11)	0.0394 (11)	0.0169 (9)	0.0060 (9)	0.0093 (9)

Geometric parameters (Å, º)

C7—O7	1.208 (2)	C16—H16A	0.9700
C7—N1	1.359 (2)	C16—H16B	0.9700
C7—C1	1.499 (3)	C17—C17A	1.499 (3)
C1—C2	1.379 (3)	C17—H17B	0.9700
C1—C6	1.380 (3)	C17—H17A	0.9700
C2—F2	1.341 (2)	C24—C25	1.522 (6)
C2—C3	1.373 (3)	C24—H24A	0.9700
C3—C4	1.365 (3)	C24—H24B	0.9700
C3—H3	0.9300	C25—C26	1.512 (6)
C4—C5	1.372 (3)	C25—H25A	0.9700
C4—H4	0.9300	C25—H25B	0.9700
C5—C6	1.368 (3)	C26—C27	1.523 (7)
C5—H5	0.9300	C26—H26A	0.9700
C6—F6	1.353 (2)	C26—H26B	0.9700
N1—C12	1.385 (2)	C27—H27A	0.9700
N1—H1	0.8600	C27—H27B	0.9700
S11—C12	1.7111 (19)	C37—O37	1.236 (2)
S11—C17A	1.7338 (19)	C37—C31	1.487 (3)
C12—C13	1.379 (2)	C31—C36	1.381 (3)
C13—C13A	1.443 (3)	C31—C32	1.386 (3)
C13—C37	1.468 (3)	C32—C33	1.374 (3)
C13A—C17A	1.351 (3)	C32—H32	0.9300
C13A—C14	1.513 (3)	C33—C34	1.377 (3)
C14—C15	1.525 (4)	C33—H33	0.9300
C14—H14A	0.9700	C34—C35	1.369 (3)
C14—H14B	0.9700	C34—H34	0.9300
C15—C16	1.513 (4)	C35—C36	1.379 (3)
C15—H15A	0.9700	C35—H35	0.9300
C15—H15B	0.9700	C36—H36	0.9300
C16—C17	1.520 (6)		

O7—C7—N1	122.35 (18)	C17—C16—H16B	109.3
O7—C7—C1	123.19 (17)	H16A—C16—H16B	107.9
N1—C7—C1	114.45 (17)	C17A—C17—C16	110.1 (3)
C2—C1—C6	114.98 (18)	C17A—C17—H17B	109.6

C2—C1—C7	120.96 (18)	C16—C17—H17B	109.6
C6—C1—C7	124.06 (18)	C17A—C17—H17A	109.6
F2—C2—C3	118.5 (2)	C16—C17—H17A	109.6
F2—C2—C1	118.20 (18)	H17B—C17—H17A	108.1
C3—C2—C1	123.3 (2)	C13A—C17A—C17	125.9 (2)
C4—C3—C2	118.7 (2)	C13A—C17A—S11	112.59 (14)
C4—C3—H3	120.6	C17—C17A—S11	121.4 (2)
C2—C3—H3	120.6	C25—C24—H24A	109.6
C3—C4—C5	120.9 (2)	C25—C24—H24B	109.6
C3—C4—H4	119.6	H24A—C24—H24B	108.1
C5—C4—H4	119.6	C26—C25—C24	111.7 (7)
C6—C5—C4	118.1 (2)	C26—C25—H25A	109.3
C6—C5—H5	120.9	C24—C25—H25A	109.3
C4—C5—H5	120.9	C26—C25—H25B	109.3
F6—C6—C5	118.1 (2)	C24—C25—H25B	109.3
F6—C6—C1	117.93 (18)	H25A—C25—H25B	107.9
C5—C6—C1	124.0 (2)	C25—C26—C27	110.3 (8)
C7—N1—C12	126.00 (16)	C25—C26—H26A	109.6
C7—N1—H1	117.0	C27—C26—H26A	109.6
C12—N1—H1	117.0	C25—C26—H26B	109.6
C12—S11—C17A	91.04 (9)	C27—C26—H26B	109.6
C13—C12—N1	124.46 (17)	H26A—C26—H26B	108.1
C13—C12—S11	112.65 (14)	C26—C27—H27A	110.0
N1—C12—S11	122.90 (13)	C26—C27—H27B	110.0
C12—C13—C13A	111.36 (16)	H27A—C27—H27B	108.4
C12—C13—C37	120.60 (16)	O37—C37—C13	120.90 (17)
C13A—C13—C37	128.04 (16)	O37—C37—C31	118.50 (17)
C17A—C13A—C13	112.28 (16)	C13—C37—C31	120.43 (16)
C17A—C13A—C14	121.3 (2)	C36—C31—C32	119.09 (18)
C13—C13A—C14	126.4 (2)	C36—C31—C37	121.26 (17)
C13A—C14—C15	109.7 (3)	C32—C31—C37	119.41 (17)
C13A—C14—H14A	109.7	C33—C32—C31	120.2 (2)
C15—C14—H14A	109.7	C33—C32—H32	119.9
C13A—C14—H14B	109.7	C31—C32—H32	119.9
C15—C14—H14B	109.7	C32—C33—C34	120.2 (2)
H14A—C14—H14B	108.2	C32—C33—H33	119.9
C16—C15—C14	110.2 (4)	C34—C33—H33	119.9
C16—C15—H15A	109.6	C35—C34—C33	120.1 (2)
C14—C15—H15A	109.6	C35—C34—H34	119.9
C16—C15—H15B	109.6	C33—C34—H34	119.9
C14—C15—H15B	109.6	C34—C35—C36	119.9 (2)
H15A—C15—H15B	108.1	C34—C35—H35	120.1
C15—C16—C17	111.7 (4)	C36—C35—H35	120.1
C15—C16—H16A	109.3	C35—C36—C31	120.51 (19)
C17—C16—H16A	109.3	C35—C36—H36	119.7
C15—C16—H16B	109.3	C31—C36—H36	119.7
O7—C7—C1—C2	44.1 (3)	C37—C13—C13A—C14	5.8 (4)

N1—C7—C1—C2	−134.5 (2)	C17A—C13A—C14—C15	−21.2 (6)
O7—C7—C1—C6	−135.1 (2)	C13—C13A—C14—C15	155.4 (3)
N1—C7—C1—C6	46.3 (3)	C13A—C14—C15—C16	52.0 (6)
C6—C1—C2—F2	−177.94 (19)	C14—C15—C16—C17	−65.0 (8)
C7—C1—C2—F2	2.8 (3)	C15—C16—C17—C17A	41.7 (10)
C6—C1—C2—C3	0.2 (3)	C13—C13A—C17A—C17	−176.6 (6)
C7—C1—C2—C3	−179.1 (2)	C14—C13A—C17A—C17	0.4 (8)
F2—C2—C3—C4	177.7 (2)	C13—C13A—C17A—S11	−0.1 (2)
C1—C2—C3—C4	−0.4 (4)	C14—C13A—C17A—S11	177.0 (3)
C2—C3—C4—C5	0.2 (4)	C16—C17—C17A—C13A	−10.4 (11)
C3—C4—C5—C6	0.2 (4)	C16—C17—C17A—S11	173.3 (4)
C4—C5—C6—F6	179.0 (2)	C12—S11—C17A—C13A	−1.37 (16)
C4—C5—C6—C1	−0.5 (4)	C12—S11—C17A—C17	175.4 (6)
C2—C1—C6—F6	−179.17 (19)	C24—C25—C26—C27	64.1 (14)
C7—C1—C6—F6	0.1 (3)	C12—C13—C37—O37	28.0 (3)
C2—C1—C6—C5	0.3 (3)	C13A—C13—C37—O37	−152.8 (2)
C7—C1—C6—C5	179.6 (2)	C12—C13—C37—C31	−147.08 (19)
O7—C7—N1—C12	0.2 (3)	C13A—C13—C37—C31	32.1 (3)
C1—C7—N1—C12	178.76 (18)	O37—C37—C31—C36	−137.2 (2)
C7—N1—C12—C13	172.90 (19)	C13—C37—C31—C36	38.0 (3)
C7—N1—C12—S11	−7.3 (3)	O37—C37—C31—C32	37.0 (3)
C17A—S11—C12—C13	2.51 (16)	C13—C37—C31—C32	−147.78 (19)
C17A—S11—C12—N1	−177.29 (17)	C36—C31—C32—C33	−0.7 (3)
N1—C12—C13—C13A	176.82 (18)	C37—C31—C32—C33	−175.0 (2)
S11—C12—C13—C13A	−3.0 (2)	C31—C32—C33—C34	1.0 (3)
N1—C12—C13—C37	−3.9 (3)	C32—C33—C34—C35	−0.3 (4)
S11—C12—C13—C37	176.34 (15)	C33—C34—C35—C36	−0.7 (3)
C12—C13—C13A—C17A	1.9 (2)	C34—C35—C36—C31	1.0 (3)
C37—C13—C13A—C17A	−177.31 (19)	C32—C31—C36—C35	−0.4 (3)
C12—C13—C13A—C14	−174.9 (4)	C37—C31—C36—C35	173.88 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O37	0.86	2.08	2.718 (2)	130
C3—H3···O37^{i}	0.93	2.50	3.326 (3)	148
C17—H17*A*···O7^{ii}	0.97	2.59	3.494 (12)	156
C4—H4···*Cg*1^{iii}	0.93	2.85	3.635 (2)	143
C35—H35···*Cg*2^{iv}	0.93	2.75	3.520 (2)	141

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

N-(3-Benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophen-2-yl)-2-methoxybenzamide (IV)

Crystal data

$C_{23}H_{21}NO_3S$
M_r = 391.47
Triclinic, $P\overline{1}$
a = 8.5129 (9) Å
b = 12.9230 (14) Å
c = 19.031 (2) Å
α = 96.811 (5)°
β = 102.665 (6)°

γ = 105.321 (5)°
V = 1935.1 (4) Å^3
Z = 4
F(000) = 824
D_x = 1.344 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 8692 reflections
θ = 1.1–27.4°
μ = 0.19 mm^{-1}
T = 296 K
Block, colourless
0.20 × 0.15 × 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} = 0.935, T_{max} = 0.972
37373 measured reflections

8015 independent reflections
4159 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 26.5°, θ_{min} = 2.2°
h = −10→10
k = −16→16
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.151
S = 0.97
8015 reflections
511 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C17	0.7291 (3)	0.4920 (2)	0.42332 (17)	0.0432 (7)
O17	0.6890 (2)	0.46367 (16)	0.35654 (11)	0.0583 (6)
C11	0.8727 (3)	0.5902 (2)	0.46126 (16)	0.0415 (7)
C12	0.9407 (3)	0.6250 (2)	0.53685 (16)	0.0445 (7)
C13	1.0831 (4)	0.7148 (3)	0.56369 (19)	0.0558 (8)
H13	1.135 (4)	0.735 (2)	0.6153 (16)	0.067*
C14	1.1547 (4)	0.7718 (2)	0.5164 (2)	0.0623 (9)
H14	1.2494	0.8325	0.5350	0.075*
C15	1.0888 (4)	0.7406 (3)	0.4423 (2)	0.0616 (9)
H15	1.1372	0.7799	0.4106	0.074*
C16	0.9503 (4)	0.6504 (2)	0.41536 (17)	0.0520 (8)
H16	0.9066	0.6287	0.3649	0.062*
O12	0.8629 (2)	0.56645 (16)	0.58071 (11)	0.0574 (5)
C18	0.9289 (4)	0.5935 (3)	0.65822 (16)	0.0706 (10)
H18A	1.0436	0.5917	0.6708	0.106*
H18B	0.8624	0.5417	0.6805	0.106*

H18C	0.9253	0.6655	0.6757	0.106*
N11	0.6436 (3)	0.43260 (17)	0.46496 (14)	0.0420 (6)
H11	0.6726	0.4563	0.5115	0.071 (11)*
S111	0.44061 (9)	0.28363 (6)	0.34524 (4)	0.0479 (2)
C112	0.5132 (3)	0.3366 (2)	0.43732 (14)	0.0376 (6)
C113	0.4327 (3)	0.2715 (2)	0.47888 (14)	0.0349 (6)
C13A	0.3002 (3)	0.1774 (2)	0.43310 (14)	0.0366 (6)
C114	0.1737 (3)	0.0934 (2)	0.45662 (15)	0.0465 (7)
H11A	0.1351	0.1293	0.4942	0.056*
H11B	0.2278	0.0429	0.4778	0.056*
C115	0.0235 (4)	0.0303 (3)	0.39350 (16)	0.0585 (8)
H11C	−0.0395	−0.0340	0.4081	0.070*
H11D	−0.0501	0.0754	0.3825	0.070*
C116	0.0732 (4)	−0.0040 (2)	0.32617 (17)	0.0617 (9)
H11E	−0.0271	−0.0467	0.2882	0.074*
H11F	0.1452	−0.0502	0.3368	0.074*
C117	0.1663 (4)	0.0936 (2)	0.29797 (16)	0.0556 (8)
H11G	0.2236	0.0694	0.2634	0.067*
H11H	0.0862	0.1269	0.2728	0.067*
C17A	0.2920 (3)	0.1753 (2)	0.36089 (15)	0.0421 (7)
C137	0.4843 (3)	0.2989 (2)	0.55881 (15)	0.0394 (7)
O137	0.5584 (3)	0.39291 (16)	0.59155 (11)	0.0584 (6)
C131	0.4570 (3)	0.2123 (2)	0.60386 (14)	0.0384 (6)
C132	0.4077 (3)	0.2322 (2)	0.66724 (15)	0.0493 (7)
H132	0.3825	0.2968	0.6791	0.059*
C133	0.3958 (4)	0.1579 (3)	0.71268 (17)	0.0661 (9)
H133	0.3608	0.1717	0.7549	0.079*
C134	0.4347 (4)	0.0635 (3)	0.69670 (19)	0.0676 (10)
H134	0.4270	0.0136	0.7281	0.081*
C135	0.4853 (3)	0.0422 (2)	0.6344 (2)	0.0596 (9)
H135	0.5134	−0.0216	0.6238	0.072*
C136	0.4944 (3)	0.1162 (2)	0.58719 (16)	0.0480 (7)
H136	0.5258	0.1010	0.5442	0.058*
C27	0.6652 (3)	−0.0187 (2)	0.90359 (16)	0.0422 (7)
O27	0.6146 (2)	−0.01895 (17)	0.83797 (11)	0.0642 (6)
C21	0.5718 (3)	−0.1027 (2)	0.93938 (16)	0.0414 (7)
C22	0.6084 (3)	−0.1044 (2)	1.01425 (17)	0.0446 (7)
C23	0.5056 (4)	−0.1841 (3)	1.04075 (19)	0.0615 (9)
H23	0.5293	−0.1847	1.0908	0.074*
C24	0.3689 (4)	−0.2621 (3)	0.9936 (2)	0.0708 (10)
H24	0.3013	−0.3156	1.0119	0.085*
C25	0.3312 (4)	−0.2623 (3)	0.9203 (2)	0.0709 (10)
H25	0.2380	−0.3153	0.8887	0.085*
C26	0.4318 (4)	−0.1833 (2)	0.89307 (18)	0.0584 (8)
H26	0.4059	−0.1838	0.8429	0.070*
O22	0.7458 (2)	−0.02601 (16)	1.05790 (11)	0.0558 (5)
C28	0.7914 (4)	−0.0238 (3)	1.13475 (16)	0.0661 (9)
H28A	0.7028	−0.0121	1.1553	0.099*

H28B	0.8937	0.0344	1.1573	0.099*
H28C	0.8085	−0.0921	1.1435	0.099*
N21	0.8057 (3)	0.05953 (16)	0.94501 (12)	0.0395 (5)
H21	0.8377	0.0567	0.9907	0.047*
S211	0.85579 (9)	0.15549 (6)	0.82917 (4)	0.0450 (2)
C212	0.9009 (3)	0.1431 (2)	0.91981 (14)	0.0361 (6)
C213	1.0392 (3)	0.2265 (2)	0.96251 (14)	0.0345 (6)
C23A	1.1148 (3)	0.2983 (2)	0.91849 (14)	0.0365 (6)
C214	1.2753 (3)	0.3921 (2)	0.94344 (15)	0.0485 (7)
H21A	1.3573	0.3730	0.9795	0.058*
H21B	1.2525	0.4556	0.9667	0.058*
C215	1.3484 (4)	0.4203 (3)	0.87987 (17)	0.0628 (9)
H21C	1.4384	0.4890	0.8958	0.075*
H21D	1.3963	0.3641	0.8642	0.075*
C216	1.2157 (4)	0.4297 (3)	0.81655 (17)	0.0649 (9)
H21E	1.2678	0.4529	0.7783	0.078*
H21F	1.1669	0.4852	0.8326	0.078*
C217	1.0772 (4)	0.3228 (2)	0.78561 (16)	0.0523 (8)
H21G	0.9790	0.3363	0.7559	0.063*
H21H	1.1154	0.2752	0.7544	0.063*
C27A	1.0301 (3)	0.2683 (2)	0.84655 (15)	0.0419 (7)
C237	1.0898 (3)	0.2345 (2)	1.04255 (15)	0.0387 (6)
O237	1.0632 (2)	0.15246 (15)	1.07003 (10)	0.0558 (5)
C231	1.1630 (3)	0.3431 (2)	1.09194 (14)	0.0362 (6)
C232	1.2766 (3)	0.3540 (2)	1.15913 (15)	0.0450 (7)
H232	1.3126	0.2949	1.1711	0.054*
C233	1.3360 (4)	0.4516 (3)	1.20796 (16)	0.0550 (8)
H233	1.4131	0.4587	1.2525	0.066*
C234	1.2813 (4)	0.5386 (3)	1.19082 (17)	0.0553 (8)
H234	1.3205	0.6044	1.2241	0.066*
C235	1.1685 (3)	0.5288 (2)	1.12438 (16)	0.0483 (7)
H235	1.1315	0.5878	1.1129	0.058*
C236	1.1106 (3)	0.4314 (2)	1.07503 (14)	0.0407 (7)
H236	1.0356	0.4252	1.0300	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C17	0.0435 (17)	0.0418 (17)	0.049 (2)	0.0131 (14)	0.0166 (15)	0.0177 (15)
O17	0.0615 (13)	0.0640 (14)	0.0417 (13)	0.0025 (11)	0.0148 (10)	0.0154 (11)
C11	0.0411 (16)	0.0343 (16)	0.0530 (19)	0.0115 (13)	0.0162 (14)	0.0161 (14)
C12	0.0451 (17)	0.0414 (17)	0.052 (2)	0.0145 (14)	0.0194 (15)	0.0125 (15)
C13	0.0477 (19)	0.047 (2)	0.068 (2)	0.0107 (16)	0.0124 (17)	0.0033 (18)
C14	0.0450 (19)	0.0430 (19)	0.092 (3)	0.0005 (15)	0.0214 (19)	0.0108 (19)
C15	0.054 (2)	0.054 (2)	0.078 (3)	0.0049 (17)	0.0275 (19)	0.0261 (19)
C16	0.0503 (18)	0.0481 (18)	0.061 (2)	0.0100 (16)	0.0211 (16)	0.0213 (16)
O12	0.0589 (13)	0.0593 (13)	0.0435 (13)	0.0011 (11)	0.0131 (10)	0.0079 (11)
C18	0.071 (2)	0.085 (3)	0.047 (2)	0.010 (2)	0.0170 (18)	0.0030 (19)

N11	0.0439 (14)	0.0374 (13)	0.0422 (16)	0.0061 (11)	0.0118 (11)	0.0104 (12)
S111	0.0529 (5)	0.0507 (5)	0.0370 (4)	0.0070 (4)	0.0127 (3)	0.0135 (4)
C112	0.0342 (15)	0.0396 (16)	0.0397 (16)	0.0116 (13)	0.0093 (12)	0.0102 (13)
C113	0.0368 (15)	0.0321 (15)	0.0353 (16)	0.0077 (12)	0.0108 (12)	0.0080 (13)
C13A	0.0391 (15)	0.0356 (15)	0.0380 (17)	0.0122 (13)	0.0130 (13)	0.0100 (13)
C114	0.0429 (17)	0.0464 (17)	0.0443 (18)	0.0028 (14)	0.0135 (14)	0.0066 (14)
C115	0.0527 (19)	0.058 (2)	0.052 (2)	−0.0011 (16)	0.0125 (16)	0.0047 (16)
C116	0.057 (2)	0.057 (2)	0.056 (2)	−0.0008 (16)	0.0158 (17)	−0.0027 (17)
C117	0.060 (2)	0.0520 (19)	0.0429 (19)	0.0054 (16)	0.0075 (15)	0.0019 (15)
C17A	0.0444 (16)	0.0386 (16)	0.0397 (17)	0.0085 (13)	0.0096 (13)	0.0059 (13)
C137	0.0434 (16)	0.0387 (17)	0.0368 (17)	0.0107 (14)	0.0142 (13)	0.0069 (14)
O137	0.0811 (15)	0.0413 (13)	0.0447 (13)	0.0050 (11)	0.0188 (11)	0.0035 (10)
C131	0.0362 (15)	0.0428 (17)	0.0309 (16)	0.0054 (13)	0.0061 (12)	0.0062 (13)
C132	0.0522 (18)	0.0560 (19)	0.0365 (17)	0.0099 (15)	0.0125 (14)	0.0096 (15)
C133	0.061 (2)	0.083 (3)	0.042 (2)	0.001 (2)	0.0105 (16)	0.0200 (19)
C134	0.058 (2)	0.078 (3)	0.053 (2)	−0.0005 (19)	−0.0025 (17)	0.037 (2)
C135	0.0419 (18)	0.050 (2)	0.082 (3)	0.0113 (15)	0.0009 (17)	0.0280 (19)
C136	0.0420 (17)	0.0532 (19)	0.0469 (18)	0.0117 (14)	0.0095 (14)	0.0114 (16)
C27	0.0414 (17)	0.0390 (17)	0.0436 (19)	0.0108 (14)	0.0077 (14)	0.0070 (14)
O27	0.0591 (13)	0.0657 (15)	0.0460 (14)	−0.0058 (11)	−0.0010 (11)	0.0110 (11)
C21	0.0379 (16)	0.0377 (16)	0.0511 (19)	0.0125 (13)	0.0135 (14)	0.0115 (14)
C22	0.0424 (17)	0.0389 (17)	0.059 (2)	0.0167 (14)	0.0171 (15)	0.0156 (15)
C23	0.062 (2)	0.055 (2)	0.080 (3)	0.0189 (18)	0.0327 (19)	0.0316 (19)
C24	0.059 (2)	0.050 (2)	0.108 (3)	0.0067 (18)	0.037 (2)	0.026 (2)
C25	0.053 (2)	0.047 (2)	0.100 (3)	−0.0028 (16)	0.018 (2)	0.009 (2)
C26	0.0520 (19)	0.0461 (19)	0.066 (2)	0.0068 (16)	0.0067 (17)	0.0024 (17)
O22	0.0563 (13)	0.0579 (13)	0.0445 (13)	0.0027 (11)	0.0089 (10)	0.0170 (10)
C28	0.071 (2)	0.086 (3)	0.044 (2)	0.0231 (19)	0.0141 (17)	0.0247 (18)
N21	0.0408 (13)	0.0384 (13)	0.0355 (13)	0.0070 (11)	0.0073 (11)	0.0087 (11)
S211	0.0495 (4)	0.0447 (4)	0.0367 (4)	0.0085 (4)	0.0105 (3)	0.0063 (3)
C212	0.0388 (15)	0.0351 (15)	0.0373 (16)	0.0123 (13)	0.0130 (13)	0.0088 (13)
C213	0.0376 (15)	0.0305 (15)	0.0351 (16)	0.0108 (12)	0.0089 (12)	0.0051 (12)
C23A	0.0398 (15)	0.0326 (15)	0.0379 (17)	0.0109 (13)	0.0129 (13)	0.0040 (13)
C214	0.0449 (17)	0.0453 (18)	0.0529 (19)	0.0067 (14)	0.0170 (15)	0.0075 (15)
C215	0.057 (2)	0.064 (2)	0.062 (2)	−0.0002 (17)	0.0284 (18)	0.0125 (18)
C216	0.071 (2)	0.062 (2)	0.062 (2)	0.0062 (19)	0.0299 (19)	0.0231 (18)
C217	0.063 (2)	0.0466 (18)	0.051 (2)	0.0134 (16)	0.0260 (16)	0.0122 (15)
C27A	0.0472 (16)	0.0396 (16)	0.0423 (18)	0.0144 (14)	0.0174 (14)	0.0065 (14)
C237	0.0365 (15)	0.0385 (16)	0.0418 (17)	0.0121 (13)	0.0093 (13)	0.0099 (14)
O237	0.0694 (14)	0.0410 (12)	0.0494 (13)	0.0107 (10)	0.0034 (10)	0.0160 (10)
C231	0.0350 (15)	0.0392 (16)	0.0338 (16)	0.0077 (12)	0.0107 (12)	0.0096 (13)
C232	0.0474 (17)	0.0466 (18)	0.0383 (17)	0.0097 (14)	0.0074 (14)	0.0141 (15)
C233	0.0528 (19)	0.066 (2)	0.0354 (18)	0.0049 (17)	0.0058 (14)	0.0090 (16)
C234	0.060 (2)	0.0483 (19)	0.049 (2)	0.0040 (16)	0.0181 (16)	−0.0013 (16)
C235	0.0531 (18)	0.0462 (18)	0.0510 (19)	0.0199 (15)	0.0192 (15)	0.0085 (16)
C236	0.0438 (16)	0.0439 (17)	0.0355 (16)	0.0152 (14)	0.0098 (13)	0.0087 (14)

Geometric parameters (Å, º)

C17—O17	1.224 (3)	C27—O27	1.228 (3)
C17—N11	1.360 (3)	C27—N21	1.354 (3)
C17—C11	1.484 (4)	C27—C21	1.493 (4)
C11—C16	1.395 (4)	C21—C22	1.395 (4)
C11—C12	1.397 (4)	C21—C26	1.396 (4)
C12—O12	1.355 (3)	C22—O22	1.354 (3)
C12—C13	1.386 (4)	C22—C23	1.386 (4)
C13—C14	1.374 (4)	C23—C24	1.372 (4)
C13—H13	0.96 (3)	C23—H23	0.9300
C14—C15	1.364 (4)	C24—C25	1.361 (5)
C14—H14	0.9300	C24—H24	0.9300
C15—C16	1.370 (4)	C25—C26	1.379 (4)
C15—H15	0.9300	C25—H25	0.9300
C16—H16	0.9300	C26—H26	0.9300
O12—C18	1.423 (3)	O22—C28	1.423 (3)
C18—H18A	0.9600	C28—H28A	0.9600
C18—H18B	0.9600	C28—H28B	0.9600
C18—H18C	0.9600	C28—H28C	0.9600
N11—C112	1.382 (3)	N21—C212	1.373 (3)
N11—H11	0.8600	N21—H21	0.8600
S111—C112	1.714 (3)	S211—C212	1.720 (3)
S111—C17A	1.725 (3)	S211—C27A	1.722 (3)
C112—C113	1.376 (3)	C212—C213	1.382 (3)
C113—C13A	1.451 (3)	C213—C23A	1.444 (3)
C113—C137	1.459 (3)	C213—C237	1.472 (3)
C13A—C17A	1.357 (3)	C23A—C27A	1.356 (3)
C13A—C114	1.498 (3)	C23A—C214	1.505 (4)
C114—C115	1.512 (4)	C214—C215	1.516 (4)
C114—H11A	0.9700	C214—H21A	0.9700
C114—H11B	0.9700	C214—H21B	0.9700
C115—C116	1.490 (4)	C215—C216	1.501 (4)
C115—H11C	0.9700	C215—H21C	0.9700
C115—H11D	0.9700	C215—H21D	0.9700
C116—C117	1.525 (4)	C216—C217	1.512 (4)
C116—H11E	0.9700	C216—H21E	0.9700
C116—H11F	0.9700	C216—H21F	0.9700
C117—C17A	1.495 (4)	C217—C27A	1.500 (4)
C117—H11G	0.9700	C217—H21G	0.9700
C117—H11H	0.9700	C217—H21H	0.9700
C137—O137	1.227 (3)	C237—O237	1.227 (3)
C137—C131	1.489 (4)	C237—C231	1.486 (3)
C131—C132	1.379 (4)	C231—C236	1.378 (3)
C131—C136	1.380 (4)	C231—C232	1.390 (3)
C132—C133	1.366 (4)	C232—C233	1.374 (4)
C132—H132	0.9300	C232—H232	0.9300
C133—C134	1.363 (4)	C233—C234	1.373 (4)

C133—H133	0.9300	C233—H233	0.9300
C134—C135	1.370 (4)	C234—C235	1.379 (4)
C134—H134	0.9300	C234—H234	0.9300
C135—C136	1.385 (4)	C235—C236	1.378 (4)
C135—H135	0.9300	C235—H235	0.9300
C136—H136	0.9300	C236—H236	0.9300
O17—C17—N11	120.3 (3)	O27—C27—N21	120.1 (3)
O17—C17—C11	121.5 (3)	O27—C27—C21	121.0 (3)
N11—C17—C11	118.1 (3)	N21—C27—C21	118.9 (3)
C16—C11—C12	117.7 (3)	C22—C21—C26	118.1 (3)
C16—C11—C17	115.3 (3)	C22—C21—C27	125.8 (3)
C12—C11—C17	126.9 (3)	C26—C21—C27	116.0 (3)
O12—C12—C13	123.1 (3)	O22—C22—C23	123.1 (3)
O12—C12—C11	117.0 (2)	O22—C22—C21	117.0 (2)
C13—C12—C11	119.9 (3)	C23—C22—C21	119.9 (3)
C14—C13—C12	120.3 (3)	C24—C23—C22	120.3 (3)
C14—C13—H13	119.6 (18)	C24—C23—H23	119.8
C12—C13—H13	120.0 (18)	C22—C23—H23	119.8
C15—C14—C13	120.8 (3)	C25—C24—C23	120.8 (3)
C15—C14—H14	119.6	C25—C24—H24	119.6
C13—C14—H14	119.6	C23—C24—H24	119.6
C14—C15—C16	119.2 (3)	C24—C25—C26	119.6 (3)
C14—C15—H15	120.4	C24—C25—H25	120.2
C16—C15—H15	120.4	C26—C25—H25	120.2
C15—C16—C11	122.1 (3)	C25—C26—C21	121.2 (3)
C15—C16—H16	119.0	C25—C26—H26	119.4
C11—C16—H16	119.0	C21—C26—H26	119.4
C12—O12—C18	120.5 (2)	C22—O22—C28	119.9 (2)
O12—C18—H18A	109.5	O22—C28—H28A	109.5
O12—C18—H18B	109.5	O22—C28—H28B	109.5
H18A—C18—H18B	109.5	H28A—C28—H28B	109.5
O12—C18—H18C	109.5	O22—C28—H28C	109.5
H18A—C18—H18C	109.5	H28A—C28—H28C	109.5
H18B—C18—H18C	109.5	H28B—C28—H28C	109.5
C17—N11—C112	124.3 (2)	C27—N21—C212	125.1 (2)
C17—N11—H11	117.9	C27—N21—H21	117.5
C112—N11—H11	117.9	C212—N21—H21	117.5
C112—S111—C17A	90.94 (13)	C212—S211—C27A	91.15 (13)
C113—C112—N11	125.1 (2)	N21—C212—C213	125.4 (2)
C113—C112—S111	112.8 (2)	N21—C212—S211	122.28 (19)
N11—C112—S111	122.0 (2)	C213—C212—S211	112.33 (19)
C112—C113—C13A	111.4 (2)	C212—C213—C23A	111.3 (2)
C112—C113—C137	121.1 (2)	C212—C213—C237	120.3 (2)
C13A—C113—C137	127.5 (2)	C23A—C213—C237	128.4 (2)
C17A—C13A—C113	111.6 (2)	C27A—C23A—C213	112.2 (2)
C17A—C13A—C114	120.5 (2)	C27A—C23A—C214	120.3 (2)
C113—C13A—C114	127.8 (2)	C213—C23A—C214	127.4 (2)

C13A—C114—C115	112.2 (2)	C23A—C214—C215	111.5 (2)
C13A—C114—H11A	109.2	C23A—C214—H21A	109.3
C115—C114—H11A	109.2	C215—C214—H21A	109.3
C13A—C114—H11B	109.2	C23A—C214—H21B	109.3
C115—C114—H11B	109.2	C215—C214—H21B	109.3
H11A—C114—H11B	107.9	H21A—C214—H21B	108.0
C116—C115—C114	112.6 (3)	C216—C215—C214	111.2 (2)
C116—C115—H11C	109.1	C216—C215—H21C	109.4
C114—C115—H11C	109.1	C214—C215—H21C	109.4
C116—C115—H11D	109.1	C216—C215—H21D	109.4
C114—C115—H11D	109.1	C214—C215—H21D	109.4
H11C—C115—H11D	107.8	H21C—C215—H21D	108.0
C115—C116—C117	112.0 (3)	C215—C216—C217	112.1 (3)
C115—C116—H11E	109.2	C215—C216—H21E	109.2
C117—C116—H11E	109.2	C217—C216—H21E	109.2
C115—C116—H11F	109.2	C215—C216—H21F	109.2
C117—C116—H11F	109.2	C217—C216—H21F	109.2
H11E—C116—H11F	107.9	H21E—C216—H21F	107.9
C17A—C117—C116	109.6 (2)	C27A—C217—C216	110.3 (2)
C17A—C117—H11G	109.7	C27A—C217—H21G	109.6
C116—C117—H11G	109.7	C216—C217—H21G	109.6
C17A—C117—H11H	109.7	C27A—C217—H21H	109.6
C116—C117—H11H	109.7	C216—C217—H21H	109.6
H11G—C117—H11H	108.2	H21G—C217—H21H	108.1
C13A—C17A—C117	126.4 (3)	C23A—C27A—C217	126.1 (3)
C13A—C17A—S111	113.2 (2)	C23A—C27A—S211	112.9 (2)
C117—C17A—S111	120.3 (2)	C217—C27A—S211	121.0 (2)
O137—C137—C113	121.6 (2)	O237—C237—C213	120.9 (2)
O137—C137—C131	117.3 (2)	O237—C237—C231	118.6 (2)
C113—C137—C131	121.0 (2)	C213—C237—C231	120.4 (2)
C132—C131—C136	119.0 (3)	C236—C231—C232	119.1 (2)
C132—C131—C137	119.4 (2)	C236—C231—C237	121.2 (2)
C136—C131—C137	121.4 (2)	C232—C231—C237	119.5 (2)
C133—C132—C131	120.5 (3)	C233—C232—C231	120.5 (3)
C133—C132—H132	119.7	C233—C232—H232	119.8
C131—C132—H132	119.7	C231—C232—H232	119.8
C134—C133—C132	120.6 (3)	C234—C233—C232	119.9 (3)
C134—C133—H133	119.7	C234—C233—H233	120.1
C132—C133—H133	119.7	C232—C233—H233	120.1
C133—C134—C135	120.1 (3)	C233—C234—C235	120.2 (3)
C133—C134—H134	120.0	C233—C234—H234	119.9
C135—C134—H134	120.0	C235—C234—H234	119.9
C134—C135—C136	119.8 (3)	C236—C235—C234	119.9 (3)
C134—C135—H135	120.1	C236—C235—H235	120.1
C136—C135—H135	120.1	C234—C235—H235	120.1
C131—C136—C135	120.1 (3)	C231—C236—C235	120.5 (3)
C131—C136—H136	119.9	C231—C236—H236	119.8
C135—C136—H136	119.9	C235—C236—H236	119.8

O17—C17—C11—C16	3.8 (4)	O27—C27—C21—C22	−173.8 (3)
N11—C17—C11—C16	−177.2 (2)	N21—C27—C21—C22	5.0 (4)
O17—C17—C11—C12	−173.4 (3)	O27—C27—C21—C26	3.6 (4)
N11—C17—C11—C12	5.6 (4)	N21—C27—C21—C26	−177.7 (2)
C16—C11—C12—O12	179.3 (2)	C26—C21—C22—O22	179.3 (2)
C17—C11—C12—O12	−3.6 (4)	C27—C21—C22—O22	−3.4 (4)
C16—C11—C12—C13	−1.6 (4)	C26—C21—C22—C23	−0.8 (4)
C17—C11—C12—C13	175.5 (3)	C27—C21—C22—C23	176.5 (2)
O12—C12—C13—C14	−179.0 (3)	O22—C22—C23—C24	−179.3 (3)
C11—C12—C13—C14	1.9 (4)	C21—C22—C23—C24	0.8 (4)
C12—C13—C14—C15	−0.8 (5)	C22—C23—C24—C25	−0.5 (5)
C13—C14—C15—C16	−0.6 (5)	C23—C24—C25—C26	0.3 (5)
C14—C15—C16—C11	0.9 (4)	C24—C25—C26—C21	−0.3 (5)
C12—C11—C16—C15	0.2 (4)	C22—C21—C26—C25	0.5 (4)
C17—C11—C16—C15	−177.3 (2)	C27—C21—C26—C25	−177.0 (3)
C13—C12—O12—C18	−2.0 (4)	C23—C22—O22—C28	0.7 (4)
C11—C12—O12—C18	177.1 (2)	C21—C22—O22—C28	−179.3 (2)
O17—C17—N11—C112	2.4 (4)	O27—C27—N21—C212	0.6 (4)
C11—C17—N11—C112	−176.7 (2)	C21—C27—N21—C212	−178.2 (2)
C17—N11—C112—C113	175.4 (2)	C27—N21—C212—C213	176.4 (2)
C17—N11—C112—S111	−1.9 (3)	C27—N21—C212—S211	−2.5 (3)
C17A—S111—C112—C113	2.9 (2)	C27A—S211—C212—N21	−177.5 (2)
C17A—S111—C112—N11	−179.6 (2)	C27A—S211—C212—C213	3.48 (19)
N11—C112—C113—C13A	179.3 (2)	N21—C212—C213—C23A	177.6 (2)
S111—C112—C113—C13A	−3.2 (3)	S211—C212—C213—C23A	−3.4 (3)
N11—C112—C113—C137	−1.6 (4)	N21—C212—C213—C237	−3.5 (4)
S111—C112—C113—C137	175.89 (18)	S211—C212—C213—C237	175.46 (17)
C112—C113—C13A—C17A	1.9 (3)	C212—C213—C23A—C27A	1.4 (3)
C137—C113—C13A—C17A	−177.2 (2)	C237—C213—C23A—C27A	−177.3 (2)
C112—C113—C13A—C114	−173.7 (2)	C212—C213—C23A—C214	−174.1 (2)
C137—C113—C13A—C114	7.3 (4)	C237—C213—C23A—C214	7.1 (4)
C17A—C13A—C114—C115	−13.8 (4)	C27A—C23A—C214—C215	−16.8 (4)
C113—C13A—C114—C115	161.4 (2)	C213—C23A—C214—C215	158.4 (2)
C13A—C114—C115—C116	44.5 (3)	C23A—C214—C215—C216	48.3 (3)
C114—C115—C116—C117	−61.3 (4)	C214—C215—C216—C217	−63.0 (3)
C115—C116—C117—C17A	43.5 (3)	C215—C216—C217—C27A	41.9 (3)
C113—C13A—C17A—C117	−177.0 (2)	C213—C23A—C27A—C217	−178.1 (2)
C114—C13A—C17A—C117	−1.1 (4)	C214—C23A—C27A—C217	−2.1 (4)
C113—C13A—C17A—S111	0.3 (3)	C213—C23A—C27A—S211	1.2 (3)
C114—C13A—C17A—S111	176.23 (19)	C214—C23A—C27A—S211	177.12 (19)
C116—C117—C17A—C13A	−13.7 (4)	C216—C217—C27A—C23A	−10.3 (4)
C116—C117—C17A—S111	169.2 (2)	C216—C217—C27A—S211	170.5 (2)
C112—S111—C17A—C13A	−1.8 (2)	C212—S211—C27A—C23A	−2.7 (2)
C112—S111—C17A—C117	175.7 (2)	C212—S211—C27A—C217	176.7 (2)
C112—C113—C137—O137	23.8 (4)	C212—C213—C237—O237	29.9 (4)
C13A—C113—C137—O137	−157.3 (3)	C23A—C213—C237—O237	−151.4 (2)
C112—C113—C137—C131	−152.9 (2)	C212—C213—C237—C231	−145.6 (2)

C13A—C113—C137—C131	26.1 (4)	C23A—C213—C237—C231	33.0 (4)
O137—C137—C131—C132	42.1 (4)	O237—C237—C231—C236	−141.3 (3)
C113—C137—C131—C132	−141.1 (3)	C213—C237—C231—C236	34.3 (4)
O137—C137—C131—C136	−132.0 (3)	O237—C237—C231—C232	33.6 (4)
C113—C137—C131—C136	44.8 (4)	C213—C237—C231—C232	−150.8 (2)
C136—C131—C132—C133	−0.2 (4)	C236—C231—C232—C233	−0.1 (4)
C137—C131—C132—C133	−174.4 (3)	C237—C231—C232—C233	−175.1 (2)
C131—C132—C133—C134	1.0 (5)	C231—C232—C233—C234	0.9 (4)
C132—C133—C134—C135	−0.4 (5)	C232—C233—C234—C235	−0.8 (4)
C133—C134—C135—C136	−0.9 (5)	C233—C234—C235—C236	−0.1 (4)
C132—C131—C136—C135	−1.2 (4)	C232—C231—C236—C235	−0.7 (4)
C137—C131—C136—C135	173.0 (2)	C237—C231—C236—C235	174.1 (2)
C134—C135—C136—C131	1.7 (4)	C234—C235—C236—C231	0.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O12	0.86	1.95	2.630 (3)	136
N11—H11···O137	0.86	2.11	2.731 (3)	128
N21—H21···O22	0.86	1.93	2.616 (3)	136
N21—H21···O237	0.86	2.13	2.748 (3)	129
C134—H134···O27	0.93	2.49	3.232 (4)	137
C235—H235···*Cg*3^{i}	0.93	2.95	3.869 (3)	170

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

2-Amino-3-benzoyl-4,5,6,7-tetrahydrobenzo[*b*]thiophene (VI)

Crystal data

$C_{15}H_{15}NOS$
M_r = 257.34
Orthorhombic, $Pna2_1$
a = 9.2080 (4) Å
b = 14.0485 (7) Å
c = 10.3826 (6) Å
V = 1343.08 (12) Å^3
Z = 4
F(000) = 544
D_x = 1.273 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.7107 Å
Cell parameters from 3556 reflections
θ = 3.3–28.2°
μ = 0.23 mm^{-1}
T = 295 K
Block, colourless
0.30 × 0.15 × 0.15 mm

Data collection

Agilent Eos Gemini diffractometer
Radiation source: fine focus sealed tube
φ and ω scans
Absorption correction: multi-scan CrysAlis RED (Agilent, 2012)
T_{min} = 0.948, T_{max} = 0.966
3556 measured reflections
1984 independent reflections
1720 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 28.2°, θ_{min} = 3.3°
h = −11→11
k = −7→17
l = −8→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.086$
$S = 1.04$
1984 reflections
176 parameters
13 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.1113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack *x* determined using 422 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.03 (6)

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)
S1	−0.01074 (7)	0.60683 (5)	0.43072 (12)	0.0549 (2)	
C2	0.1557 (3)	0.6376 (2)	0.3673 (3)	0.0439 (7)	
C3	0.2490 (3)	0.55885 (19)	0.3594 (3)	0.0377 (6)	
C3A	0.1817 (3)	0.47431 (19)	0.4150 (3)	0.0397 (6)	0.887 (9)
C4	0.2556 (5)	0.3800 (4)	0.4395 (11)	0.0546 (9)	0.887 (9)
H4A	0.3549	0.3910	0.4671	0.066*	0.887 (9)
H4B	0.2583	0.3434	0.3604	0.066*	0.887 (9)
C5	0.1742 (4)	0.3240 (3)	0.5432 (6)	0.0645 (15)	0.887 (9)
H5A	0.2120	0.2596	0.5470	0.077*	0.887 (9)
H5B	0.1900	0.3536	0.6265	0.077*	0.887 (9)
C6	0.0132 (4)	0.3210 (3)	0.5147 (6)	0.0682 (16)	0.887 (9)
H6A	−0.0348	0.2788	0.5751	0.082*	0.887 (9)
H6B	−0.0025	0.2966	0.4284	0.082*	0.887 (9)
C7	−0.0508 (5)	0.4208 (4)	0.5257 (10)	0.0624 (10)	0.887 (9)
H7A	−0.1476	0.4218	0.4890	0.075*	0.887 (9)
H7B	−0.0576	0.4390	0.6156	0.075*	0.887 (9)
C7A	0.0453 (3)	0.4895 (2)	0.4549 (3)	0.0458 (7)	0.887 (9)
C23A	0.1817 (3)	0.47431 (19)	0.4150 (3)	0.0397 (6)	0.113 (9)
C24	0.260 (3)	0.383 (2)	0.445 (7)	0.0546 (9)	0.113 (9)
H24A	0.3259	0.3927	0.5170	0.066*	0.113 (9)
H24B	0.3169	0.3630	0.3714	0.066*	0.113 (9)
C25	0.151 (3)	0.3044 (10)	0.480 (3)	0.0645 (15)	0.113 (9)
H25A	0.0994	0.2847	0.4029	0.077*	0.113 (9)
H25B	0.2024	0.2496	0.5138	0.077*	0.113 (9)
C26	0.044 (3)	0.3399 (15)	0.579 (3)	0.0682 (16)	0.113 (9)
H26A	0.0954	0.3645	0.6531	0.082*	0.113 (9)
H26B	−0.0173	0.2876	0.6074	0.082*	0.113 (9)
C27	−0.051 (3)	0.418 (2)	0.521 (7)	0.0624 (10)	0.113 (9)

H27A	−0.1184	0.3909	0.4597	0.075*	0.113 (9)
H27B	−0.1063	0.4492	0.5885	0.075*	0.113 (9)
C27A	0.0453 (3)	0.4895 (2)	0.4549 (3)	0.0458 (7)	0.113 (9)
N2	0.1839 (3)	0.72731 (17)	0.3320 (3)	0.0586 (7)	
H2A	0.2674	0.7418	0.3005	0.070*	
H2B	0.1185	0.7705	0.3410	0.070*	
C37	0.3896 (3)	0.5705 (2)	0.3022 (3)	0.0444 (7)	
O37	0.4441 (2)	0.65081 (16)	0.2880 (3)	0.0665 (7)	
C31	0.4730 (3)	0.4869 (2)	0.2516 (3)	0.0448 (7)	
C32	0.6207 (3)	0.4802 (3)	0.2752 (4)	0.0637 (10)	
H32	0.6668	0.5258	0.3256	0.076*	
C33	0.6992 (4)	0.4058 (3)	0.2237 (5)	0.0843 (13)	
H33	0.7981	0.4007	0.2404	0.101*	
C34	0.6313 (5)	0.3388 (3)	0.1474 (5)	0.0834 (13)	
H34	0.6842	0.2882	0.1137	0.100*	
C35	0.4867 (4)	0.3466 (3)	0.1214 (4)	0.0726 (11)	
H35	0.4416	0.3025	0.0679	0.087*	
C36	0.4079 (4)	0.4196 (2)	0.1740 (4)	0.0573 (8)	
H36	0.3089	0.4239	0.1571	0.069*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0484 (3)	0.0496 (4)	0.0667 (5)	0.0123 (3)	0.0063 (4)	0.0006 (5)
C2	0.0470 (15)	0.0387 (15)	0.0459 (16)	−0.0003 (13)	−0.0054 (14)	−0.0016 (14)
C3	0.0396 (13)	0.0323 (13)	0.0411 (15)	0.0012 (12)	−0.0030 (11)	−0.0008 (13)
C3A	0.0426 (11)	0.0355 (13)	0.0409 (16)	0.0002 (11)	0.0000 (13)	0.0013 (14)
C4	0.0520 (14)	0.0404 (16)	0.071 (2)	0.0046 (12)	0.0094 (19)	0.0110 (19)
C5	0.067 (2)	0.051 (2)	0.076 (4)	0.0078 (19)	0.013 (2)	0.024 (2)
C6	0.066 (2)	0.054 (2)	0.084 (4)	−0.0099 (19)	0.019 (2)	0.021 (3)
C7	0.0488 (16)	0.067 (2)	0.071 (2)	0.0030 (16)	0.0158 (16)	0.016 (2)
C7A	0.0434 (12)	0.0446 (16)	0.0494 (19)	0.0010 (11)	0.0033 (14)	0.0052 (15)
C23A	0.0426 (11)	0.0355 (13)	0.0409 (16)	0.0002 (11)	0.0000 (13)	0.0013 (14)
C24	0.0520 (14)	0.0404 (16)	0.071 (2)	0.0046 (12)	0.0094 (19)	0.0110 (19)
C25	0.067 (2)	0.051 (2)	0.076 (4)	0.0078 (19)	0.013 (2)	0.024 (2)
C26	0.066 (2)	0.054 (2)	0.084 (4)	−0.0099 (19)	0.019 (2)	0.021 (3)
C27	0.0488 (16)	0.067 (2)	0.071 (2)	0.0030 (16)	0.0158 (16)	0.016 (2)
C27A	0.0434 (12)	0.0446 (16)	0.0494 (19)	0.0010 (11)	0.0033 (14)	0.0052 (15)
N2	0.0606 (14)	0.0332 (14)	0.082 (2)	0.0061 (12)	0.0000 (14)	0.0055 (15)
C37	0.0420 (13)	0.0399 (16)	0.0514 (17)	−0.0059 (13)	−0.0039 (13)	0.0056 (15)
O37	0.0547 (12)	0.0414 (13)	0.104 (2)	−0.0117 (11)	0.0080 (13)	0.0090 (14)
C31	0.0467 (14)	0.0439 (17)	0.0439 (17)	0.0007 (13)	0.0076 (13)	0.0072 (15)
C32	0.0475 (15)	0.074 (2)	0.070 (3)	0.0010 (16)	0.0045 (16)	0.000 (2)
C33	0.058 (2)	0.099 (3)	0.096 (3)	0.025 (2)	0.021 (2)	0.013 (3)
C34	0.101 (3)	0.071 (3)	0.078 (3)	0.021 (2)	0.038 (3)	−0.002 (2)
C35	0.091 (3)	0.065 (3)	0.062 (2)	0.000 (2)	0.018 (2)	−0.014 (2)
C36	0.0590 (17)	0.059 (2)	0.0542 (19)	−0.0043 (16)	0.0060 (17)	−0.0061 (18)

Geometric parameters (Å, º)

S1—C2	1.723 (3)	C25—C26	1.511 (8)
S1—C7A	1.745 (3)	C25—H25A	0.9700
C2—N2	1.338 (4)	C25—H25B	0.9700
C2—C3	1.403 (4)	C26—C27	1.524 (9)
C3—C37	1.433 (4)	C26—H26A	0.9700
C3—C3A	1.459 (4)	C26—H26B	0.9700
C3A—C7A	1.339 (3)	C27—H27A	0.9700
C3A—C4	1.511 (4)	C27—H27B	0.9700
C4—C5	1.531 (7)	N2—H2A	0.8600
C4—H4A	0.9700	N2—H2B	0.8600
C4—H4B	0.9700	C37—O37	1.244 (3)
C5—C6	1.513 (6)	C37—C31	1.498 (4)
C5—H5A	0.9700	C31—C36	1.379 (4)
C5—H5B	0.9700	C31—C32	1.386 (4)
C6—C7	1.524 (7)	C32—C33	1.379 (5)
C6—H6A	0.9700	C32—H32	0.9300
C6—H6B	0.9700	C33—C34	1.379 (6)
C7—C7A	1.502 (4)	C33—H33	0.9300
C7—H7A	0.9700	C34—C35	1.363 (5)
C7—H7B	0.9700	C34—H34	0.9300
C24—C25	1.531 (9)	C35—C36	1.370 (5)
C24—H24A	0.9700	C35—H35	0.9300
C24—H24B	0.9700	C36—H36	0.9300
C2—S1—C7A	91.63 (13)	C26—C25—C24	110.6 (11)
N2—C2—C3	127.5 (3)	C26—C25—H25A	109.5
N2—C2—S1	120.9 (2)	C24—C25—H25A	109.5
C3—C2—S1	111.7 (2)	C26—C25—H25B	109.5
C2—C3—C37	119.2 (3)	C24—C25—H25B	109.5
C2—C3—C3A	111.0 (2)	H25A—C25—H25B	108.1
C37—C3—C3A	129.8 (2)	C25—C26—C27	110.1 (11)
C7A—C3A—C3	113.0 (2)	C25—C26—H26A	109.7
C7A—C3A—C4	120.7 (3)	C27—C26—H26A	109.7
C3—C3A—C4	126.1 (2)	C25—C26—H26B	109.7
C3A—C4—C5	110.4 (4)	C27—C26—H26B	109.7
C3A—C4—H4A	109.6	H26A—C26—H26B	108.2
C5—C4—H4A	109.6	C26—C27—H27A	109.9
C3A—C4—H4B	109.6	C26—C27—H27B	109.9
C5—C4—H4B	109.6	H27A—C27—H27B	108.3
H4A—C4—H4B	108.1	C2—N2—H2A	120.0
C6—C5—C4	110.9 (6)	C2—N2—H2B	120.0
C6—C5—H5A	109.5	H2A—N2—H2B	120.0
C4—C5—H5A	109.5	O37—C37—C3	121.1 (3)
C6—C5—H5B	109.5	O37—C37—C31	117.5 (3)
C4—C5—H5B	109.5	C3—C37—C31	121.3 (3)
H5A—C5—H5B	108.1	C36—C31—C32	118.9 (3)

C5—C6—C7	109.8 (6)	C36—C31—C37	121.3 (3)
C5—C6—H6A	109.7	C32—C31—C37	119.6 (3)
C7—C6—H6A	109.7	C33—C32—C31	119.8 (4)
C5—C6—H6B	109.7	C33—C32—H32	120.1
C7—C6—H6B	109.7	C31—C32—H32	120.1
H6A—C6—H6B	108.2	C34—C33—C32	120.1 (4)
C7A—C7—C6	109.1 (4)	C34—C33—H33	119.9
C7A—C7—H7A	109.9	C32—C33—H33	119.9
C6—C7—H7A	109.9	C35—C34—C33	120.1 (4)
C7A—C7—H7B	109.9	C35—C34—H34	119.9
C6—C7—H7B	109.9	C33—C34—H34	119.9
H7A—C7—H7B	108.3	C34—C35—C36	119.9 (4)
C3A—C7A—C7	126.9 (3)	C34—C35—H35	120.0
C3A—C7A—S1	112.6 (2)	C36—C35—H35	120.0
C7—C7A—S1	120.2 (2)	C35—C36—C31	121.1 (3)
C25—C24—H24A	109.6	C35—C36—H36	119.5
C25—C24—H24B	109.6	C31—C36—H36	119.5
H24A—C24—H24B	108.1		

C7A—S1—C2—N2	−177.3 (3)	C6—C7—C7A—S1	171.3 (5)
C7A—S1—C2—C3	2.7 (2)	C2—S1—C7A—C3A	−1.0 (3)
N2—C2—C3—C37	−3.4 (5)	C2—S1—C7A—C7	173.3 (6)
S1—C2—C3—C37	176.6 (2)	C24—C25—C26—C27	66.1 (18)
N2—C2—C3—C3A	176.4 (3)	C2—C3—C37—O37	17.5 (4)
S1—C2—C3—C3A	−3.7 (3)	C3A—C3—C37—O37	−162.2 (3)
C2—C3—C3A—C7A	3.0 (4)	C2—C3—C37—C31	−159.5 (3)
C37—C3—C3A—C7A	−177.3 (3)	C3A—C3—C37—C31	20.8 (5)
C2—C3—C3A—C4	−171.5 (6)	O37—C37—C31—C36	−130.1 (3)
C37—C3—C3A—C4	8.2 (8)	C3—C37—C31—C36	47.0 (5)
C7A—C3A—C4—C5	−16.5 (11)	O37—C37—C31—C32	45.3 (4)
C3—C3A—C4—C5	157.6 (5)	C3—C37—C31—C32	−137.6 (3)
C3A—C4—C5—C6	49.0 (11)	C36—C31—C32—C33	−1.5 (5)
C4—C5—C6—C7	−66.4 (11)	C37—C31—C32—C33	−177.0 (3)
C5—C6—C7—C7A	46.7 (11)	C31—C32—C33—C34	0.9 (6)
C3—C3A—C7A—C7	−174.7 (6)	C32—C33—C34—C35	0.8 (6)
C4—C3A—C7A—C7	0.1 (10)	C33—C34—C35—C36	−1.8 (7)
C3—C3A—C7A—S1	−0.9 (3)	C34—C35—C36—C31	1.2 (6)
C4—C3A—C7A—S1	173.9 (6)	C32—C31—C36—C35	0.5 (6)
C6—C7—C7A—C3A	−15.3 (11)	C37—C31—C36—C35	175.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O37	0.86	2.07	2.666 (3)	125
N2—H2*B*···O37^{i}	0.86	2.03	2.831 (3)	156

Symmetry code: (i) *x*−1/2, −*y*+3/2, *z*.