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Synthesis and structures of six closely related *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]-arylamides, together with an isolated reaction intermediate: order *versus* disorder, molecular conformations and hydrogen bonding in zero, one and two dimensions

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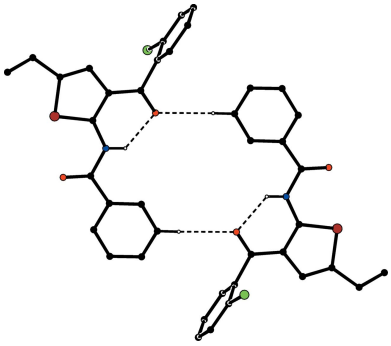
Six closely related *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]arylamides have been synthesized and structurally characterized, together with a representative reaction intermediate. In each of *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]benzamide, $C_{20}H_{16}ClNO_2S$, (I), *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]-4-phenylbenzamide, $C_{26}H_{20}ClNO_2S$, (II), and 2-bromo-*N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]benzamide, $C_{20}H_{15}BrClNO_2S$, (III), the molecules are disordered over two sets of atomic sites, with occupancies of 0.894 (8) and 0.106 (8) in (I), 0.832 (5) and 0.168 (5) in (II), and 0.7006 (12) and 0.2994 (12) in (III). In each of *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]-2-iodobenzamide, $C_{20}H_{15}ClINO_2S$, (IV), and *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]-2-methoxybenzamide, $C_{21}H_{18}ClNO_3S$, (V), the molecules are fully ordered, but in *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]-2,6-difluorobenzamide, $C_{20}H_{14}ClF_2NO_2S$, (VI), which crystallizes with $Z' = 2$ in the space group $C2/c$, one of the two independent molecules is fully ordered, while the other is disordered over two sets of atomic sites having occupancies of 0.916 (3) and 0.084 (3). All of the molecules in compounds (I)–(VI) exhibit an intramolecular N—H···O hydrogen bond. The molecules of (I) and (VI) are linked by C—H···O hydrogen bonds to form finite zero-dimensional dimers, which are cyclic in (I) and acyclic in (VI), those of (III) are linked by C—H···π(arene) hydrogen bonds to form simple chains, and those of (IV) and (V) are linked into different types of chains of rings, built in each case from a combination of C—H···O and C—H···π(arene) hydrogen bonds. Two C—H···O hydrogen bonds link the molecules of (II) into sheets containing three types of ring. In benzotriazol-1-yl 3,4-dimethoxybenzoate, $C_{15}H_{13}N_3O_4$, (VII), the benzoate component is planar and makes a dihedral angle of 84.51 (6)° with the benzotriazole unit. Comparisons are made with related compounds.



1. Introduction

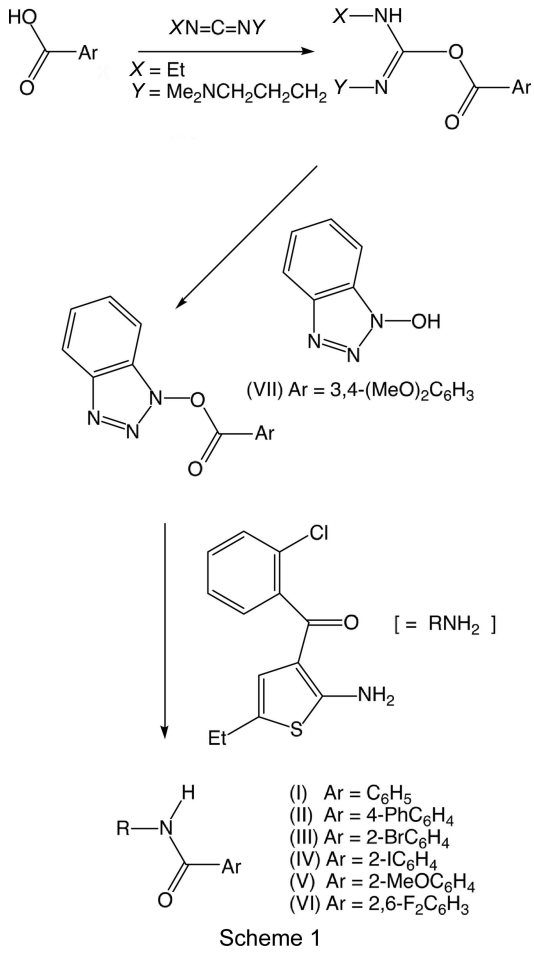
2-Amino-3-benzoylthiophenes can act (Aurelio *et al.*, 2010) as allosteric enhancers at the A_1 adenosine receptor, whose role in obesity and diabetes has been reviewed fairly recently (Dhalia *et al.*, 2009). More broadly, thiophene derivatives have been found to exhibit a wide range of biological properties, 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 nitric-oxide-

scavenging activity (Molvi *et al.*, 2007). Based on these observations, particularly the first, we have now synthesized and characterized a series of closely related arylamides derived from 2-amino-3-(2-chlorobenzoyl)-5-ethylthiophene, namely *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]benzamide, (I), *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]-4-phenylbenzamide, (II), 2-bromo-*N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]benzamide, (III), *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]-2-iodobenzamide, (IV), *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]-2-methoxybenzamide, (V), and *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]-2,6-difluorobenzamide, (VI). Compounds (I)–(VI) were all prepared using condensation reactions between 2-amino-3-(2-chlorobenzoyl)-5-ethylthiophene and a substituted benzoic acid in the presence of 1-[3-(dimethylamino)propyl]-3-ethylcarbodiimide as dehydrating agent and 1-hydroxybenzotriazole as the acyl carrier (König & Geiger, 1970) (see Scheme 1). We have also isolated and characterized the reaction intermediate benzotriazol-1-yl 3,4-dimethoxybenzoate, (VII), by the straightforward expedient of stopping the reaction sequence before the addition of the aminothiophene reactant, confirming this aspect of the proposed mechanism. We report here the molecular and supramolecular structures of compounds (I)–(VII).

2. Experimental

2.1. Synthesis and crystallization

2-Amino-3-(2-chlorobenzoyl)-5-ethylthiophene 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)–(VI), 1-[3-(dimethylamino)propyl]-3-ethylcarbodiimide (173 mg, 0.9 mmol), 1-hydroxybenzotriazole (126.32 mg, 0.825 mmol) and triethylamine (0.5 ml, 3.75 mmol) were added to a solution of the appropriately substituted benzoic acid (0.75 mmol) (see Scheme 1) in *N*,*N*-dimethylformamide (DMF, 5 ml) [*i.e.* benzoic acid for (I), biphenyl-4-carboxylic acid for (II), 2-bromobenzoic acid for (III), 2-iodobenzoic acid for (IV), 2-methoxybenzoic acid for (V) and 2,6-difluorobenzoic acid for (VI)] at 273 K and the resulting mixtures were stirred for 20 min at 273 K. A solution of 2-amino-3-(2-chlorobenzoyl)-5-ethylthiophene (200 mg, 0.75 mmol) in DMF (5 ml) was then added to each of the above mixtures and stirring was continued overnight at ambient temperature. The reactions were then judged to be complete using thin-layer chromatography (TLC). The mixtures were poured into an excess of water and extracted exhaustively with ethyl acetate. For each, the organic extract was washed first with aqueous hydrochloric acid solution (1 mol dm^{-3}) and then with brine, and then dried over anhydrous sodium sulfate and concentrated under reduced pressure. The crude products of (I)–(VI) were purified using silica-gel column chromatography (60:120 mesh) using ethyl acetate–hexane (1:4 *v*/*v*) as eluent and 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 DMF. An entirely similar procedure, using 3,4-dimethoxybenzoic acid, but omitting the addition of 2-amino-3-(2-chlorobenzoyl)-5-ethylthiophene, yielded (VII).



Scheme 1

2.1.1. Analytical data. Compound (I): yield 85%, m.p. 433 K; ^{1}H NMR ($CDCl_3$): δ 12.99 (*s*, NH), 8.11–8.09 (*m*, 2H, Ar—H), 7.60–7.37 (*m*, 7H, Ar—H), 6.42 (*s*, 1H, thiophene), 2.71 (*q*, J = 7.4 Hz, 2H, CH_2), 1.25 (*t*, J = 7.4 Hz, 3H, CH_3). Compound (II): yield 85%, m.p. 462–464 K; ^{1}H NMR ($CDCl_3$): δ 13.04 (*s*, NH), 8.18 (*m*, 2H, Ar—H), 7.50–7.38 (*m*, 11H, Ar—H), 6.43 (*s*, 1H, thiophene), 2.72 (*q*, J = 7.6 Hz, 2H, CH_2), 1.27 (*t*, J = 7.6 Hz, 3H, CH_3). Compound (III): yield 86%, m.p. 373 K; ^{1}H NMR ($CDCl_3$): δ 12.11 (*s*, NH), 7.81–7.75 (*m*, 2H, Ar—H), 7.59–7.47 (*m*, 6H, Ar—H), 6.45 (*s*, 1H, thiophene), 2.69 (*q*, J = 7.4 Hz, 2H, CH_2), 1.15 (*t*, J = 7.4 Hz, 3H, CH_3). Compound (IV): yield 82%, m.p. 403–405 K; ^{1}H NMR ($CDCl_3$): δ 12.32 (*s*, NH), 8.00–7.98 (*m*, 1H, Ar—H), 7.66–7.64 (*m*, 1H, Ar—H), 7.49–7.20 (*m*, 6H, Ar—H), 6.43 (*s*, 1H, thiophene), 2.73–2.70 (*q*, J = 7.6 Hz, 2H, CH_2), 1.26 (*t*, J = 7.6 Hz, 3H, CH_3). Compound (V): yield 83%, m.p. 430–432 K; ^{1}H NMR ($CDCl_3$): δ 12.64 (*s*, NH), 7.96–7.93 (*m*, 2H, Ar—H), 7.61–7.48 (*m*, 4H, Ar—H), 7.19–7.16 (*m*, 2H, Ar—H), 6.39 (*s*, 1H, thiophene), 2.68 (*q*, J = 7.6 Hz, 2H, CH_2), 1.14 (*t*, J = 7.6 Hz, CH_3). Compound (VI): yield 60%, m.p. 410–414 K; ^{1}H NMR ($CDCl_3$): δ 12.82 (*s*, NH), 7.89–7.77 (*m*, 2H, Ar—H), 7.56–6.45 (*m*, 5H, Ar—H), 6.34 (*s*, 1H, thiophene), 2.69 (*q*, J = 7.6 Hz, 2H, CH_2), 1.26 (*t*, J = 7.6 Hz, CH_3). Compound (VII): yield 86%, m.p. 435 K; ^{1}H NMR ($CDCl_3$): δ 8.10–8.08 (*m*, 1H,

Table 1
Experimental details.

	(I)	(II)	(III)	(IV)
Crystal data				
Chemical formula	$C_{20}H_{16}ClNO_2S$	$C_{26}H_{20}ClNO_2S$	$C_{20}H_{15}BrClNO_2S$	$C_{20}H_{15}ClINO_2S$
M_r	369.85	445.94	448.74	495.74
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$	Monoclinic, $C2/c$	Monoclinic, $P2_1/n$
Temperature (K)	294	296	294	294
a, b, c (Å)	9.7868 (9), 13.9567 (12), 13.3764 (11)	10.7264 (6), 9.1208 (5), 22.9103 (15)	18.8096 (10), 11.5021 (7), 19.9112 (11)	8.1813 (6), 11.8276 (7), 20.1347 (12)
α, β, γ (°)	90, 101.816 (2), 90	90, 94.414 (2), 90	90, 118.030 (2), 90	90, 94.388 (2), 90
V (Å^3)	1788.4 (3)	2234.7 (2)	3802.5 (4)	1942.6 (2)
Z	4	4	8	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.34	0.29	2.43	1.91
Crystal size (mm)	0.25 × 0.20 × 0.20	0.28 × 0.27 × 0.22	0.25 × 0.20 × 0.10	0.16 × 0.14 × 0.11
Data collection				
Diffractometer	Bruker Kappa APEXII	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)	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.864, 0.934	0.929, 0.939	0.614, 0.785	0.722, 0.811
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	26489, 3726, 2092	24401, 5148, 3570	25510, 4009, 2370	20297, 3860, 2525
R_{int}	0.058	0.033	0.047	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629	0.651	0.635	0.620
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.131, 1.00	0.047, 0.130, 1.04	0.045, 0.122, 1.01	0.045, 0.128, 1.02
No. of reflections	3726	5148	4009	3860
No. of parameters	304	373	322	236
No. of restraints	72	89	64	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.26	0.39, −0.35	0.40, −0.50	0.53, −0.71

	(V)	(VI)	(VII)
Crystal data			
Chemical formula	$C_{21}H_{18}ClNO_3S$	$C_{20}H_{14}ClF_2NO_2S$	$C_{15}H_{13}N_3O_4$
M_r	399.87	405.83	299.28
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $C2/c$	Triclinic, $P\overline{1}$
Temperature (K)	294	294	294
a, b, c (Å)	7.4798 (3), 11.4237 (5), 12.0933 (5)	58.0164 (10), 7.8002 (2), 16.1365 (3)	8.1296 (6), 9.5899 (6), 10.8824 (8)
α, β, γ (°)	105.814 (2), 101.959 (3), 99.187 (3)	90, 92.105 (1), 90	66.840 (4), 71.533 (4), 71.350 (4)
V (Å^3)	946.66 (7)	7297.5 (3)	721.10 (9)
Z	2	16	2
Radiation type	Mo $K\alpha$	Cu $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.33	3.24	0.10
Crystal size (mm)	0.25 × 0.20 × 0.15	0.15 × 0.15 × 0.10	0.15 × 0.15 × 0.10
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}	0.908, 0.951	0.601, 0.753	0.921, 0.990
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20919, 3959, 2406	42600, 6468, 3979	15518, 2992, 1667
R_{int}	0.047	0.109	0.051
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629	0.596	0.629
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.121, 1.03	0.066, 0.159, 1.03	0.052, 0.156, 1.03
No. of reflections	3959	6468	2992
No. of parameters	246	572	201
No. of restraints	0	70	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.30	0.35, −0.43	0.17, −0.21

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

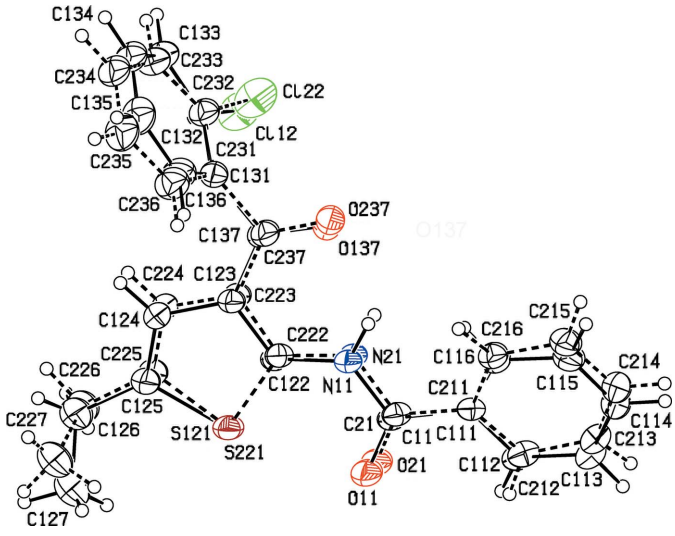


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme and the disorder. The major-disorder component is drawn with full lines and the minor-disorder component is drawn with broken lines. Displacement ellipsoids are drawn at the 30% probability level.

Ar—H), 7.98–7.96 (*m*, 1H, Ar—H), 7.68–7.67 (*m*, 1H, Ar—H), 7.54–7.42 (*m*, 3H, Ar—H), 7.03–7.01 (*m*, 1H, Ar—H), 4.01 (*s*, 3H, –OCH_3), 3.97 (*s*, 3H, –OCH_3).

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. It was apparent from an early stage in the refinements of compounds (I)–(III) that the molecules were disordered over two sets of atomic sites having different occupancies, as was one of the two independent molecules in (VI). For the minor-disorder forms, the bond lengths and 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 of the corresponding pairs of atoms in the two 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 and thienyl), 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, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. Subject to these conditions, the refined occupancies of the two disorder forms were 0.894 (8) and 0.106 (8) in (I), 0.832 (5) and 0.168 (5) in (II), 0.7006 (12) and 0.2994 (12) in (III), and 0.916 (3) and 0.084 (3) in molecule 2 of compound (VI). One low-angle reflection, *i.e.* 001, which had been attenuated by the beam stop was omitted from the final refinement for (V). The 002 reflection was omitted from the refinement of (II). In the final analyses of variance for (I)–(V) and (VII), there were negative values of K = [mean(F_o^2)/mean(F_c^2)] for the groups of the very weakest reflections having low values of F_c/F_c(max): in (I), −4.417 for 441 reflections in the F_c/F_c(max) range 0.000–0.005; in (II), −4.457 for 532 reflections in the F_c/F_c(max) range 0.000–0.004; in (III), −1.720 for 407 reflections in the F_c/F_c(max) range 0.000–0.008; in (IV), −0.462 for 393 reflections in the F_c/F_c(max) range 0.000–0.005; in (V), −2.853 for 447 reflections in the F_c/F_c(max) range 0.000–0.006; in (VII), −3.669 for 393 reflections in the F_c/F_c(max) range 0.000–0.004; in (VI), there was a large positive value of 11.570 for 756 reflections in the F_c/F_c(max) range 0.000–0.006; these are all probably statistical artefacts.

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

For compounds (I)–(VI) τ_1, τ_2, τ_3, τ_4 and τ_5 represent, respectively, the torsion angles S*x*21—C*x*22—N*x*1—C*x*1, C*x*22—N*x*1—C*x*1—C*x*11, N*x*1—C*x*1—C*x*11—C*x*12, C*x*23—C*x*37—C*x*31—C*x*32 and S*x*21—C*x*25—C*x*26—C*x*27. For (IV) and (V), *x* = nul; for the major- and minor-disorder components of (I), (II) and (III), *x* = 1 or 2, respectively; for compound (VI), *x* = 1 for molecule 1 and *x* = 2 or 3 for the major- and minor-disorder components, respectively, of molecule 2.

	τ_1	τ_2	τ_3	τ_4	τ_5
(I), major	−0.7 (11)	−179.0 (8)	−163.2 (6)	−103.8 (5)	25.8 (8)
(I), minor	−9 (9)	−158 (7)	−158 (5)	−97 (4)	56 (4)
(II), major	6.3 (15)	179.1 (4)	179.1 (9)	−68.8 (7)	−55.2 (5)
(II), minor	14 (8)	163 (6)	−157 (5)	−65 (3)	49 (3)
(III), major	−2 (3)	176.9 (18)	146.9 (10)	−71.2 (19)	59 (2)
(III), minor	14 (8)	−159 (4)	136 (4)	−61 (5)	33 (6)
(IV)	0.5 (6)	173.9 (4)	142.0 (4)	−123.7 (5)	−177.8 (5)
(V)	−4.5 (3)	−170.4 (2)	−16.1 (4)	−111.7 (3)	21.1 (4)
(VI), mol. 1	3.0 (6)	−174.5 (4)	137.2 (4)	−110.7 (5)	160.5 (4)
(VI), mol. 2, major	4.5 (11)	−174.3 (6)	134.6 (7)	−70.0 (7)	−174.4 (6)
(VI), mol. 2, minor	16 (11)	172 (6)	127 (8)	−75 (6)	105 (5)

3. Results and discussion

The molecules of compounds (I)–(III) are disordered over two sets of atomic sites, with occupancies of 0.894 (8) and 0.106 (8)

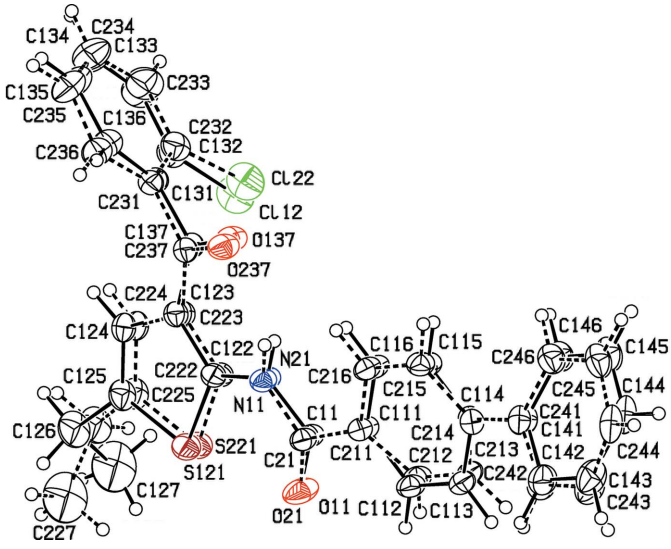


Figure 2
The molecular structure of compound (II), showing the atom-labelling scheme and the disorder. The major-disorder component is drawn with full lines and the minor-disorder component is drawn with broken lines. Displacement ellipsoids are drawn at the 30% probability level.

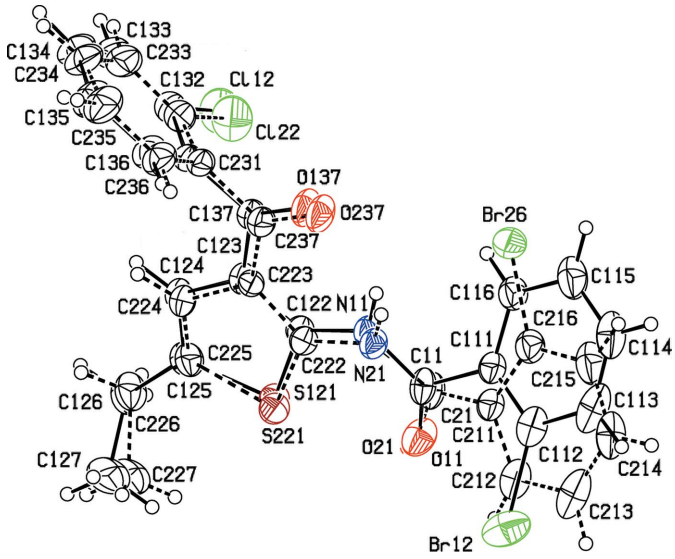


Figure 3
The molecular structure of compound (III), showing the atom-labelling scheme and the disorder. The major-disorder component is drawn with full lines and the minor-disorder component is drawn with broken lines. Displacement ellipsoids are drawn at the 30% probability level and, for the sake of clarity, some of the atom labels have been omitted.

in (I), 0.832 (5) and 0.168 (5) in (II), and 0.7006 (12) and 0.2994 (12) in (III). By contrast, compounds (IV) and (V) are fully ordered, while in compound (VI), which crystallizes with $Z' = 2$, molecule 1, containing atom O11, is fully ordered, but molecule 2, containing atom O21, is disordered over two sets of atomic sites having occupancies of 0.916 (3) and 0.084 (3). For each of (I)–(VI), which are all conformationally chiral (Figs. 1–6), the reference molecules were selected to have the same sign of torsion angle τ_4 (Table 2), which defines the orientation of the 2-chlorobenzoyl substituent relative to the thiophene ring. In each compound, the amide unit is effectively coplanar with the thiophene ring, as shown by torsion angles τ_1 and τ_2; the relative orientation of the thiophene and

Table 3
Hydrogen bonds and short intra- and intermolecular contacts (Å, °) for compounds (I)–(VII).

*Cg*1 and *Cg*2 represent the centroids of the C131–C136 and C31–C36 rings, respectively.

Compound	*D*—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)	N11—H11···O137	0.86	2.03	2.671 (12)	131
	N21—H21···O237	0.86	2.12	2.73 (10)	129
	C115—H115···O137^{i}	0.93	2.53	3.438 (9)	166
	C215—H215···O237^{i}	0.93	2.13	3.04 (7)	166
(II)	N11—H11···O137	0.86	2.08	2.719 (10)	130
	N21—H21···O237	0.86	2.01	2.65 (5)	131
	C112—H112···O11^{ii}	0.93	2.47	3.230 (4)	139
	C144—H144···O137^{iii}	0.93	2.56	3.397 (6)	150
(III)	N11—H11···O137	0.86	2.06	2.70 (2)	131
	N21—H21···O237	0.86	2.12	2.71 (6)	126
	C114—H114···*Cg*1^{iv}	0.93	2.82	3.565 (12)	138
	C214—H214···O237^{v}	0.93	2.24	2.96 (4)	134
(IV)	N1—H1···O37	0.86	2.02	2.665 (5)	131
	C33—H33···O37^{vi}	0.93	2.44	3.322 (7)	159
	C15—H15···*Cg*2^{vii}	0.93	2.92	3.453 (6)	118
(V)	N1—H1···O12	0.86	1.97	2.651 (3)	135
	N1—H1···O37	0.86	2.08	2.716 (3)	130
	C13—H13···*Cg*2^{viii}	0.93	2.90	3.782 (4)	158
	C36—H36···O1^{ix}	0.93	2.48	3.306 (4)	147
(VI)	N11—H11···O137	0.86	2.13	2.753 (4)	129
	N21—H21···O237	0.86	2.10	2.733 (11)	129
	N31—H31···O337	0.86	2.22	2.76 (10)	121
	N31—H31···O11	0.86	2.60	3.28 (10)	137
	C114—H114···O337^{x}	0.93	2.50	3.33 (5)	150
	C214—H214···O137^{xi}	0.93	2.59	3.407 (6)	146
	C314—H314···O137^{xi}	0.93	2.58	3.42 (4)	150
(VII)	C27—H27···O14^{ii}	0.93	2.52	3.175 (3)	127

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

2-chlorobenzoyl substituents is probably controlled in each compound by the short intramolecular N—H···O contact involving the carbonyl O atom (Table 3). It is similarly likely that the orientation of the methoxy group in compound (V) is controlled by a second intramolecular N—H···O contact

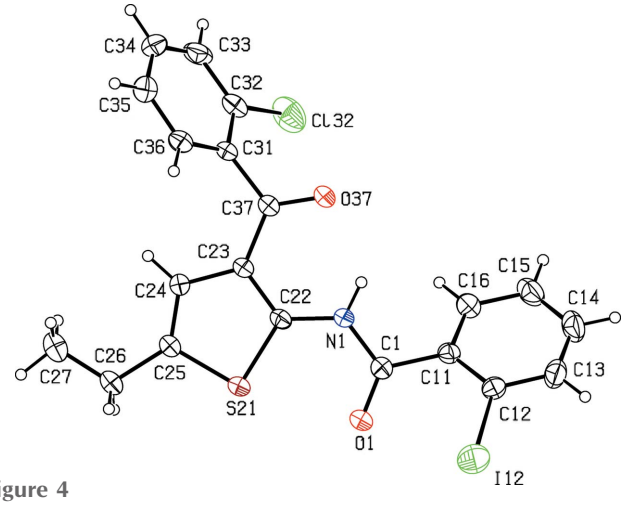


Figure 4
The molecular structure of compound (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

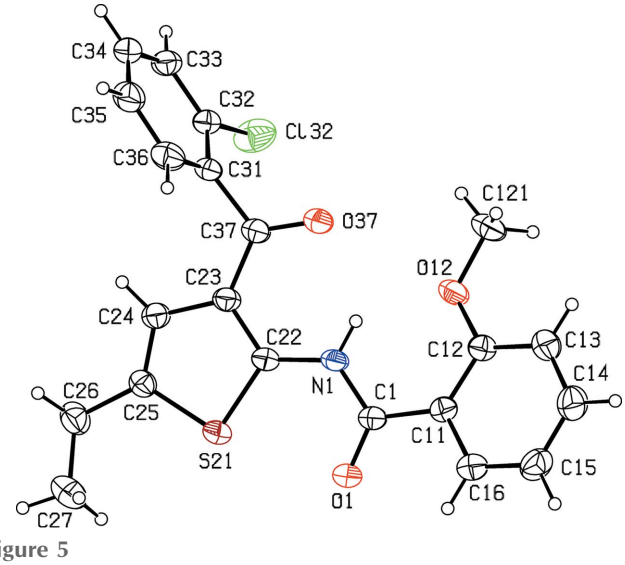


Figure 5
The molecular structure of compound (V), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

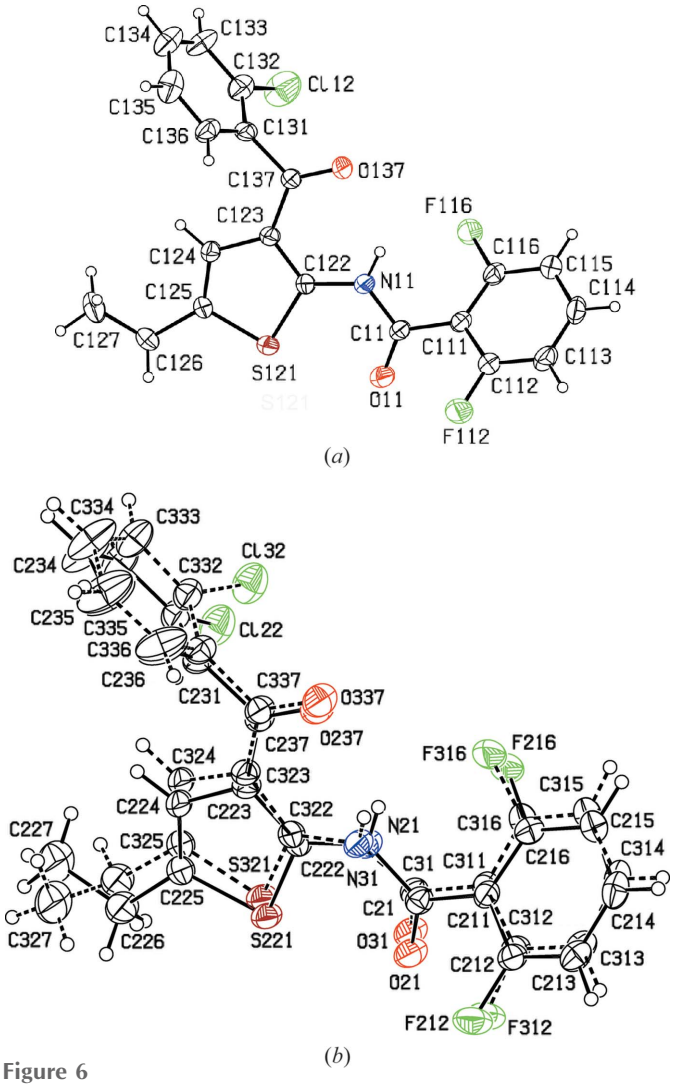


Figure 6
The structures and atom-labelling schemes of the two independent molecules in compound (VI), showing (*a*) molecule 1 and (*b*) molecule 2. For the disorder in molecule 2, the major-disorder component is drawn with full lines and the minor-disorder component is drawn with broken lines. Displacement ellipsoids are drawn at the 30% probability level and, for the sake of clarity, some of the atom labels in part (*b*) have been omitted.

(Table 3 and Fig. 5). It is of interest to note that in compound (III), the two disorder forms are conformational isomers, in

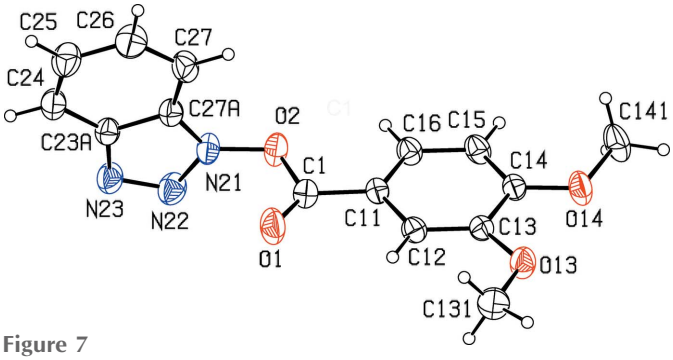
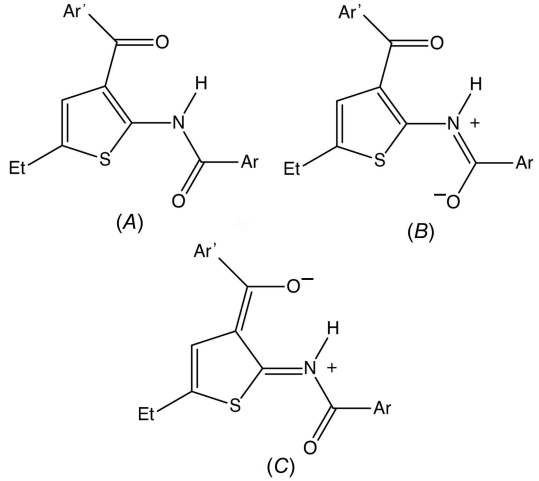


Figure 7
The molecular structure of compound (VII), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

that the Br substituent is on opposite edges of the 2-bromophenyl ring in the two forms (Fig. 3). The orientation of the ethyl group relative to the thiophene ring is extremely variable, as shown by the values of τ_5 and as can be seen in Figs. 1–6, and this variability extends even to the two disorder components in a given compound, most markedly in (I), (II) and (VI). Since this substituent plays no role in the supramolecular assembly, it seems likely that it is acting simply as a space filler, occupying whatever space is available to it in the overall supramolecular structure. The variations in torsion angle τ_3 (Table 2), which defines the orientation of the substituted aryl ring (C*x*1–C*x*6), may be associated with the observation that the C—H units in the ring participate in the hydrogen bonds in each of (I)–(VI) (Table 3).



Scheme 2

In each compound, the bond lengths provide evidence for a modest delocalization of the lone pair of electrons on the N atom, not only into the adjacent carbonyl unit, but also into the remote carbonyl unit since atoms N*x*1, C*x*22, C*x*23, C*x*37 and O*x*37 (the values of *x* are defined in Table 2) constitute a vinylogous amide system, so that forms (*A*), (*B*) and (*C*) (Scheme 2) all contribute to the overall electronic structure. The polarization in form (*C*) enhances the strength of the intramolecular N—H···O interactions. In this connection, we note here that, as discussed below, the acyl carbonyl O atom is used as an acceptor in intermolecular hydrogen bonds in each of compounds (I), (II), (IV) and (VI), while the amide carbonyl O atoms act as such an acceptor only in compounds (II) and (V); neither of these O atoms participates in the supramolecular assembly of compound (III).

In compound (VII) (Fig. 7), the benzoate unit is effectively planar, with an r.m.s. deviation of only 0.0192 Å from the mean plane of atoms C11–C16, C1, O1 and O2, and this plane makes a dihedral angle of 84.51 (6)° with that of the benzotriazole unit; the two methoxy C atoms are effectively coplanar with the adjacent ring, with displacements from this plane of 0.102 (4) and 0.215 (4) Å for atoms C131 and C141, respectively. Consistent with this observation, the corresponding pairs of exocyclic C—C—O angles differ at both C13 and C14 by *ca* 10°, as typically found in planar or near-planar alkoxyarenes (Seip & Seip, 1973; Ferguson *et al.*, 1996).

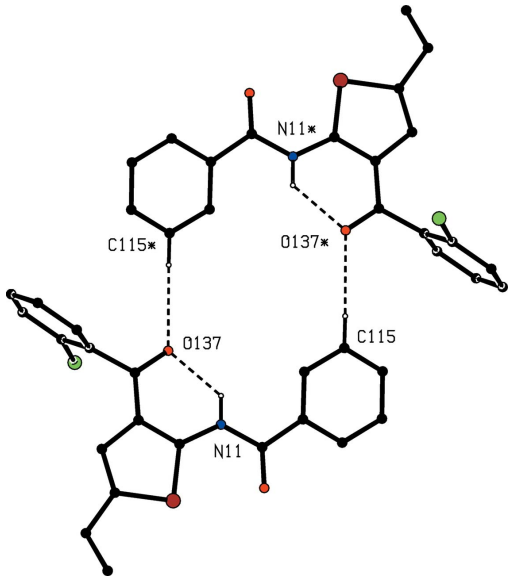


Figure 8
Part of the crystal structure of compound (I), showing the formation of a cyclic hydrogen-bonded dimer. For the sake of clarity, the unit-cell outline, H atoms bonded to C atoms which are not involved in the motif shown and the minor-disorder component have all been omitted. Hydrogen bonds are shown as dashed lines and atoms marked with an asterisk (*) are at the symmetry position $(-x, -y + 1, -z + 1)$.

As noted above, the molecules of (I)–(VI) all contain an intramolecular N—H···O interaction, but the N—H unit plays no role in the supramolecular assembly in any of (I)–(VI); this assembly depends upon C—H···O and C—H···π(arene) hydrogen bonds (Table 3). Because of the relatively low abundances of the minor-disorder forms in compounds (I)–(III) and (VI), we discuss here only the supramolecular interactions involving the major-disorder forms, although those for the minor forms are included in Table 3 for the sake of completeness.

In compound (I), a single C—H···O hydrogen bond links inversion-related pairs of molecules to form a cyclic centrosymmetric dimer in which an outer $R_2^2(20)$ (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) ring encloses an inner $R_4^2(16)$ ring flanked by two inversion-related $S(6)$ rings (Fig. 8). Two dimers of this type, related to one another by the translational symmetry elements, are present in each unit cell, but there are no direction-specific interactions between adjacent dimers.

Two independent C—H···O hydrogen bonds link the molecules of compound (II) into sheets, whose formation is readily analysed in terms of two simpler substructures (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000). In the simplest of these substructures, inversion-related pairs of molecules are linked into cyclic $R_2^2(10)$ dimers, where the reference dimer is centred at $(\frac{1}{2}, 1, \frac{1}{2})$. In the second substructure, molecules related by a 2_1 screw axis are linked into $C_2^1(13)$ chains running parallel to the [010] direction. The action of these chains is to link the reference dimer centred at $(\frac{1}{2}, 1, \frac{1}{2})$ directly to four other such dimers, centred at $(\frac{1}{2}, \frac{1}{2}, 0)$, $(\frac{1}{2}, \frac{3}{2}, 0)$, $(\frac{1}{2}, \frac{1}{2}, 1)$ and $(\frac{1}{2}, \frac{3}{2}, 1)$, so forming a sheet lying parallel to (100) and containing rings of $R_2^2(10)$ and $R_{10}^6(54)$ types (Fig. 9).

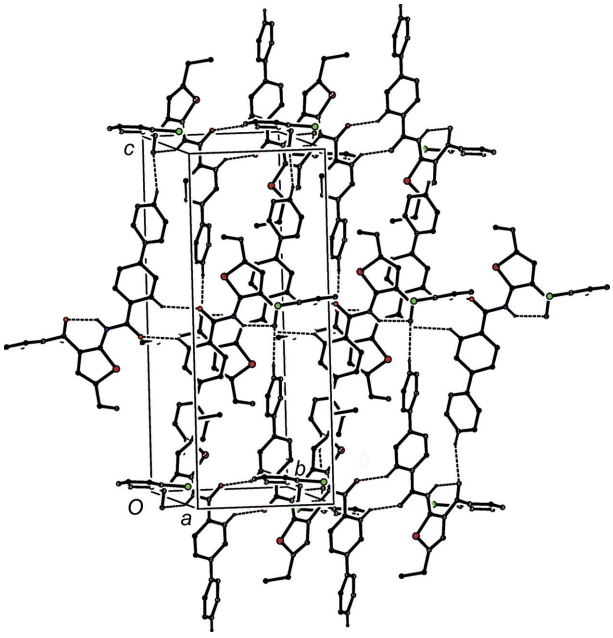


Figure 9
Part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded sheet lying parallel to (100). For the sake of clarity, H atoms bonded to C atoms which are not involved in the motif shown and the minor-disorder component have been omitted. Hydrogen bonds are shown as dashed lines.

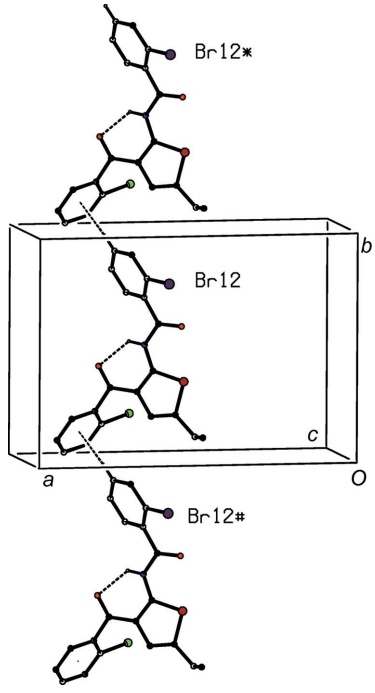


Figure 10
Part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded chain running parallel to [010]. For the sake of clarity, H atoms bonded to C atoms which are not involved in the motif shown and the minor-disorder component have been omitted. Hydrogen bonds are shown as dashed lines and the Br atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, y + 1, z)$ and $(x, y - 1, z)$, respectively.

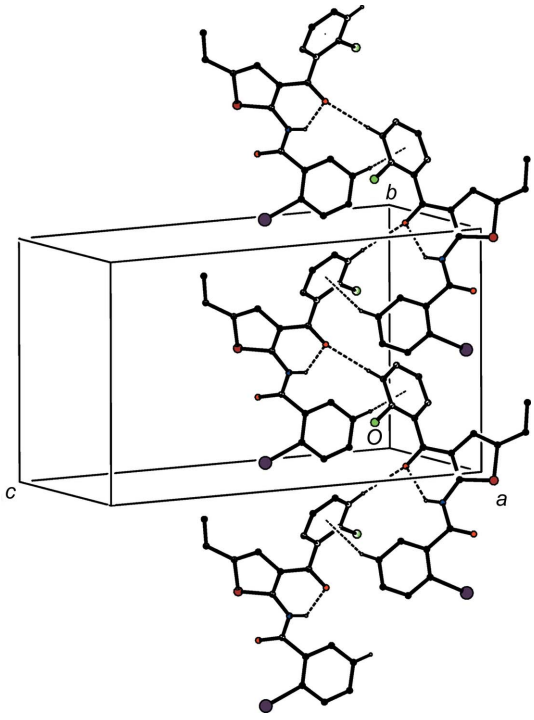
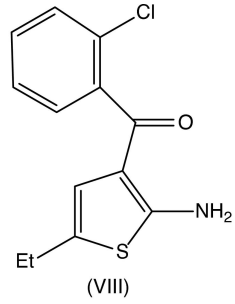


Figure 11
Part of the crystal structure of compound (IV), showing the formation of a chain of rings running parallel to [010] and built from C—H···O and C—H···π(arene) hydrogen bonds, shown as dashed lines. For the sake of clarity, H atoms bonded to C atoms which are not involved in the motif shown have been omitted.

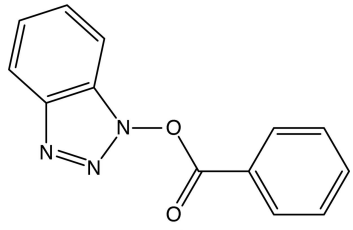
The major components of compound (III) are linked by a single C—H···π(arene) hydrogen bond to form simple chains running parallel to the [010] direction in which the component molecules are related by translation (Fig. 10). Eight chains of this type pass through each unit cell but there are no direction-specific interactions between adjacent chains. The molecules of compound (IV) which are related by a 2_1 screw axis are linked by a C—H···O hydrogen bond to form a $C(6)$ chain running parallel to the [010] direction; the action of this hydrogen bond is weakly reinforced by a C—H···π(arene) hydrogen bond, so forming a chain of rings parallel to the [010] direction (Fig. 11). Two chains of this type, related to one another by inversion, pass through each unit cell, but there are no direction-specific interactions between adjacent chains.

The crystal structure of compound (V) also contains a chain of rings built from C—H···O and C—H···π(arene) hydrogen bonds and again running parallel to the [010] direction. However, this chain contains two types of ring, both centrosymmetric, with $R_2^2(18)$ rings containing inversion-related pairs of C—H···O hydrogen bonds centred at $(0, n+\frac{1}{2}, \frac{1}{2})$ and rings built from inversion-related pairs of C—H···π(arene) hydrogen bonds centred at $(0, n, \frac{1}{2})$, where n represents an integer in each case (Fig. 12). The two independent molecules of compound (VI) are linked by a single C—H···O hydrogen bond, but there are no further direction-specific interactions in the structure, which simply contains finite acyclic dimers [*cf.* the cyclic dimer formed in compound (I)]. There is a single C—H···O contact in compound (VII) between inversion-related pairs of molecules, but the very small C—H···O angle suggests that this may not be structurally significant (Wood *et al.*, 2009).

It is of interest briefly to compare the structures of compounds (I)–(VII) reported here with those of related compounds. The structure of the amine 2-amino-3-(2-chlorobenzoyl)-5-ethylthiophene, (VIII) (see Scheme 3), has been reported recently (Kubicki *et al.*, 2012). The molecule contains an intramolecular N—H···O hydrogen bond, as found in each of (I)–(VI), and the values of torsion angles τ_4 and τ_5 for the conformational enantiomer of (VIII) corresponding to those of (I)–(VI) are −82.2 and 56.7°, respectively (no s.u. values are associated with the deposited atomic coordinates) The value of τ_4 is very similar to those found here, but that for τ_5 again suggests a space-filling role for the ethyl substituent. The molecules of (VIII) are linked by N—H···O hydrogen bonds into $C_2^1(4)$ chains. The original report on (VIII) also refers to three intermolecular C—H···Cl interactions. However, for all of these interactions, the reported H···Cl distances are larger than the sum of the van der Waals radii for H and Cl (Rowland & Taylor, 1996) and, in any event, it has been convincingly shown from database analyses that a Cl atom bonded to a C atom is a very poor acceptor of hydrogen bonds, even from good donors such as O and N, and thus even worse from C (Brammer *et al.*, 2001; Thallypally & Nangia, 2001).



(VIII)



(IX)

Scheme 3

The structure of benzotriazol-1-yl benzoate, (IX), was reported some 40 years ago (McCarthy *et al.*, 1977) in a study focused on determining whether the compound was an *O*-acyl or an *N*-acyl derivative, based upon the observation that such benzotriazole compounds effect acyl transfer to amines at rates comparable with those found for *N*-acylimidazoles. The product was indeed found to be a benzoate ester and like compound (VII), the benzoate fragment is planar, the two rings making a dihedral angle of 84.8° (no s.u. values are associated with the deposited atomic coordinates), almost identical to the corresponding value in (VII). However, the

supramolecular assembly in (IX) is entirely different from that in (VII); in (IX), four hydrogen bonds, one each of the C—H···O and C—H···π(arene) types and two of the C—H···N type, all having D—H···A angles greater than 140° (*cf.* Wood *et al.*, 2009), link the molecules into a three-dimensional framework structure, which is reinforced by aromatic π–π stacking interactions.

Acknowledgements

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Funding information

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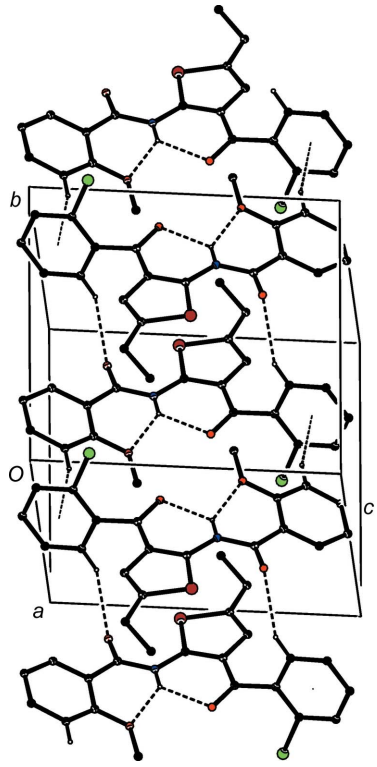

Figure 12
Part of the crystal structure of compound (V), showing the formation of a chain containing two types of centrosymmetric rings running parallel to [010] and built from C—H···O and C—H···π(arene) hydrogen bonds, shown as dashed lines. For the sake of clarity, H atoms bonded to C atoms which are not involved in the motif shown have been omitted.

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supporting information

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Synthesis and structures of six closely related *N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]arylamides, together with an isolated reaction intermediate: order *versus* disorder, molecular conformations and hydrogen bonding in zero, one and two dimensions

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

Computing details

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

N-[3-(2-Chlorobenzoyl)-5-ethylthiophen-2-yl]benzamide (I)

Crystal data

$C_{20}H_{16}ClNO_2S$
M_r = 369.85
Monoclinic, $P2_1/c$
a = 9.7868 (9) Å
b = 13.9567 (12) Å
c = 13.3764 (11) Å
β = 101.816 (2)°
V = 1788.4 (3) Å^3
Z = 4
$F(000)$ = 768
D_x = 1.374 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4162 reflections
θ = 2.1–27.6°
μ = 0.34 mm^{-1}
T = 294 K
Block, yellow
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} = 0.864, T_{max} = 0.934
26489 measured reflections
3726 independent reflections
2092 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 26.6°, θ_{min} = 2.1°
h = −12→12
k = −17→17
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.131
S = 1.00
3726 reflections
304 parameters
72 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 0.5759P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ 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)
C11	0.4662 (5)	0.4257 (5)	0.6023 (6)	0.0450 (7)	0.894 (8)
O11	0.5871 (4)	0.4022 (3)	0.6349 (4)	0.0654 (14)	0.894 (8)
N11	0.3840 (6)	0.3740 (4)	0.5264 (5)	0.0469 (9)	0.894 (8)
H11	0.2994	0.3932	0.5055	0.056*	0.894 (8)
C111	0.3996 (4)	0.5092 (4)	0.6421 (5)	0.0394 (9)	0.894 (8)
C112	0.4867 (4)	0.5772 (4)	0.6982 (5)	0.0482 (11)	0.894 (8)
H112	0.5830	0.5708	0.7071	0.058*	0.894 (8)
C113	0.4309 (5)	0.6544 (4)	0.7409 (6)	0.0567 (11)	0.894 (8)
H113	0.4898	0.6993	0.7791	0.068*	0.894 (8)
C114	0.2884 (5)	0.6649 (4)	0.7270 (6)	0.0618 (12)	0.894 (8)
H114	0.2512	0.7177	0.7545	0.074*	0.894 (8)
C115	0.2014 (5)	0.5981 (5)	0.6728 (6)	0.0608 (11)	0.894 (8)
H115	0.1052	0.6045	0.6650	0.073*	0.894 (8)
C116	0.2564 (4)	0.5205 (5)	0.6295 (8)	0.0509 (10)	0.894 (8)
H116	0.1968	0.4758	0.5917	0.061*	0.894 (8)
S121	0.5895 (2)	0.24663 (19)	0.51192 (17)	0.0545 (4)	0.894 (8)
C122	0.4252 (4)	0.2938 (4)	0.4807 (5)	0.0424 (7)	0.894 (8)
C123	0.3390 (5)	0.2415 (5)	0.4055 (5)	0.0430 (8)	0.894 (8)
C124	0.4093 (5)	0.1595 (4)	0.3766 (4)	0.0520 (14)	0.894 (8)
H124	0.3669	0.1158	0.3274	0.062*	0.894 (8)
C125	0.5426 (5)	0.1516 (4)	0.4272 (3)	0.0610 (11)	0.894 (8)
C126	0.6508 (6)	0.0790 (4)	0.4134 (4)	0.1070 (15)	0.894 (8)
H16A	0.6042	0.0178	0.3985	0.128*	0.894 (8)
H16B	0.6863	0.0973	0.3535	0.128*	0.894 (8)
C127	0.7653 (6)	0.0650 (4)	0.4936 (4)	0.118 (2)	0.894 (8)
H17A	0.8178	0.1234	0.5063	0.178*	0.894 (8)
H17B	0.8236	0.0153	0.4756	0.178*	0.894 (8)
H17C	0.7330	0.0467	0.5540	0.178*	0.894 (8)
C137	0.1991 (4)	0.2729 (3)	0.3611 (3)	0.0493 (8)	0.894 (8)
O137	0.1483 (6)	0.3470 (6)	0.3882 (10)	0.0763 (10)	0.894 (8)
C131	0.1082 (4)	0.2122 (3)	0.2808 (3)	0.0471 (8)	0.894 (8)
C132	0.0831 (4)	0.2350 (2)	0.1786 (3)	0.0529 (8)	0.894 (8)
Cl12	0.1722 (4)	0.32924 (12)	0.13678 (12)	0.0900 (6)	0.894 (8)
C133	−0.0123 (7)	0.1840 (4)	0.1081 (3)	0.0660 (10)	0.894 (8)

H133	−0.0291	0.2010	0.0394	0.079*	0.894 (8)
C134	−0.0821 (5)	0.1085 (3)	0.1395 (4)	0.0696 (13)	0.894 (8)
H134	−0.1470	0.0746	0.0921	0.084*	0.894 (8)
C135	−0.0571 (5)	0.0828 (3)	0.2396 (4)	0.0736 (13)	0.894 (8)
H135	−0.1035	0.0308	0.2607	0.088*	0.894 (8)
C136	0.0383 (7)	0.1348 (4)	0.3097 (4)	0.0667 (10)	0.894 (8)
H136	0.0555	0.1169	0.3781	0.080*	0.894 (8)
C21	0.458 (4)	0.432 (4)	0.600 (5)	0.0450 (7)	0.106 (8)
O21	0.584 (3)	0.423 (3)	0.619 (4)	0.0654 (14)	0.106 (8)
N21	0.379 (5)	0.384 (4)	0.519 (4)	0.0469 (9)	0.106 (8)
H21	0.3034	0.4107	0.4883	0.056*	0.106 (8)
C211	0.382 (3)	0.504 (3)	0.650 (4)	0.0394 (9)	0.106 (8)
C212	0.461 (3)	0.576 (3)	0.707 (5)	0.0482 (11)	0.106 (8)
H212	0.5572	0.5680	0.7283	0.058*	0.106 (8)
C213	0.397 (4)	0.658 (3)	0.731 (6)	0.0567 (11)	0.106 (8)
H213	0.4505	0.7107	0.7585	0.068*	0.106 (8)
C214	0.253 (5)	0.663 (4)	0.715 (6)	0.0618 (12)	0.106 (8)
H214	0.2104	0.7127	0.7427	0.074*	0.106 (8)
C215	0.175 (4)	0.594 (4)	0.657 (6)	0.0608 (11)	0.106 (8)
H215	0.0788	0.6027	0.6354	0.073*	0.106 (8)
C216	0.238 (3)	0.512 (5)	0.630 (7)	0.0509 (10)	0.106 (8)
H216	0.1833	0.4611	0.5995	0.061*	0.106 (8)
S221	0.566 (2)	0.2386 (18)	0.5242 (16)	0.0545 (4)	0.106 (8)
C222	0.411 (3)	0.296 (3)	0.483 (4)	0.0424 (7)	0.106 (8)
C223	0.329 (3)	0.250 (4)	0.401 (4)	0.0430 (8)	0.106 (8)
C224	0.404 (4)	0.174 (4)	0.365 (4)	0.0520 (14)	0.106 (8)
H224	0.3639	0.1331	0.3115	0.062*	0.106 (8)
C225	0.538 (4)	0.167 (3)	0.415 (2)	0.0610 (11)	0.106 (8)
C226	0.641 (4)	0.089 (3)	0.407 (3)	0.1070 (15)	0.106 (8)
H26A	0.6082	0.0549	0.3431	0.128*	0.106 (8)
H26B	0.7290	0.1189	0.4023	0.128*	0.106 (8)
C227	0.668 (6)	0.021 (3)	0.485 (3)	0.118 (2)	0.106 (8)
H27A	0.5992	−0.0291	0.4716	0.178*	0.106 (8)
H27B	0.6635	0.0513	0.5490	0.178*	0.106 (8)
H27C	0.7591	−0.0058	0.4891	0.178*	0.106 (8)
C237	0.182 (3)	0.271 (2)	0.3688 (17)	0.0493 (8)	0.106 (8)
O237	0.131 (5)	0.348 (5)	0.388 (9)	0.0763 (10)	0.106 (8)
C231	0.092 (4)	0.207 (2)	0.2915 (17)	0.0471 (8)	0.106 (8)
C232	0.060 (2)	0.2286 (12)	0.1890 (14)	0.0529 (8)	0.106 (8)
Cl22	0.116 (2)	0.3359 (12)	0.1463 (12)	0.0900 (6)	0.106 (8)
C233	−0.015 (6)	0.166 (2)	0.119 (2)	0.0660 (10)	0.106 (8)
H233	−0.0326	0.1801	0.0497	0.079*	0.106 (8)
C234	−0.064 (5)	0.082 (2)	0.152 (3)	0.0696 (13)	0.106 (8)
H234	−0.1183	0.0411	0.1053	0.084*	0.106 (8)
C235	−0.033 (5)	0.058 (2)	0.253 (3)	0.0736 (13)	0.106 (8)
H235	−0.0646	0.0007	0.2748	0.088*	0.106 (8)
C236	0.045 (6)	0.121 (3)	0.322 (2)	0.0667 (10)	0.106 (8)
H236	0.0667	0.1044	0.3907	0.080*	0.106 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0384 (17)	0.0543 (19)	0.0391 (14)	−0.0016 (13)	0.0007 (12)	−0.0015 (14)
O11	0.0427 (12)	0.079 (3)	0.066 (2)	0.0094 (14)	−0.0089 (12)	−0.021 (2)
N11	0.0384 (13)	0.0531 (19)	0.0437 (16)	0.0060 (12)	−0.0049 (10)	−0.0070 (16)
C111	0.0350 (17)	0.0488 (16)	0.0328 (17)	0.0006 (14)	0.0030 (14)	0.0030 (14)
C112	0.043 (2)	0.0629 (19)	0.039 (2)	−0.0073 (17)	0.0076 (18)	−0.0010 (14)
C113	0.060 (3)	0.0585 (19)	0.049 (3)	−0.009 (2)	0.006 (3)	−0.0108 (17)
C114	0.067 (4)	0.065 (2)	0.053 (3)	0.012 (2)	0.010 (3)	−0.0077 (16)
C115	0.047 (2)	0.074 (2)	0.058 (3)	0.013 (2)	0.002 (2)	−0.005 (2)
C116	0.042 (2)	0.057 (2)	0.0502 (16)	−0.0018 (17)	0.001 (2)	−0.0079 (19)
S121	0.0413 (8)	0.0731 (7)	0.0446 (7)	0.0114 (7)	−0.0020 (5)	−0.0082 (5)
C122	0.0379 (17)	0.0509 (15)	0.0364 (13)	0.0016 (13)	0.0029 (13)	0.0005 (12)
C123	0.0367 (15)	0.049 (2)	0.0400 (15)	0.0028 (12)	0.0009 (11)	−0.0026 (15)
C124	0.0530 (18)	0.054 (3)	0.045 (2)	0.0065 (15)	0.0005 (14)	−0.010 (2)
C125	0.0558 (19)	0.069 (3)	0.052 (2)	0.0186 (17)	−0.0041 (15)	−0.0120 (18)
C126	0.089 (3)	0.122 (4)	0.094 (3)	0.057 (3)	−0.019 (2)	−0.041 (3)
C127	0.093 (4)	0.122 (4)	0.123 (4)	0.061 (4)	−0.019 (3)	−0.032 (3)
C137	0.0412 (18)	0.0552 (17)	0.0483 (16)	0.0001 (14)	0.0019 (13)	−0.0091 (13)
O137	0.049 (2)	0.0768 (15)	0.0895 (15)	0.0200 (16)	−0.017 (2)	−0.0348 (12)
C131	0.0347 (18)	0.0518 (17)	0.0522 (17)	0.0019 (13)	0.0022 (13)	−0.0077 (13)
C132	0.0444 (18)	0.0575 (18)	0.0534 (17)	−0.0037 (14)	0.0020 (14)	−0.0080 (14)
Cl12	0.1077 (16)	0.0914 (7)	0.0720 (6)	−0.0329 (9)	0.0209 (8)	0.0019 (5)
C133	0.060 (2)	0.074 (3)	0.0564 (19)	0.002 (2)	−0.0056 (17)	−0.0107 (18)
C134	0.046 (2)	0.072 (3)	0.082 (3)	−0.003 (2)	−0.0066 (19)	−0.026 (2)
C135	0.061 (3)	0.063 (3)	0.097 (3)	−0.016 (2)	0.016 (2)	−0.012 (2)
C136	0.070 (2)	0.064 (3)	0.063 (2)	−0.009 (2)	0.0065 (18)	0.0004 (17)
C21	0.0384 (17)	0.0543 (19)	0.0391 (14)	−0.0016 (13)	0.0007 (12)	−0.0015 (14)
O21	0.0427 (12)	0.079 (3)	0.066 (2)	0.0094 (14)	−0.0089 (12)	−0.021 (2)
N21	0.0384 (13)	0.0531 (19)	0.0437 (16)	0.0060 (12)	−0.0049 (10)	−0.0070 (16)
C211	0.0350 (17)	0.0488 (16)	0.0328 (17)	0.0006 (14)	0.0030 (14)	0.0030 (14)
C212	0.043 (2)	0.0629 (19)	0.039 (2)	−0.0073 (17)	0.0076 (18)	−0.0010 (14)
C213	0.060 (3)	0.0585 (19)	0.049 (3)	−0.009 (2)	0.006 (3)	−0.0108 (17)
C214	0.067 (4)	0.065 (2)	0.053 (3)	0.012 (2)	0.010 (3)	−0.0077 (16)
C215	0.047 (2)	0.074 (2)	0.058 (3)	0.013 (2)	0.002 (2)	−0.005 (2)
C216	0.042 (2)	0.057 (2)	0.0502 (16)	−0.0018 (17)	0.001 (2)	−0.0079 (19)
S221	0.0413 (8)	0.0731 (7)	0.0446 (7)	0.0114 (7)	−0.0020 (5)	−0.0082 (5)
C222	0.0379 (17)	0.0509 (15)	0.0364 (13)	0.0016 (13)	0.0029 (13)	0.0005 (12)
C223	0.0367 (15)	0.049 (2)	0.0400 (15)	0.0028 (12)	0.0009 (11)	−0.0026 (15)
C224	0.0530 (18)	0.054 (3)	0.045 (2)	0.0065 (15)	0.0005 (14)	−0.010 (2)
C225	0.0558 (19)	0.069 (3)	0.052 (2)	0.0186 (17)	−0.0041 (15)	−0.0120 (18)
C226	0.089 (3)	0.122 (4)	0.094 (3)	0.057 (3)	−0.019 (2)	−0.041 (3)
C227	0.093 (4)	0.122 (4)	0.123 (4)	0.061 (4)	−0.019 (3)	−0.032 (3)
C237	0.0412 (18)	0.0552 (17)	0.0483 (16)	0.0001 (14)	0.0019 (13)	−0.0091 (13)
O237	0.049 (2)	0.0768 (15)	0.0895 (15)	0.0200 (16)	−0.017 (2)	−0.0348 (12)
C231	0.0347 (18)	0.0518 (17)	0.0522 (17)	0.0019 (13)	0.0022 (13)	−0.0077 (13)
C232	0.0444 (18)	0.0575 (18)	0.0534 (17)	−0.0037 (14)	0.0020 (14)	−0.0080 (14)

Cl22	0.1077 (16)	0.0914 (7)	0.0720 (6)	−0.0329 (9)	0.0209 (8)	0.0019 (5)
C233	0.060 (2)	0.074 (3)	0.0564 (19)	0.002 (2)	−0.0056 (17)	−0.0107 (18)
C234	0.046 (2)	0.072 (3)	0.082 (3)	−0.003 (2)	−0.0066 (19)	−0.026 (2)
C235	0.061 (3)	0.063 (3)	0.097 (3)	−0.016 (2)	0.016 (2)	−0.012 (2)
C236	0.070 (2)	0.064 (3)	0.063 (2)	−0.009 (2)	0.0065 (18)	0.0004 (17)

Geometric parameters (Å, º)

C11—O11	1.219 (3)	C21—O21	1.219 (6)
C11—N11	1.365 (3)	C21—N21	1.365 (6)
C11—C111	1.487 (4)	C21—C211	1.488 (6)
N11—C122	1.376 (3)	N21—C222	1.375 (6)
N11—H11	0.8600	N21—H21	0.8600
C111—C116	1.385 (3)	C211—C216	1.386 (5)
C111—C112	1.389 (3)	C211—C212	1.389 (6)
C112—C113	1.384 (4)	C212—C213	1.385 (6)
C112—H112	0.9300	C212—H212	0.9300
C113—C114	1.377 (4)	C213—C214	1.377 (6)
C113—H113	0.9300	C213—H213	0.9300
C114—C115	1.367 (4)	C214—C215	1.368 (6)
C114—H114	0.9300	C214—H214	0.9300
C115—C116	1.387 (4)	C215—C216	1.388 (6)
C115—H115	0.9300	C215—H215	0.9300
C116—H116	0.9300	C216—H216	0.9300
S121—C122	1.708 (3)	S221—C222	1.708 (5)
S121—C125	1.744 (3)	S221—C225	1.747 (6)
C122—C123	1.381 (3)	C222—C223	1.383 (5)
C123—C124	1.429 (3)	C223—C224	1.429 (5)
C123—C137	1.444 (4)	C223—C237	1.445 (6)
C124—C125	1.346 (4)	C224—C225	1.349 (6)
C124—H124	0.9300	C224—H224	0.9300
C125—C126	1.503 (4)	C225—C226	1.507 (6)
C126—C127	1.397 (6)	C226—C227	1.398 (8)
C126—H16A	0.9700	C226—H26A	0.9700
C126—H16B	0.9700	C226—H26B	0.9700
C127—H17A	0.9600	C227—H27A	0.9600
C127—H17B	0.9600	C227—H27B	0.9600
C127—H17C	0.9600	C227—H27C	0.9600
C137—O137	1.235 (3)	C237—O237	1.236 (6)
C137—C131	1.507 (4)	C237—C231	1.507 (5)
C131—C136	1.376 (4)	C231—C236	1.375 (6)
C131—C132	1.377 (4)	C231—C232	1.376 (5)
C132—C133	1.380 (4)	C232—C233	1.380 (6)
C132—Cl12	1.733 (3)	C232—Cl22	1.733 (5)
C133—C134	1.368 (5)	C233—C234	1.368 (6)
C133—H133	0.9300	C233—H233	0.9300
C134—C135	1.359 (5)	C234—C235	1.358 (7)
C134—H134	0.9300	C234—H234	0.9300

C135—C136	1.386 (4)	C235—C236	1.386 (6)
C135—H135	0.9300	C235—H235	0.9300
C136—H136	0.9300	C236—H236	0.9300
O11—C11—N11	120.4 (3)	O21—C21—N21	120.2 (10)
O11—C11—C111	123.2 (2)	O21—C21—C211	123.1 (9)
N11—C11—C111	116.4 (2)	N21—C21—C211	116.0 (8)
C11—N11—C122	125.4 (3)	C21—N21—C222	125.8 (11)
C11—N11—H11	117.3	C21—N21—H21	117.1
C122—N11—H11	117.3	C222—N21—H21	117.1
C116—C111—C112	118.8 (2)	C216—C211—C212	118.7 (6)
C116—C111—C11	123.5 (2)	C216—C211—C21	123.1 (8)
C112—C111—C11	117.6 (2)	C212—C211—C21	117.5 (8)
C113—C112—C111	120.3 (3)	C213—C212—C211	119.9 (8)
C113—C112—H112	119.8	C213—C212—H212	120.0
C111—C112—H112	119.8	C211—C212—H212	120.0
C114—C113—C112	120.1 (3)	C214—C213—C212	119.9 (9)
C114—C113—H113	119.9	C214—C213—H213	120.1
C112—C113—H113	119.9	C212—C213—H213	120.1
C115—C114—C113	120.2 (3)	C215—C214—C213	119.8 (9)
C115—C114—H114	119.9	C215—C214—H214	120.1
C113—C114—H114	119.9	C213—C214—H214	120.1
C114—C115—C116	120.1 (3)	C214—C215—C216	119.7 (9)
C114—C115—H115	119.9	C214—C215—H215	120.1
C116—C115—H115	119.9	C216—C215—H215	120.1
C111—C116—C115	120.5 (3)	C211—C216—C215	120.4 (8)
C111—C116—H116	119.8	C211—C216—H216	119.8
C115—C116—H116	119.8	C215—C216—H216	119.8
C122—S121—C125	91.45 (15)	C222—S221—C225	91.1 (4)
N11—C122—C123	124.2 (2)	N21—C222—C223	124.0 (9)
N11—C122—S121	123.5 (2)	N21—C222—S221	123.7 (8)
C123—C122—S121	112.2 (2)	C223—C222—S221	111.8 (5)
C122—C123—C124	111.4 (2)	C222—C223—C224	111.2 (5)
C122—C123—C137	121.7 (2)	C222—C223—C237	121.4 (9)
C124—C123—C137	126.8 (2)	C224—C223—C237	126.9 (9)
C125—C124—C123	113.6 (3)	C225—C224—C223	113.4 (6)
C125—C124—H124	123.2	C225—C224—H224	123.3
C123—C124—H124	123.2	C223—C224—H224	123.3
C124—C125—C126	128.9 (3)	C224—C225—C226	128.0 (9)
C124—C125—S121	111.3 (2)	C224—C225—S221	110.5 (7)
C126—C125—S121	119.6 (3)	C226—C225—S221	118.6 (7)
C127—C126—C125	118.3 (3)	C227—C226—C225	117.5 (9)
C127—C126—H16A	107.7	C227—C226—H26A	107.9
C125—C126—H16A	107.7	C225—C226—H26A	107.9
C127—C126—H16B	107.7	C227—C226—H26B	107.9
C125—C126—H16B	107.7	C225—C226—H26B	107.9
H16A—C126—H16B	107.1	H26A—C226—H26B	107.2
C126—C127—H17A	109.5	C226—C227—H27A	109.5

C126—C127—H17B	109.5	C226—C227—H27B	109.5
H17A—C127—H17B	109.5	H27A—C227—H27B	109.5
C126—C127—H17C	109.5	C226—C227—H27C	109.5
H17A—C127—H17C	109.5	H27A—C227—H27C	109.5
H17B—C127—H17C	109.5	H27B—C227—H27C	109.5
O137—C137—C123	122.6 (3)	O237—C237—C223	122.2 (10)
O137—C137—C131	117.7 (3)	O237—C237—C231	117.4 (8)
C123—C137—C131	119.7 (2)	C223—C237—C231	119.2 (8)
C136—C131—C132	117.5 (3)	C236—C231—C232	117.7 (6)
C136—C131—C137	119.8 (3)	C236—C231—C237	119.8 (7)
C132—C131—C137	122.5 (3)	C232—C231—C237	122.4 (7)
C131—C132—C133	121.2 (3)	C231—C232—C233	121.1 (6)
C131—C132—Cl12	119.8 (2)	C231—C232—Cl22	119.7 (6)
C133—C132—Cl12	119.0 (2)	C233—C232—Cl22	119.2 (6)
C134—C133—C132	119.8 (3)	C234—C233—C232	119.8 (7)
C134—C133—H133	120.1	C234—C233—H233	120.1
C132—C133—H133	120.1	C232—C233—H233	120.1
C135—C134—C133	120.4 (3)	C235—C234—C233	120.5 (7)
C135—C134—H134	119.8	C235—C234—H234	119.8
C133—C134—H134	119.8	C233—C234—H234	119.8
C134—C135—C136	119.3 (3)	C234—C235—C236	119.2 (7)
C134—C135—H135	120.4	C234—C235—H235	120.4
C136—C135—H135	120.4	C236—C235—H235	120.4
C131—C136—C135	121.7 (3)	C231—C236—C235	121.7 (7)
C131—C136—H136	119.1	C231—C236—H236	119.2
C135—C136—H136	119.1	C235—C236—H236	119.2

O11—C11—N11—C122	0.2 (11)	O21—C21—N21—C222	32 (9)
C111—C11—N11—C122	−179.0 (8)	C211—C21—N21—C222	−158 (7)
O11—C11—C111—C116	−159.8 (8)	O21—C21—C211—C216	−178 (8)
N11—C11—C111—C116	19.4 (9)	N21—C21—C211—C216	12 (8)
O11—C11—C111—C112	17.6 (10)	O21—C21—C211—C212	12 (8)
N11—C11—C111—C112	−163.2 (6)	N21—C21—C211—C212	−158 (5)
C116—C111—C112—C113	0.3 (8)	C216—C211—C212—C213	−8 (8)
C11—C111—C112—C113	−177.2 (7)	C21—C211—C212—C213	162 (7)
C111—C112—C113—C114	−0.8 (10)	C211—C212—C213—C214	11 (9)
C112—C113—C114—C115	1.4 (10)	C212—C213—C214—C215	−13 (10)
C113—C114—C115—C116	−1.6 (11)	C213—C214—C215—C216	11 (10)
C112—C111—C116—C115	−0.4 (10)	C212—C211—C216—C215	7 (9)
C11—C111—C116—C115	176.9 (9)	C21—C211—C216—C215	−163 (8)
C114—C115—C116—C111	1.1 (12)	C214—C215—C216—C211	−9 (11)
C11—N11—C122—C123	178.5 (7)	C21—N21—C222—C223	−180 (5)
C11—N11—C122—S121	−0.7 (11)	C21—N21—C222—S221	−9 (9)
C125—S121—C122—N11	176.9 (7)	C225—S221—C222—N21	−160 (6)
C125—S121—C122—C123	−2.4 (5)	C225—S221—C222—C223	11 (4)
N11—C122—C123—C124	−177.1 (8)	N21—C222—C223—C224	165 (7)
S121—C122—C123—C124	2.1 (7)	S221—C222—C223—C224	−7 (6)
N11—C122—C123—C137	6.1 (10)	N21—C222—C223—C237	−23 (8)

S121—C122—C123—C137	−174.6 (7)	S221—C222—C223—C237	165 (6)
C122—C123—C124—C125	−0.6 (8)	C222—C223—C224—C225	−3 (7)
C137—C123—C124—C125	175.9 (7)	C237—C223—C224—C225	−175 (6)
C123—C124—C125—C126	−176.1 (6)	C223—C224—C225—C226	171 (5)
C123—C124—C125—S121	−1.1 (7)	C223—C224—C225—S221	12 (6)
C122—S121—C125—C124	2.0 (5)	C222—S221—C225—C224	−13 (4)
C122—S121—C125—C126	177.5 (5)	C222—S221—C225—C226	−175 (3)
C124—C125—C126—C127	−159.7 (8)	C224—C225—C226—C227	−103 (6)
S121—C125—C126—C127	25.8 (8)	S221—C225—C226—C227	56 (4)
C122—C123—C137—O137	−0.9 (11)	C222—C223—C237—O237	24 (9)
C124—C123—C137—O137	−177.1 (12)	C224—C223—C237—O237	−165 (10)
C122—C123—C137—C131	−178.7 (5)	C222—C223—C237—C231	−169 (3)
C124—C123—C137—C131	5.1 (10)	C224—C223—C237—C231	2 (8)
O137—C137—C131—C136	−96.8 (10)	O237—C237—C231—C236	−113 (8)
C123—C137—C131—C136	81.2 (6)	C223—C237—C231—C236	80 (5)
O137—C137—C131—C132	78.3 (10)	O237—C237—C231—C232	71 (8)
C123—C137—C131—C132	−103.8 (5)	C223—C237—C231—C232	−97 (4)
C136—C131—C132—C133	2.2 (5)	C236—C231—C232—C233	−1 (3)
C137—C131—C132—C133	−173.0 (5)	C237—C231—C232—C233	176 (4)
C136—C131—C132—Cl12	−177.9 (3)	C236—C231—C232—Cl22	−180 (2)
C137—C131—C132—Cl12	7.0 (5)	C237—C231—C232—Cl22	−3 (3)
C131—C132—C133—C134	−1.0 (7)	C231—C232—C233—C234	3 (5)
Cl12—C132—C133—C134	179.0 (4)	Cl22—C232—C233—C234	−178 (3)
C132—C133—C134—C135	−0.6 (8)	C232—C233—C234—C235	−3 (6)
C133—C134—C135—C136	1.0 (7)	C233—C234—C235—C236	1 (6)
C132—C131—C136—C135	−1.8 (7)	C232—C231—C236—C235	−1 (5)
C137—C131—C136—C135	173.5 (5)	C237—C231—C236—C235	−178 (4)
C134—C135—C136—C131	0.2 (8)	C234—C235—C236—C231	1 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O137	0.86	2.03	2.671 (12)	131
N21—H21···O237	0.86	2.12	2.73 (10)	129
C115—H115···O137^{i}	0.93	2.53	3.438 (9)	166
C215—H215···O237^{i}	0.93	2.13	3.04 (7)	166

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

N-[3-(2-Chlorobenzoyl)-5-ethylthiophen-2-yl]-4-phenylbenzamide (II)

Crystal data

$C_{26}H_{20}ClNO_2S$
M_r = 445.94
Monoclinic, $P2_1/c$
a = 10.7264 (6) Å
b = 9.1208 (5) Å
c = 22.9103 (15) Å
β = 94.414 (2)°
V = 2234.7 (2) Å^3
Z = 4
$F(000)$ = 928
D_x = 1.325 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7049 reflections
θ = 1.8–30.9°

μ = 0.29 mm^{-1}
T = 296 K

Block, colourless
0.28 × 0.27 × 0.22 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} = 0.929, T_{max} = 0.939
24401 measured reflections

5148 independent reflections
3570 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.6°, θ_{min} = 2.4°
h = −13→13
k = −11→11
l = −29→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.130
S = 1.04
5148 reflections
373 parameters
89 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 1.1314P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C11	0.3958 (6)	0.7159 (4)	0.49542 (14)	0.0388 (11)	0.832 (5)
O11	0.40738 (18)	0.81059 (17)	0.45845 (7)	0.0652 (5)	0.832 (5)
N11	0.3397 (12)	0.5857 (6)	0.4797 (3)	0.0398 (10)	0.832 (5)
H11	0.3373	0.5195	0.5064	0.048*	0.832 (5)
C111	0.4457 (9)	0.7310 (4)	0.55744 (15)	0.0352 (11)	0.832 (5)
C112	0.5052 (6)	0.8626 (4)	0.57421 (17)	0.0441 (13)	0.832 (5)
H112	0.5103	0.9375	0.5470	0.053*	0.832 (5)
C113	0.5562 (5)	0.8822 (5)	0.63050 (16)	0.0449 (12)	0.832 (5)
H113	0.5946	0.9709	0.6407	0.054*	0.832 (5)
C114	0.5519 (14)	0.7726 (8)	0.6727 (3)	0.0391 (5)	0.832 (5)
C115	0.4928 (14)	0.6418 (7)	0.6557 (2)	0.0430 (15)	0.832 (5)
H115	0.4875	0.5671	0.6831	0.052*	0.832 (5)
C116	0.4418 (11)	0.6208 (5)	0.59912 (17)	0.0410 (15)	0.832 (5)
H116	0.4044	0.5316	0.5888	0.049*	0.832 (5)
S121	0.2951 (2)	0.66308 (19)	0.36452 (9)	0.0482 (4)	0.832 (5)
C122	0.2865 (7)	0.5520 (5)	0.42463 (16)	0.0361 (11)	0.832 (5)
C123	0.2174 (7)	0.4269 (5)	0.41147 (18)	0.0374 (11)	0.832 (5)
C124	0.1733 (3)	0.4230 (3)	0.35089 (14)	0.0421 (10)	0.832 (5)
H124	0.1254	0.3464	0.3344	0.050*	0.832 (5)

C125	0.2070 (4)	0.5394 (4)	0.32011 (13)	0.0498 (11)	0.832 (5)
C126	0.1811 (4)	0.5720 (4)	0.25571 (13)	0.0697 (12)	0.832 (5)
H16A	0.2592	0.5691	0.2371	0.084*	0.832 (5)
H16B	0.1272	0.4960	0.2381	0.084*	0.832 (5)
C127	0.1202 (6)	0.7183 (5)	0.24404 (19)	0.123 (2)	0.832 (5)
H17A	0.0915	0.7252	0.2034	0.185*	0.832 (5)
H17B	0.1797	0.7948	0.2537	0.185*	0.832 (5)
H17C	0.0505	0.7285	0.2676	0.185*	0.832 (5)
C137	0.1907 (3)	0.3189 (4)	0.45606 (18)	0.0384 (6)	0.832 (5)
O137	0.2433 (7)	0.3199 (7)	0.5056 (2)	0.0531 (14)	0.832 (5)
C131	0.0991 (3)	0.1982 (3)	0.4394 (3)	0.0427 (5)	0.832 (5)
C132	−0.0272 (3)	0.2248 (3)	0.42736 (15)	0.0510 (8)	0.832 (5)
Cl12	−0.08524 (14)	0.4023 (2)	0.43201 (9)	0.0749 (4)	0.832 (5)
C133	−0.1105 (4)	0.1119 (5)	0.4147 (3)	0.0758 (14)	0.832 (5)
H133	−0.1954	0.1314	0.4074	0.091*	0.832 (5)
C134	−0.0674 (5)	−0.0279 (5)	0.4131 (5)	0.0955 (19)	0.832 (5)
H134	−0.1232	−0.1042	0.4042	0.115*	0.832 (5)
C135	0.0577 (5)	−0.0580 (4)	0.4244 (5)	0.0921 (13)	0.832 (5)
H135	0.0861	−0.1541	0.4226	0.111*	0.832 (5)
C136	0.1416 (4)	0.0547 (4)	0.4384 (4)	0.0625 (10)	0.832 (5)
H136	0.2260	0.0342	0.4470	0.075*	0.832 (5)
C141	0.6107 (9)	0.7926 (10)	0.73308 (18)	0.0418 (11)	0.832 (5)
C142	0.7192 (14)	0.8753 (18)	0.7432 (3)	0.0574 (11)	0.832 (5)
H142	0.7536	0.9212	0.7120	0.069*	0.832 (5)
C143	0.7770 (11)	0.8902 (12)	0.7991 (3)	0.0649 (12)	0.832 (5)
H143	0.8501	0.9448	0.8050	0.078*	0.832 (5)
C144	0.7267 (4)	0.8248 (7)	0.84572 (17)	0.0556 (11)	0.832 (5)
H144	0.7643	0.8376	0.8833	0.067*	0.832 (5)
C145	0.6218 (5)	0.7410 (8)	0.83703 (17)	0.0573 (12)	0.832 (5)
H145	0.5888	0.6946	0.8686	0.069*	0.832 (5)
C146	0.5641 (6)	0.7248 (9)	0.7809 (2)	0.0515 (15)	0.832 (5)
H146	0.4926	0.6673	0.7754	0.062*	0.832 (5)
C21	0.412 (4)	0.7075 (17)	0.4927 (7)	0.0388 (11)	0.168 (5)
O21	0.40738 (18)	0.81059 (17)	0.45845 (7)	0.0652 (5)	0.168 (5)
N21	0.347 (6)	0.582 (3)	0.4777 (13)	0.0398 (10)	0.168 (5)
H21	0.3536	0.5096	0.5018	0.048*	0.168 (5)
C211	0.458 (5)	0.722 (2)	0.5554 (7)	0.0352 (11)	0.168 (5)
C212	0.482 (4)	0.864 (2)	0.5769 (8)	0.0441 (13)	0.168 (5)
H212	0.4649	0.9447	0.5528	0.053*	0.168 (5)
C213	0.530 (3)	0.884 (3)	0.6336 (8)	0.0449 (12)	0.168 (5)
H213	0.5515	0.9779	0.6466	0.054*	0.168 (5)
C214	0.549 (7)	0.766 (4)	0.6721 (13)	0.0391 (5)	0.168 (5)
C215	0.502 (8)	0.630 (4)	0.6532 (11)	0.0430 (15)	0.168 (5)
H215	0.5036	0.5523	0.6795	0.052*	0.168 (5)
C216	0.455 (6)	0.609 (3)	0.5960 (9)	0.0410 (15)	0.168 (5)
H216	0.4203	0.5190	0.5847	0.049*	0.168 (5)
S221	0.2749 (12)	0.6821 (11)	0.3678 (5)	0.0482 (4)	0.168 (5)
C222	0.273 (4)	0.565 (3)	0.4264 (8)	0.0361 (11)	0.168 (5)

C223	0.204 (4)	0.440 (3)	0.4128 (9)	0.0374 (11)	0.168 (5)
C224	0.149 (2)	0.4448 (19)	0.3538 (7)	0.0421 (10)	0.168 (5)
H224	0.0943	0.3737	0.3381	0.050*	0.168 (5)
C225	0.184 (2)	0.561 (2)	0.3232 (5)	0.0498 (11)	0.168 (5)
C226	0.1403 (17)	0.606 (2)	0.2615 (6)	0.0697 (12)	0.168 (5)
H26A	0.1046	0.5211	0.2408	0.084*	0.168 (5)
H26B	0.0747	0.6787	0.2632	0.084*	0.168 (5)
C227	0.243 (2)	0.668 (3)	0.2278 (8)	0.123 (2)	0.168 (5)
H27A	0.2090	0.6948	0.1893	0.185*	0.168 (5)
H27B	0.3066	0.5950	0.2248	0.185*	0.168 (5)
H27C	0.2776	0.7524	0.2477	0.185*	0.168 (5)
C237	0.2050 (16)	0.3146 (18)	0.4529 (9)	0.0384 (6)	0.168 (5)
O237	0.263 (4)	0.317 (4)	0.5013 (12)	0.0531 (14)	0.168 (5)
C231	0.1151 (12)	0.1902 (12)	0.4391 (13)	0.0427 (5)	0.168 (5)
C232	−0.0129 (12)	0.2080 (11)	0.4390 (7)	0.0510 (8)	0.168 (5)
Cl22	−0.0753 (9)	0.3806 (11)	0.4505 (4)	0.0749 (4)	0.168 (5)
C233	−0.0937 (17)	0.0918 (16)	0.4281 (17)	0.0758 (14)	0.168 (5)
H233	−0.1797	0.1069	0.4254	0.091*	0.168 (5)
C234	−0.046 (2)	−0.0447 (17)	0.421 (3)	0.0955 (19)	0.168 (5)
H234	−0.1006	−0.1237	0.4148	0.115*	0.168 (5)
C235	0.080 (2)	−0.0673 (16)	0.424 (3)	0.0921 (13)	0.168 (5)
H235	0.1118	−0.1617	0.4202	0.111*	0.168 (5)
C236	0.1621 (18)	0.0502 (16)	0.432 (2)	0.0625 (10)	0.168 (5)
H236	0.2480	0.0352	0.4327	0.075*	0.168 (5)
C241	0.619 (5)	0.783 (5)	0.7301 (9)	0.0418 (11)	0.168 (5)
C242	0.715 (7)	0.886 (9)	0.7380 (15)	0.0574 (11)	0.168 (5)
H242	0.7375	0.9411	0.7063	0.069*	0.168 (5)
C243	0.778 (6)	0.907 (7)	0.7926 (16)	0.0649 (12)	0.168 (5)
H243	0.8355	0.9828	0.7984	0.078*	0.168 (5)
C244	0.753 (3)	0.816 (4)	0.8380 (10)	0.0556 (11)	0.168 (5)
H244	0.8077	0.8137	0.8716	0.067*	0.168 (5)
C245	0.649 (3)	0.730 (4)	0.8336 (8)	0.0573 (12)	0.168 (5)
H245	0.6225	0.6840	0.8667	0.069*	0.168 (5)
C246	0.584 (4)	0.711 (5)	0.7794 (11)	0.0515 (15)	0.168 (5)
H246	0.5144	0.6497	0.7762	0.062*	0.168 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.043 (2)	0.0336 (11)	0.0401 (12)	−0.0061 (12)	0.0016 (12)	0.0029 (9)
O11	0.0996 (13)	0.0470 (9)	0.0461 (9)	−0.0262 (9)	−0.0137 (9)	0.0143 (7)
N11	0.049 (2)	0.0364 (9)	0.0334 (10)	−0.0076 (11)	−0.0008 (12)	0.0053 (8)
C111	0.036 (2)	0.0323 (11)	0.0374 (11)	−0.0027 (13)	0.0027 (11)	0.0008 (9)
C112	0.056 (3)	0.0357 (11)	0.0405 (12)	−0.0113 (12)	0.0007 (14)	0.0061 (9)
C113	0.055 (3)	0.0367 (11)	0.0434 (13)	−0.0132 (15)	0.0043 (15)	−0.0037 (9)
C114	0.0417 (14)	0.0388 (12)	0.0371 (11)	−0.0002 (11)	0.0055 (8)	−0.0023 (9)
C115	0.051 (3)	0.0383 (16)	0.0394 (12)	−0.005 (2)	0.0016 (15)	0.0073 (11)
C116	0.047 (3)	0.0320 (13)	0.0430 (13)	−0.0078 (18)	−0.0006 (14)	0.0015 (10)

S121	0.0628 (9)	0.0437 (6)	0.0379 (4)	−0.0019 (5)	0.0022 (5)	0.0094 (4)
C122	0.038 (2)	0.0365 (14)	0.0337 (10)	0.0003 (16)	0.0028 (10)	0.0036 (9)
C123	0.039 (2)	0.0393 (14)	0.0340 (10)	0.0002 (16)	0.0017 (10)	−0.0001 (10)
C124	0.045 (2)	0.0449 (15)	0.0351 (12)	0.0024 (14)	−0.0021 (12)	−0.0053 (10)
C125	0.061 (2)	0.0515 (17)	0.0357 (12)	0.0066 (16)	−0.0011 (12)	0.0019 (11)
C126	0.096 (3)	0.076 (3)	0.0348 (15)	0.007 (2)	−0.0049 (17)	0.0089 (15)
C127	0.188 (5)	0.116 (4)	0.061 (3)	0.034 (4)	−0.026 (3)	0.032 (2)
C137	0.0413 (13)	0.0398 (11)	0.0343 (12)	−0.0030 (10)	0.0041 (10)	−0.0019 (9)
O137	0.070 (3)	0.0515 (10)	0.0364 (12)	−0.0188 (15)	−0.0062 (16)	0.0080 (9)
C131	0.0480 (14)	0.0439 (12)	0.0370 (11)	−0.0079 (10)	0.0069 (12)	−0.0018 (9)
C132	0.0507 (14)	0.0647 (16)	0.0369 (18)	−0.0097 (12)	−0.0018 (12)	0.0008 (12)
Cl12	0.0577 (5)	0.0824 (7)	0.0845 (11)	0.0191 (4)	0.0036 (7)	0.0109 (7)
C133	0.0593 (19)	0.099 (3)	0.067 (4)	−0.0310 (18)	−0.0057 (17)	−0.005 (2)
C134	0.102 (3)	0.084 (3)	0.101 (5)	−0.051 (2)	0.008 (3)	−0.023 (3)
C135	0.120 (4)	0.0459 (16)	0.115 (3)	−0.0192 (19)	0.036 (3)	−0.0180 (18)
C136	0.068 (2)	0.0465 (14)	0.076 (3)	−0.0061 (13)	0.021 (2)	−0.0044 (13)
C141	0.0490 (19)	0.0407 (17)	0.0357 (12)	0.0056 (15)	0.0032 (12)	−0.0064 (11)
C142	0.070 (2)	0.058 (3)	0.0430 (19)	−0.011 (2)	−0.0003 (18)	−0.0055 (17)
C143	0.0725 (17)	0.066 (4)	0.053 (2)	−0.006 (2)	−0.0123 (19)	−0.013 (2)
C144	0.073 (3)	0.0538 (16)	0.0384 (16)	0.024 (2)	−0.0051 (17)	−0.0122 (14)
C145	0.074 (3)	0.060 (2)	0.0380 (14)	0.017 (2)	0.0091 (15)	0.0025 (13)
C146	0.057 (3)	0.055 (2)	0.0429 (13)	0.003 (2)	0.0062 (14)	0.0006 (12)
C21	0.043 (2)	0.0336 (11)	0.0401 (12)	−0.0061 (12)	0.0016 (12)	0.0029 (9)
O21	0.0996 (13)	0.0470 (9)	0.0461 (9)	−0.0262 (9)	−0.0137 (9)	0.0143 (7)
N21	0.049 (2)	0.0364 (9)	0.0334 (10)	−0.0076 (11)	−0.0008 (12)	0.0053 (8)
C211	0.036 (2)	0.0323 (11)	0.0374 (11)	−0.0027 (13)	0.0027 (11)	0.0008 (9)
C212	0.056 (3)	0.0357 (11)	0.0405 (12)	−0.0113 (12)	0.0007 (14)	0.0061 (9)
C213	0.055 (3)	0.0367 (11)	0.0434 (13)	−0.0132 (15)	0.0043 (15)	−0.0037 (9)
C214	0.0417 (14)	0.0388 (12)	0.0371 (11)	−0.0002 (11)	0.0055 (8)	−0.0023 (9)
C215	0.051 (3)	0.0383 (16)	0.0394 (12)	−0.005 (2)	0.0016 (15)	0.0073 (11)
C216	0.047 (3)	0.0320 (13)	0.0430 (13)	−0.0078 (18)	−0.0006 (14)	0.0015 (10)
S221	0.0628 (9)	0.0437 (6)	0.0379 (4)	−0.0019 (5)	0.0022 (5)	0.0094 (4)
C222	0.038 (2)	0.0365 (14)	0.0337 (10)	0.0003 (16)	0.0028 (10)	0.0036 (9)
C223	0.039 (2)	0.0393 (14)	0.0340 (10)	0.0002 (16)	0.0017 (10)	−0.0001 (10)
C224	0.045 (2)	0.0449 (15)	0.0351 (12)	0.0024 (14)	−0.0021 (12)	−0.0053 (10)
C225	0.061 (2)	0.0515 (17)	0.0357 (12)	0.0066 (16)	−0.0011 (12)	0.0019 (11)
C226	0.096 (3)	0.076 (3)	0.0348 (15)	0.007 (2)	−0.0049 (17)	0.0089 (15)
C227	0.188 (5)	0.116 (4)	0.061 (3)	0.034 (4)	−0.026 (3)	0.032 (2)
C237	0.0413 (13)	0.0398 (11)	0.0343 (12)	−0.0030 (10)	0.0041 (10)	−0.0019 (9)
O237	0.070 (3)	0.0515 (10)	0.0364 (12)	−0.0188 (15)	−0.0062 (16)	0.0080 (9)
C231	0.0480 (14)	0.0439 (12)	0.0370 (11)	−0.0079 (10)	0.0069 (12)	−0.0018 (9)
C232	0.0507 (14)	0.0647 (16)	0.0369 (18)	−0.0097 (12)	−0.0018 (12)	0.0008 (12)
Cl22	0.0577 (5)	0.0824 (7)	0.0845 (11)	0.0191 (4)	0.0036 (7)	0.0109 (7)
C233	0.0593 (19)	0.099 (3)	0.067 (4)	−0.0310 (18)	−0.0057 (17)	−0.005 (2)
C234	0.102 (3)	0.084 (3)	0.101 (5)	−0.051 (2)	0.008 (3)	−0.023 (3)
C235	0.120 (4)	0.0459 (16)	0.115 (3)	−0.0192 (19)	0.036 (3)	−0.0180 (18)
C236	0.068 (2)	0.0465 (14)	0.076 (3)	−0.0061 (13)	0.021 (2)	−0.0044 (13)
C241	0.0490 (19)	0.0407 (17)	0.0357 (12)	0.0056 (15)	0.0032 (12)	−0.0064 (11)

C242	0.070 (2)	0.058 (3)	0.0430 (19)	−0.011 (2)	−0.0003 (18)	−0.0055 (17)
C243	0.0725 (17)	0.066 (4)	0.053 (2)	−0.006 (2)	−0.0123 (19)	−0.013 (2)
C244	0.073 (3)	0.0538 (16)	0.0384 (16)	0.024 (2)	−0.0051 (17)	−0.0122 (14)
C245	0.074 (3)	0.060 (2)	0.0380 (14)	0.017 (2)	0.0091 (15)	0.0025 (13)
C246	0.057 (3)	0.055 (2)	0.0429 (13)	0.003 (2)	0.0062 (14)	0.0006 (12)

Geometric parameters (Å, º)

C11—O11	1.223 (3)	C21—N21	1.370 (5)
C11—N11	1.367 (3)	C21—C211	1.486 (5)
C11—C111	1.486 (3)	N21—C222	1.381 (6)
N11—C122	1.379 (4)	N21—H21	0.8600
N11—H11	0.8600	C211—C216	1.391 (5)
C111—C116	1.389 (3)	C211—C212	1.401 (6)
C111—C112	1.399 (4)	C212—C213	1.374 (5)
C112—C113	1.373 (3)	C212—H212	0.9300
C112—H112	0.9300	C213—C214	1.394 (6)
C113—C114	1.394 (4)	C213—H213	0.9300
C113—H113	0.9300	C214—C215	1.392 (5)
C114—C115	1.392 (4)	C214—C241	1.487 (5)
C114—C141	1.487 (3)	C215—C216	1.383 (5)
C115—C116	1.382 (3)	C215—H215	0.9300
C115—H115	0.9300	C216—H216	0.9300
C116—H116	0.9300	S221—C222	1.719 (4)
S121—C122	1.718 (2)	S221—C225	1.748 (5)
S121—C125	1.747 (3)	C222—C223	1.382 (5)
C122—C123	1.381 (3)	C223—C224	1.431 (5)
C123—C124	1.432 (3)	C223—C237	1.464 (6)
C123—C137	1.463 (4)	C224—C225	1.341 (5)
C124—C125	1.340 (3)	C224—H224	0.9300
C124—H124	0.9300	C225—C226	1.511 (5)
C125—C126	1.510 (3)	C226—C227	1.500 (7)
C126—C127	1.501 (6)	C226—H26A	0.9700
C126—H16A	0.9700	C226—H26B	0.9700
C126—H16B	0.9700	C227—H27A	0.9600
C127—H17A	0.9600	C227—H27B	0.9600
C127—H17B	0.9600	C227—H27C	0.9600
C127—H17C	0.9600	C237—O237	1.230 (5)
C137—O137	1.228 (3)	C237—C231	1.506 (5)
C137—C131	1.506 (3)	C231—C232	1.382 (5)
C131—C132	1.382 (3)	C231—C236	1.387 (5)
C131—C136	1.387 (4)	C232—C233	1.380 (5)
C132—C133	1.380 (4)	C232—Cl22	1.739 (5)
C132—Cl12	1.741 (3)	C233—C234	1.358 (6)
C133—C134	1.358 (5)	C233—H233	0.9300
C133—H133	0.9300	C234—C235	1.374 (7)
C134—C135	1.375 (5)	C234—H234	0.9300
C134—H134	0.9300	C235—C236	1.388 (6)

C135—C136	1.388 (4)	C235—H235	0.9300
C135—H135	0.9300	C236—H236	0.9300
C136—H136	0.9300	C241—C246	1.385 (5)
C141—C146	1.385 (3)	C241—C242	1.392 (7)
C141—C142	1.392 (4)	C242—C243	1.386 (5)
C142—C143	1.385 (4)	C242—H242	0.9300
C142—H142	0.9300	C243—C244	1.370 (8)
C143—C144	1.370 (6)	C243—H243	0.9300
C143—H143	0.9300	C244—C245	1.364 (6)
C144—C145	1.363 (4)	C244—H244	0.9300
C144—H144	0.9300	C245—C246	1.392 (5)
C145—C146	1.391 (4)	C245—H245	0.9300
C145—H145	0.9300	C246—H246	0.9300
C146—H146	0.9300		

O11—C11—N11	119.8 (2)	C145—C146—H146	119.3
O11—C11—C111	123.3 (2)	N21—C21—C211	116.1 (7)
N11—C11—C111	116.8 (2)	C21—N21—C222	124.1 (8)
C11—N11—C122	125.2 (2)	C21—N21—H21	118.0
C11—N11—H11	117.4	C222—N21—H21	118.0
C122—N11—H11	117.4	C216—C211—C212	117.6 (9)
C116—C111—C112	118.0 (2)	C216—C211—C21	123.8 (7)
C116—C111—C11	124.2 (2)	C212—C211—C21	117.4 (7)
C112—C111—C11	117.7 (2)	C213—C212—C211	120.0 (7)
C113—C112—C111	120.6 (2)	C213—C212—H212	120.0
C113—C112—H112	119.7	C211—C212—H212	120.0
C111—C112—H112	119.7	C212—C213—C214	121.4 (7)
C112—C113—C114	121.7 (2)	C212—C213—H213	119.3
C112—C113—H113	119.1	C214—C213—H213	119.3
C114—C113—H113	119.1	C215—C214—C213	117.5 (6)
C115—C114—C113	117.4 (2)	C215—C214—C241	121.1 (8)
C115—C114—C141	121.1 (2)	C213—C214—C241	121.4 (6)
C113—C114—C141	121.5 (2)	C216—C215—C214	120.9 (6)
C116—C115—C114	121.3 (3)	C216—C215—H215	119.5
C116—C115—H115	119.3	C214—C215—H215	119.5
C114—C115—H115	119.3	C215—C216—C211	120.3 (6)
C115—C116—C111	120.9 (2)	C215—C216—H216	119.8
C115—C116—H116	119.5	C211—C216—H216	119.8
C111—C116—H116	119.5	C222—S221—C225	91.3 (3)
C122—S121—C125	91.49 (12)	N21—C222—C223	123.5 (7)
N11—C122—C123	124.1 (2)	N21—C222—S221	123.5 (7)
N11—C122—S121	123.93 (18)	C223—C222—S221	112.1 (4)
C123—C122—S121	111.91 (17)	C222—C223—C224	111.0 (4)
C122—C123—C124	111.4 (2)	C222—C223—C237	121.5 (7)
C122—C123—C137	122.3 (2)	C224—C223—C237	127.0 (8)
C124—C123—C137	126.3 (2)	C225—C224—C223	114.2 (5)
C125—C124—C123	114.1 (2)	C225—C224—H224	122.9
C125—C124—H124	123.0	C223—C224—H224	122.9

C123—C124—H124	123.0	C224—C225—C226	128.5 (7)
C124—C125—C126	129.3 (3)	C224—C225—S221	111.0 (4)
C124—C125—S121	111.17 (18)	C226—C225—S221	119.7 (6)
C126—C125—S121	119.5 (2)	C227—C226—C225	113.2 (8)
C127—C126—C125	113.2 (3)	C227—C226—H26A	108.9
C127—C126—H16A	108.9	C225—C226—H26A	108.9
C125—C126—H16A	108.9	C227—C226—H26B	108.9
C127—C126—H16B	108.9	C225—C226—H26B	108.9
C125—C126—H16B	108.9	H26A—C226—H26B	107.7
H16A—C126—H16B	107.8	C226—C227—H27A	109.5
C126—C127—H17A	109.5	C226—C227—H27B	109.5
C126—C127—H17B	109.5	H27A—C227—H27B	109.5
H17A—C127—H17B	109.5	C226—C227—H27C	109.5
C126—C127—H17C	109.5	H27A—C227—H27C	109.5
H17A—C127—H17C	109.5	H27B—C227—H27C	109.5
H17B—C127—H17C	109.5	O237—C237—C223	122.0 (8)
O137—C137—C123	122.7 (2)	O237—C237—C231	118.4 (7)
O137—C137—C131	118.9 (2)	C223—C237—C231	118.7 (6)
C123—C137—C131	118.3 (2)	C232—C231—C236	118.5 (5)
C132—C131—C136	118.8 (2)	C232—C231—C237	122.1 (6)
C132—C131—C137	122.2 (2)	C236—C231—C237	119.0 (6)
C136—C131—C137	119.0 (2)	C233—C232—C231	121.4 (6)
C133—C132—C131	121.3 (3)	C233—C232—Cl22	118.6 (6)
C133—C132—Cl12	118.6 (2)	C231—C232—Cl22	120.0 (6)
C131—C132—Cl12	120.0 (2)	C234—C233—C232	119.3 (6)
C134—C133—C132	119.4 (3)	C234—C233—H233	120.3
C134—C133—H133	120.3	C232—C233—H233	120.3
C132—C133—H133	120.3	C233—C234—C235	120.7 (6)
C133—C134—C135	120.8 (3)	C233—C234—H234	119.7
C133—C134—H134	119.6	C235—C234—H234	119.7
C135—C134—H134	119.6	C234—C235—C236	120.2 (6)
C134—C135—C136	120.2 (3)	C234—C235—H235	119.9
C134—C135—H135	119.9	C236—C235—H235	119.9
C136—C135—H135	119.9	C231—C236—C235	119.7 (6)
C131—C136—C135	119.6 (3)	C231—C236—H236	120.1
C131—C136—H136	120.2	C235—C236—H236	120.1
C135—C136—H136	120.2	C246—C241—C242	117.4 (6)
C146—C141—C142	117.3 (2)	C246—C241—C214	121.6 (7)
C146—C141—C114	121.9 (2)	C242—C241—C214	120.6 (7)
C142—C141—C114	120.7 (3)	C243—C242—C241	120.8 (6)
C143—C142—C141	121.1 (4)	C243—C242—H242	119.6
C143—C142—H142	119.4	C241—C242—H242	119.6
C141—C142—H142	119.4	C244—C243—C242	119.6 (10)
C144—C143—C142	120.2 (4)	C244—C243—H243	120.2
C144—C143—H143	119.9	C242—C243—H243	120.2
C142—C143—H143	119.9	C245—C244—C243	119.7 (8)
C145—C144—C143	120.1 (3)	C245—C244—H244	120.1
C145—C144—H144	120.0	C243—C244—H244	119

C143—C144—H144	120.0	C244—C245—C246	119.4 (7)
C144—C145—C146	119.9 (3)	C244—C245—H245	120.3
C144—C145—H145	120.1	C246—C245—H245	120.3
C146—C145—H145	120.1	C241—C246—C245	121.2 (6)
C141—C146—C145	121.5 (3)	C241—C246—H246	119.4
C141—C146—H146	119.3	C245—C246—H246	119.4
O11—C11—N11—C122	−3.8 (14)	C144—C145—C146—C141	−0.1 (12)
C111—C11—N11—C122	179.1 (11)	C211—C21—N21—C222	163 (6)
O11—C11—C111—C116	−175.0 (9)	N21—C21—C211—C216	10 (6)
N11—C11—C111—C116	1.9 (10)	N21—C21—C211—C212	−157 (5)
O11—C11—C111—C112	2.1 (10)	C216—C211—C212—C213	16 (6)
N11—C11—C111—C112	179.1 (9)	C21—C211—C212—C213	−177 (4)
C116—C111—C112—C113	−1.0 (10)	C211—C212—C213—C214	−5 (7)
C11—C111—C112—C113	−178.3 (6)	C212—C213—C214—C215	−7 (8)
C111—C112—C113—C114	0.6 (12)	C212—C213—C214—C241	170 (4)
C112—C113—C114—C115	−0.5 (16)	C213—C214—C215—C216	8 (10)
C112—C113—C114—C141	178.1 (7)	C241—C214—C215—C216	−169 (7)
C113—C114—C115—C116	0.8 (18)	C214—C215—C216—C211	3 (10)
C141—C114—C115—C116	−177.7 (12)	C212—C211—C216—C215	−15 (8)
C114—C115—C116—C111	−1.3 (19)	C21—C211—C216—C215	178 (6)
C112—C111—C116—C115	1.4 (14)	C21—N21—C222—C223	−178 (5)
C11—C111—C116—C115	178.5 (11)	C21—N21—C222—S221	14 (8)
C11—N11—C122—C123	−171.9 (10)	C225—S221—C222—N21	169 (4)
C11—N11—C122—S121	6.3 (15)	C225—S221—C222—C223	−1 (4)
C125—S121—C122—N11	−179.5 (8)	N21—C222—C223—C224	−172 (5)
C125—S121—C122—C123	−1.0 (6)	S221—C222—C223—C224	−2 (5)
N11—C122—C123—C124	179.3 (8)	N21—C222—C223—C237	0 (7)
S121—C122—C123—C124	0.9 (8)	S221—C222—C223—C237	170 (4)
N11—C122—C123—C137	1.9 (13)	C222—C223—C224—C225	5 (5)
S121—C122—C123—C137	−176.6 (6)	C237—C223—C224—C225	−166 (4)
C122—C123—C124—C125	−0.2 (8)	C223—C224—C225—C226	−176 (3)
C137—C123—C124—C125	177.1 (6)	C223—C224—C225—S221	−6 (4)
C123—C124—C125—C126	178.4 (5)	C222—S221—C225—C224	4 (3)
C123—C124—C125—S121	−0.5 (6)	C222—S221—C225—C226	175 (3)
C122—S121—C125—C124	0.9 (4)	C224—C225—C226—C227	−142 (3)
C122—S121—C125—C126	−178.2 (4)	S221—C225—C226—C227	49 (3)
C124—C125—C126—C127	125.9 (5)	C222—C223—C237—O237	3 (6)
S121—C125—C126—C127	−55.2 (5)	C224—C223—C237—O237	174 (4)
C122—C123—C137—O137	−11.7 (11)	C222—C223—C237—C231	172 (3)
C124—C123—C137—O137	171.3 (7)	C224—C223—C237—C231	−17 (6)
C122—C123—C137—C131	171.0 (6)	O237—C237—C231—C232	104 (4)
C124—C123—C137—C131	−6.0 (10)	C223—C237—C231—C232	−65 (3)
O137—C137—C131—C132	113.7 (7)	O237—C237—C231—C236	−69 (4)
C123—C137—C131—C132	−68.8 (7)	C223—C237—C231—C236	121 (3)
O137—C137—C131—C136	−63.6 (7)	C236—C231—C232—C233	−5 (3)
C123—C137—C131—C136	113.8 (6)	C237—C231—C232—C233	−178 (2)
C136—C131—C132—C133	0.3 (6)	C236—C231—C232—Cl22	177.6 (19)

C137—C131—C132—C133	−177.1 (4)	C237—C231—C232—Cl22	4 (2)
C136—C131—C132—Cl12	176.6 (4)	C231—C232—C233—C234	5 (4)
C137—C131—C132—Cl12	−0.7 (5)	Cl22—C232—C233—C234	−177 (3)
C131—C132—C133—C134	−1.2 (8)	C232—C233—C234—C235	−2 (6)
Cl12—C132—C133—C134	−177.5 (6)	C233—C234—C235—C236	−2 (7)
C132—C133—C134—C135	0.6 (12)	C232—C231—C236—C235	1 (4)
C133—C134—C135—C136	0.8 (13)	C237—C231—C236—C235	175 (4)
C132—C131—C136—C135	1.1 (9)	C234—C235—C236—C231	2 (6)
C137—C131—C136—C135	178.6 (7)	C215—C214—C241—C246	−41 (7)
C134—C135—C136—C131	−1.7 (12)	C213—C214—C241—C246	142 (7)
C115—C114—C141—C146	−31.7 (13)	C215—C214—C241—C242	147 (8)
C113—C114—C141—C146	149.9 (13)	C213—C214—C241—C242	−30 (8)
C115—C114—C141—C142	145.2 (16)	C246—C241—C242—C243	4 (12)
C113—C114—C141—C142	−33.3 (16)	C214—C241—C242—C243	177 (7)
C146—C141—C142—C143	−1 (2)	C241—C242—C243—C244	6 (12)
C114—C141—C142—C143	−177.7 (13)	C242—C243—C244—C245	−16 (9)
C141—C142—C143—C144	−1 (2)	C243—C244—C245—C246	14 (6)
C142—C143—C144—C145	2.0 (16)	C242—C241—C246—C245	−7 (9)
C143—C144—C145—C146	−1.6 (10)	C214—C241—C246—C245	−179 (4)
C142—C141—C146—C145	1.2 (16)	C244—C245—C246—C241	−2 (7)
C114—C141—C146—C145	178.2 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O137	0.86	2.08	2.719 (10)	130
N21—H21···O237	0.86	2.01	2.65 (5)	131
C112—H112···O11^{i}	0.93	2.47	3.230 (4)	139
C144—H144···O137^{ii}	0.93	2.56	3.397 (6)	150

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

2-Bromo-*N*-[3-(2-chlorobenzoyl)-5-ethylthiophen-2-yl]benzamide (III)

Crystal data

$C_{20}H_{15}BrClNO_2S$
M_r = 448.74
Monoclinic, *C*2/*c*
a = 18.8096 (10) Å
b = 11.5021 (7) Å
c = 19.9112 (11) Å
β = 118.030 (2)°
V = 3802.5 (4) Å^3
Z = 8

F(000) = 1808
D_x = 1.568 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4009 reflections
θ = 2.2–26.8°
μ = 2.43 mm^{-1}
T = 294 K
Block, yellow
0.25 × 0.20 × 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} = 0.614, T_{max} = 0.785
25510 measured reflections
4009 independent reflections

2370 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 26.8°$, $\theta_{min} = 2.2°$

$h = -23 \rightarrow 23$
$k = -14 \rightarrow 14$
$l = -24 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.122$
$S = 1.01$
4009 reflections
322 parameters
64 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 2.9782P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ 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)
C11	0.5823 (3)	0.5906 (4)	0.4593 (4)	0.0496 (13)	0.7006 (12)
O11	0.5148 (4)	0.5810 (7)	0.4072 (8)	0.0687 (16)	0.7006 (12)
N11	0.6219 (6)	0.4975 (5)	0.5055 (8)	0.0482 (17)	0.7006 (12)
H11	0.6672	0.5118	0.5448	0.058*	0.7006 (12)
C111	0.6254 (3)	0.7051 (3)	0.4898 (3)	0.0469 (11)	0.7006 (12)
C112	0.6211 (3)	0.7899 (4)	0.4387 (3)	0.0614 (12)	0.7006 (12)
C113	0.6670 (4)	0.8934 (5)	0.4679 (5)	0.0871 (18)	0.7006 (12)
H113	0.6644	0.9522	0.4348	0.105*	0.7006 (12)
C114	0.7141 (3)	0.9067 (5)	0.5434 (4)	0.0749 (16)	0.7006 (12)
H114	0.7443	0.9743	0.5615	0.090*	0.7006 (12)
C115	0.7185 (3)	0.8245 (4)	0.5931 (3)	0.0747 (14)	0.7006 (12)
H115	0.7512	0.8357	0.6450	0.090*	0.7006 (12)
C116	0.6745 (3)	0.7236 (4)	0.5671 (4)	0.0559 (12)	0.7006 (12)
H116	0.6777	0.6671	0.6018	0.067*	0.7006 (12)
S121	0.5058 (4)	0.3394 (6)	0.4205 (4)	0.0503 (10)	0.7006 (12)
C122	0.5958 (8)	0.3838 (5)	0.4946 (8)	0.042 (2)	0.7006 (12)
C123	0.6416 (7)	0.2913 (6)	0.5371 (8)	0.0467 (15)	0.7006 (12)
C124	0.5995 (6)	0.1840 (5)	0.5103 (6)	0.0467 (15)	0.7006 (12)
H124	0.6190	0.1135	0.5351	0.056*	0.7006 (12)
C125	0.5291 (10)	0.1936 (6)	0.4460 (12)	0.0523 (9)	0.7006 (12)
C126	0.4719 (10)	0.0994 (9)	0.3996 (10)	0.0660 (10)	0.7006 (12)
H16A	0.4217	0.1088	0.4015	0.079*	0.7006 (12)
H16B	0.4945	0.0247	0.4220	0.079*	0.7006 (12)
C127	0.4548 (12)	0.100 (2)	0.3178 (9)	0.120 (6)	0.7006 (12)
H17A	0.5047	0.0973	0.3156	0.180*	0.7006 (12)
H17B	0.4260	0.1692	0.2934	0.180*	0.7006 (12)
H17C	0.4229	0.0330	0.2923	0.180*	0.7006 (12)

C137	0.7199 (5)	0.3072 (6)	0.6033 (5)	0.0491 (11)	0.7006 (12)
O137	0.7551 (8)	0.4013 (6)	0.6196 (11)	0.0694 (13)	0.7006 (12)
C131	0.7633 (5)	0.2024 (7)	0.6494 (6)	0.0459 (13)	0.7006 (12)
C132	0.7380 (10)	0.1439 (15)	0.695 (2)	0.0590 (12)	0.7006 (12)
Cl12	0.6507 (7)	0.1871 (8)	0.6970 (8)	0.0916 (12)	0.7006 (12)
C133	0.7822 (13)	0.050 (2)	0.7400 (16)	0.0754 (17)	0.7006 (12)
H133	0.7642	0.0092	0.7694	0.090*	0.7006 (12)
C134	0.8519 (13)	0.020 (2)	0.7397 (17)	0.0838 (16)	0.7006 (12)
H134	0.8829	−0.0402	0.7710	0.101*	0.7006 (12)
C135	0.8777 (13)	0.076 (2)	0.6946 (19)	0.080 (3)	0.7006 (12)
H135	0.9240	0.0507	0.6933	0.096*	0.7006 (12)
C136	0.8354 (12)	0.170 (2)	0.6510 (17)	0.063 (2)	0.7006 (12)
H136	0.8549	0.2108	0.6228	0.076*	0.7006 (12)
Br12	0.56130 (4)	0.7721 (7)	0.33313 (4)	0.0997 (3)	0.7006 (12)
C21	0.5651 (9)	0.5994 (9)	0.4558 (8)	0.0496 (13)	0.2994 (12)
O21	0.4990 (12)	0.5722 (17)	0.4048 (19)	0.0687 (16)	0.2994 (12)
N21	0.6193 (14)	0.5109 (11)	0.494 (2)	0.0482 (17)	0.2994 (12)
H21	0.6683	0.5292	0.5250	0.058*	0.2994 (12)
C211	0.6065 (7)	0.7113 (8)	0.4527 (5)	0.0469 (11)	0.2994 (12)
C212	0.5735 (6)	0.7690 (8)	0.3833 (6)	0.0614 (12)	0.2994 (12)
H212	0.5302	0.7378	0.3406	0.074*	0.2994 (12)
C213	0.6082 (6)	0.878 (2)	0.3795 (7)	0.0871 (18)	0.2994 (12)
H213	0.5938	0.9126	0.3326	0.105*	0.2994 (12)
C214	0.6616 (8)	0.9299 (9)	0.4440 (7)	0.0749 (16)	0.2994 (12)
H214	0.6806	1.0036	0.4415	0.090*	0.2994 (12)
C215	0.6883 (10)	0.8782 (10)	0.5119 (7)	0.0747 (14)	0.2994 (12)
H215	0.7245	0.9171	0.5556	0.090*	0.2994 (12)
C216	0.6627 (6)	0.7681 (8)	0.5178 (6)	0.0559 (12)	0.2994 (12)
S221	0.5091 (11)	0.345 (2)	0.4115 (10)	0.0503 (10)	0.2994 (12)
C222	0.5979 (19)	0.3948 (10)	0.485 (2)	0.042 (2)	0.2994 (12)
C223	0.6420 (16)	0.3068 (10)	0.5338 (18)	0.043 (4)	0.2994 (12)
C224	0.6063 (14)	0.1960 (10)	0.505 (2)	0.0467 (15)	0.2994 (12)
H224	0.6326	0.1259	0.5241	0.056*	0.2994 (12)
C225	0.531 (2)	0.2026 (14)	0.446 (3)	0.0523 (9)	0.2994 (12)
C226	0.475 (2)	0.1052 (19)	0.404 (2)	0.0660 (10)	0.2994 (12)
H26A	0.4345	0.0983	0.4207	0.079*	0.2994 (12)
H26B	0.5048	0.0330	0.4154	0.079*	0.2994 (12)
C227	0.433 (3)	0.123 (5)	0.319 (2)	0.120 (6)	0.2994 (12)
H27A	0.4024	0.0545	0.2942	0.180*	0.2994 (12)
H27B	0.4724	0.1363	0.3019	0.180*	0.2994 (12)
H27C	0.3976	0.1885	0.3060	0.180*	0.2994 (12)
C237	0.7215 (12)	0.327 (2)	0.5976 (13)	0.0491 (11)	0.2994 (12)
O237	0.751 (2)	0.4247 (17)	0.616 (3)	0.0694 (13)	0.2994 (12)
C231	0.7665 (11)	0.226 (2)	0.6470 (14)	0.0459 (13)	0.2994 (12)
C232	0.737 (2)	0.162 (4)	0.688 (3)	0.0590 (12)	0.2994 (12)
Cl22	0.6515 (17)	0.209 (2)	0.6916 (19)	0.0916 (12)	0.2994 (12)
C233	0.782 (3)	0.070 (5)	0.734 (4)	0.0754 (17)	0.2994 (12)
H233	0.7650	0.0321	0.7656	0.090*	0.2994 (12)

C234	0.850 (3)	0.035 (5)	0.731 (4)	0.0838 (16)	0.2994 (12)
H234	0.8766	−0.0309	0.7580	0.101*	0.2994 (12)
C235	0.879 (3)	0.096 (5)	0.691 (4)	0.080 (3)	0.2994 (12)
H235	0.9275	0.0735	0.6928	0.096*	0.2994 (12)
C236	0.837 (3)	0.189 (5)	0.646 (4)	0.063 (2)	0.2994 (12)
H236	0.8552	0.2270	0.6164	0.076*	0.2994 (12)
Br26	0.70489 (8)	0.7080 (2)	0.61638 (8)	0.0658 (5)	0.2994 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.039 (3)	0.0408 (19)	0.074 (2)	0.0098 (19)	0.030 (2)	−0.0003 (17)
O11	0.044 (3)	0.0569 (18)	0.0816 (18)	−0.0024 (16)	0.010 (3)	0.0079 (17)
N11	0.0462 (15)	0.0374 (19)	0.051 (5)	−0.0027 (15)	0.0141 (17)	0.0005 (18)
C111	0.045 (3)	0.0412 (19)	0.055 (3)	0.0048 (19)	0.023 (3)	0.001 (3)
C112	0.053 (3)	0.049 (3)	0.090 (4)	0.002 (2)	0.040 (2)	0.006 (3)
C113	0.087 (4)	0.056 (3)	0.143 (5)	0.020 (3)	0.074 (5)	0.030 (4)
C114	0.056 (3)	0.049 (3)	0.120 (5)	−0.001 (2)	0.042 (3)	−0.013 (3)
C115	0.069 (3)	0.052 (3)	0.095 (4)	−0.004 (2)	0.030 (3)	−0.018 (3)
C116	0.050 (3)	0.047 (3)	0.060 (4)	0.001 (2)	0.016 (3)	−0.005 (2)
S121	0.0446 (7)	0.0471 (8)	0.0521 (18)	−0.0037 (5)	0.0170 (10)	−0.0004 (9)
C122	0.0428 (16)	0.0407 (19)	0.045 (4)	−0.0039 (18)	0.022 (2)	0.0008 (18)
C123	0.052 (2)	0.0352 (17)	0.054 (2)	−0.0015 (15)	0.0254 (18)	0.0012 (15)
C124	0.052 (2)	0.0352 (17)	0.054 (2)	−0.0015 (15)	0.0254 (18)	0.0012 (15)
C125	0.0529 (19)	0.044 (2)	0.0585 (19)	−0.0060 (18)	0.0246 (16)	−0.008 (2)
C126	0.059 (2)	0.053 (2)	0.077 (3)	−0.0120 (18)	0.0243 (19)	−0.0149 (19)
C127	0.168 (13)	0.079 (11)	0.065 (3)	−0.043 (9)	0.016 (6)	−0.018 (4)
C137	0.0540 (18)	0.040 (3)	0.049 (2)	−0.0018 (19)	0.0205 (16)	0.001 (2)
O137	0.060 (2)	0.036 (3)	0.079 (2)	−0.005 (3)	0.0047 (14)	0.000 (4)
C131	0.0516 (18)	0.038 (4)	0.0402 (17)	−0.004 (2)	0.0151 (14)	0.002 (2)
C132	0.073 (2)	0.051 (6)	0.053 (4)	−0.003 (3)	0.030 (2)	0.000 (3)
Cl12	0.1105 (8)	0.088 (4)	0.113 (2)	−0.002 (2)	0.0822 (10)	0.004 (2)
C133	0.106 (3)	0.060 (8)	0.056 (5)	−0.009 (4)	0.035 (3)	0.010 (4)
C134	0.090 (3)	0.069 (7)	0.058 (7)	0.014 (3)	0.006 (4)	0.021 (3)
C135	0.059 (2)	0.080 (9)	0.080 (4)	0.017 (5)	0.017 (2)	0.016 (5)
C136	0.053 (2)	0.061 (8)	0.062 (4)	0.010 (4)	0.0158 (18)	0.009 (4)
Br12	0.1039 (5)	0.1178 (6)	0.0784 (5)	0.0125 (4)	0.0437 (4)	0.0350 (4)
C21	0.039 (3)	0.0408 (19)	0.074 (2)	0.0098 (19)	0.030 (2)	−0.0003 (17)
O21	0.044 (3)	0.0569 (18)	0.0816 (18)	−0.0024 (16)	0.010 (3)	0.0079 (17)
N21	0.0462 (15)	0.0374 (19)	0.051 (5)	−0.0027 (15)	0.0141 (17)	0.0005 (18)
C211	0.045 (3)	0.0412 (19)	0.055 (3)	0.0048 (19)	0.023 (3)	0.001 (3)
C212	0.053 (3)	0.049 (3)	0.090 (4)	0.002 (2)	0.040 (2)	0.006 (3)
C213	0.087 (4)	0.056 (3)	0.143 (5)	0.020 (3)	0.074 (5)	0.030 (4)
C214	0.056 (3)	0.049 (3)	0.120 (5)	−0.001 (2)	0.042 (3)	−0.013 (3)
C215	0.069 (3)	0.052 (3)	0.095 (4)	−0.004 (2)	0.030 (3)	−0.018 (3)
C216	0.050 (3)	0.047 (3)	0.060 (4)	0.001 (2)	0.016 (3)	−0.005 (2)
S221	0.0446 (7)	0.0471 (8)	0.0521 (18)	−0.0037 (5)	0.0170 (10)	−0.0004 (9)
C222	0.0428 (16)	0.0407 (19)	0.045 (4)	−0.0039 (18)	0.022 (2)	0.0008 (18)

C223	0.039 (6)	0.035 (7)	0.037 (7)	−0.002 (6)	0.003 (5)	−0.005 (7)
C224	0.052 (2)	0.0352 (17)	0.054 (2)	−0.0015 (15)	0.0254 (18)	0.0012 (15)
C225	0.0529 (19)	0.044 (2)	0.0585 (19)	−0.0060 (18)	0.0246 (16)	−0.008 (2)
C226	0.059 (2)	0.053 (2)	0.077 (3)	−0.0120 (18)	0.0243 (19)	−0.0149 (19)
C227	0.168 (13)	0.079 (11)	0.065 (3)	−0.043 (9)	0.016 (6)	−0.018 (4)
C237	0.0540 (18)	0.040 (3)	0.049 (2)	−0.0018 (19)	0.0205 (16)	0.001 (2)
O237	0.060 (2)	0.036 (3)	0.079 (2)	−0.005 (3)	0.0047 (14)	0.000 (4)
C231	0.0516 (18)	0.038 (4)	0.0402 (17)	−0.004 (2)	0.0151 (14)	0.002 (2)
C232	0.073 (2)	0.051 (6)	0.053 (4)	−0.003 (3)	0.030 (2)	0.000 (3)
Cl22	0.1105 (8)	0.088 (4)	0.113 (2)	−0.002 (2)	0.0822 (10)	0.004 (2)
C233	0.106 (3)	0.060 (8)	0.056 (5)	−0.009 (4)	0.035 (3)	0.010 (4)
C234	0.090 (3)	0.069 (7)	0.058 (7)	0.014 (3)	0.006 (4)	0.021 (3)
C235	0.059 (2)	0.080 (9)	0.080 (4)	0.017 (5)	0.017 (2)	0.016 (5)
C236	0.053 (2)	0.061 (8)	0.062 (4)	0.010 (4)	0.0158 (18)	0.009 (4)
Br26	0.0641 (8)	0.0600 (8)	0.0514 (8)	−0.0015 (6)	0.0090 (6)	−0.0028 (6)

Geometric parameters (Å, º)

C11—O11	1.209 (4)	C21—O21	1.219 (5)
C11—N11	1.380 (5)	C21—N21	1.388 (6)
C11—C111	1.516 (5)	C21—C211	1.521 (7)
N11—C122	1.377 (4)	N21—C222	1.382 (5)
N11—H11	0.8600	N21—H21	0.8600
C111—C112	1.384 (6)	C211—C212	1.388 (7)
C111—C116	1.391 (7)	C211—C216	1.391 (8)
C112—C113	1.425 (8)	C212—C213	1.432 (9)
C112—Br12	1.871 (6)	C212—H212	0.9300
C113—C114	1.347 (9)	C213—C214	1.343 (10)
C113—H113	0.9300	C213—H213	0.9300
C114—C115	1.342 (8)	C214—C215	1.341 (9)
C114—H114	0.9300	C214—H214	0.9300
C115—C116	1.377 (6)	C215—C216	1.380 (7)
C115—H115	0.9300	C215—H215	0.9300
C116—H116	0.9300	C216—Br26	1.870 (10)
S121—C122	1.720 (3)	S221—C222	1.721 (4)
S121—C125	1.747 (4)	S221—C225	1.748 (6)
C122—C123	1.380 (5)	C222—C223	1.378 (6)
C123—C124	1.428 (5)	C223—C224	1.430 (6)
C123—C137	1.454 (5)	C223—C237	1.455 (6)
C124—C125	1.345 (5)	C224—C225	1.346 (7)
C124—H124	0.9300	C224—H224	0.9300
C125—C126	1.500 (4)	C225—C226	1.501 (5)
C126—C127	1.504 (9)	C226—C227	1.504 (9)
C126—H16A	0.9700	C226—H26A	0.9700
C126—H16B	0.9700	C226—H26B	0.9700
C127—H17A	0.9600	C227—H27A	0.9600
C127—H17B	0.9600	C227—H27B	0.9600
C127—H17C	0.9600	C227—H27C	0.9600

C137—O137	1.231 (5)	C237—O237	1.230 (6)
C137—C131	1.502 (4)	C237—C231	1.502 (5)
C131—C132	1.382 (5)	C231—C232	1.382 (6)
C131—C136	1.394 (4)	C231—C236	1.394 (6)
C132—C133	1.398 (5)	C232—C233	1.399 (6)
C132—Cl12	1.734 (4)	C232—Cl22	1.734 (5)
C133—C134	1.360 (6)	C233—C234	1.360 (7)
C133—H133	0.9300	C233—H233	0.9300
C134—C135	1.365 (7)	C234—C235	1.365 (8)
C134—H134	0.9300	C234—H234	0.9300
C135—C136	1.379 (5)	C235—C236	1.379 (6)
C135—H135	0.9300	C235—H235	0.9300
C136—H136	0.9300	C236—H236	0.9300

O11—C11—N11	121.4 (3)	O21—C21—N21	117.9 (8)
O11—C11—C111	124.8 (4)	O21—C21—C211	121.6 (9)
N11—C11—C111	112.8 (4)	N21—C21—C211	112.6 (8)
C122—N11—C11	126.2 (4)	C222—N21—C21	122.9 (8)
C122—N11—H11	116.9	C222—N21—H21	118.6
C11—N11—H11	116.9	C21—N21—H21	118.6
C112—C111—C116	119.0 (4)	C212—C211—C216	119.4 (6)
C112—C111—C11	118.8 (5)	C212—C211—C21	116.6 (6)
C116—C111—C11	122.0 (4)	C216—C211—C21	122.6 (7)
C111—C112—C113	118.3 (5)	C211—C212—C213	118.1 (7)
C111—C112—Br12	123.0 (3)	C211—C212—H212	121.0
C113—C112—Br12	118.6 (4)	C213—C212—H212	121.0
C114—C113—C112	120.3 (5)	C214—C213—C212	119.5 (7)
C114—C113—H113	119.8	C214—C213—H213	120.2
C112—C113—H113	119.8	C212—C213—H213	120.2
C115—C114—C113	121.5 (5)	C215—C214—C213	121.7 (7)
C115—C114—H114	119.3	C215—C214—H214	119.2
C113—C114—H114	119.3	C213—C214—H214	119.2
C114—C115—C116	119.9 (5)	C214—C215—C216	120.9 (8)
C114—C115—H115	120.0	C214—C215—H215	119.6
C116—C115—H115	120.0	C216—C215—H215	119.6
C115—C116—C111	121.0 (6)	C215—C216—C211	119.6 (8)
C115—C116—H116	119.5	C215—C216—Br26	115.8 (7)
C111—C116—H116	119.5	C211—C216—Br26	124.5 (6)
C122—S121—C125	91.37 (19)	C222—S221—C225	91.2 (3)
N11—C122—C123	124.2 (4)	C223—C222—N21	125.1 (6)
N11—C122—S121	123.4 (3)	C223—C222—S221	112.1 (4)
C123—C122—S121	112.1 (3)	N21—C222—S221	122.7 (5)
C122—C123—C124	111.1 (3)	C222—C223—C224	110.8 (6)
C122—C123—C137	122.2 (4)	C222—C223—C237	122.0 (7)
C124—C123—C137	126.5 (4)	C224—C223—C237	126.1 (7)
C125—C124—C123	114.1 (3)	C225—C224—C223	113.8 (6)
C125—C124—H124	122.9	C225—C224—H224	123.1
C123—C124—H124	122.9	C223—C224—H224	123.1

C124—C125—C126	128.8 (3)	C224—C225—C226	128.5 (8)
C124—C125—S121	110.9 (3)	C224—C225—S221	110.6 (7)
C126—C125—S121	120.3 (3)	C226—C225—S221	120.2 (6)
C125—C126—C127	113.1 (5)	C225—C226—C227	113.0 (7)
C125—C126—H16A	109.0	C225—C226—H26A	109.0
C127—C126—H16A	109.0	C227—C226—H26A	109.0
C125—C126—H16B	109.0	C225—C226—H26B	109.0
C127—C126—H16B	109.0	C227—C226—H26B	109.0
H16A—C126—H16B	107.8	H26A—C226—H26B	107.8
C126—C127—H17A	109.5	C226—C227—H27A	109.5
C126—C127—H17B	109.5	C226—C227—H27B	109.5
H17A—C127—H17B	109.5	H27A—C227—H27B	109.5
C126—C127—H17C	109.5	C226—C227—H27C	109.5
H17A—C127—H17C	109.5	H27A—C227—H27C	109.5
H17B—C127—H17C	109.5	H27B—C227—H27C	109.5
O137—C137—C123	122.7 (4)	O237—C237—C223	122.5 (7)
O137—C137—C131	118.3 (3)	O237—C237—C231	118.5 (6)
C123—C137—C131	118.8 (3)	C223—C237—C231	118.7 (6)
C132—C131—C136	119.0 (4)	C232—C231—C236	119.0 (6)
C132—C131—C137	122.4 (3)	C232—C231—C237	122.5 (6)
C136—C131—C137	118.3 (3)	C236—C231—C237	118.3 (6)
C131—C132—C133	120.8 (4)	C231—C232—C233	120.7 (7)
C131—C132—Cl12	119.8 (3)	C231—C232—Cl22	119.8 (7)
C133—C132—Cl12	119.4 (3)	C233—C232—Cl22	119.1 (6)
C134—C133—C132	118.6 (4)	C234—C233—C232	118.5 (6)
C134—C133—H133	120.7	C234—C233—H233	120.8
C132—C133—H133	120.7	C232—C233—H233	120.8
C133—C134—C135	121.6 (4)	C233—C234—C235	121.6 (8)
C133—C134—H134	119.2	C233—C234—H234	118
C135—C134—H134	119.2	C235—C234—H234	121
C134—C135—C136	120.3 (4)	C234—C235—C236	120.3 (7)
C134—C135—H135	119.9	C234—C235—H235	119.8
C136—C135—H135	119.9	C236—C235—H235	119.8
C135—C136—C131	119.6 (4)	C235—C236—C231	119.5 (6)
C135—C136—H136	120.2	C235—C236—H236	120.2
C131—C136—H136	120.2	C231—C236—H236	120.2

O11—C11—N11—C122	8 (2)	O21—C21—N21—C222	−10 (6)
C111—C11—N11—C122	176.9 (18)	C211—C21—N21—C222	−159 (4)
O11—C11—C111—C112	−44.1 (13)	O21—C21—C211—C212	−12 (3)
N11—C11—C111—C112	146.9 (10)	N21—C21—C211—C212	136 (2)
O11—C11—C111—C116	141.4 (12)	O21—C21—C211—C216	154 (3)
N11—C11—C111—C116	−27.5 (10)	N21—C21—C211—C216	−58 (3)
C116—C111—C112—C113	−0.5 (7)	C216—C211—C212—C213	9.8 (18)
C11—C111—C112—C113	−175.1 (5)	C21—C211—C212—C213	176.5 (12)
C116—C111—C112—Br12	176.8 (4)	C211—C212—C213—C214	−10.7 (17)
C11—C111—C112—Br12	2.3 (6)	C212—C213—C214—C215	5 (2)
C111—C112—C113—C114	1.0 (8)	C213—C214—C215—C216	1 (3)

Br12—C112—C113—C114	−176.5 (4)	C214—C215—C216—C211	−2 (3)
C112—C113—C114—C115	−1.0 (9)	C214—C215—C216—Br26	−179.8 (15)
C113—C114—C115—C116	0.4 (8)	C212—C211—C216—C215	−4 (2)
C114—C115—C116—C111	0.1 (7)	C21—C211—C216—C215	−169.5 (15)
C112—C111—C116—C115	0.0 (7)	C212—C211—C216—Br26	173.8 (9)
C11—C111—C116—C115	174.4 (5)	C21—C211—C216—Br26	7.9 (18)
C11—N11—C122—C123	172.1 (19)	C21—N21—C222—C223	−163 (5)
C11—N11—C122—S121	−2 (3)	C21—N21—C222—S221	14 (8)
C125—S121—C122—N11	174 (2)	C225—S221—C222—C223	0 (5)
C125—S121—C122—C123	0 (2)	C225—S221—C222—N21	−178 (5)
N11—C122—C123—C124	−177.5 (19)	N21—C222—C223—C224	−175 (5)
S121—C122—C123—C124	−3 (2)	S221—C222—C223—C224	7 (5)
N11—C122—C123—C137	6 (3)	N21—C222—C223—C237	−6 (8)
S121—C122—C123—C137	−179.4 (16)	S221—C222—C223—C237	176 (4)
C122—C123—C124—C125	6 (2)	C222—C223—C224—C225	−13 (6)
C137—C123—C124—C125	−178 (2)	C237—C223—C224—C225	179 (5)
C123—C124—C125—C126	176 (2)	C223—C224—C225—C226	−177 (5)
C123—C124—C125—S121	−6 (3)	C223—C224—C225—S221	13 (6)
C122—S121—C125—C124	4 (2)	C222—S221—C225—C224	−7 (5)
C122—S121—C125—C126	−178 (2)	C222—S221—C225—C226	−178 (5)
C124—C125—C126—C127	−124 (3)	C224—C225—C226—C227	−136 (7)
S121—C125—C126—C127	59 (2)	S221—C225—C226—C227	33 (6)
C122—C123—C137—O137	−10 (3)	C222—C223—C237—O237	7 (8)
C124—C123—C137—O137	174 (2)	C224—C223—C237—O237	174 (5)
C122—C123—C137—C131	176.0 (16)	C222—C223—C237—C231	−178 (4)
C124—C123—C137—C131	0 (2)	C224—C223—C237—C231	−11 (5)
O137—C137—C131—C132	115 (2)	O237—C237—C231—C232	113 (6)
C123—C137—C131—C132	−71.2 (19)	C223—C237—C231—C232	−61 (5)
O137—C137—C131—C136	−60 (2)	O237—C237—C231—C236	−72 (6)
C123—C137—C131—C136	114 (2)	C223—C237—C231—C236	113 (5)
C136—C131—C132—C133	−3 (4)	C236—C231—C232—C233	6 (9)
C137—C131—C132—C133	−177 (2)	C237—C231—C232—C233	−179 (5)
C136—C131—C132—Cl12	177 (2)	C236—C231—C232—Cl22	178 (5)
C137—C131—C132—Cl12	3 (3)	C237—C231—C232—Cl22	−7 (6)
C131—C132—C133—C134	2 (4)	C231—C232—C233—C234	−7 (11)
Cl12—C132—C133—C134	−178 (3)	Cl22—C232—C233—C234	−179 (7)
C132—C133—C134—C135	−3 (5)	C232—C233—C234—C235	6 (13)
C133—C134—C135—C136	4 (6)	C233—C234—C235—C236	−5 (15)
C134—C135—C136—C131	−4 (6)	C234—C235—C236—C231	4 (14)
C132—C131—C136—C135	4 (4)	C232—C231—C236—C235	−5 (11)
C137—C131—C136—C135	178 (3)	C237—C231—C236—C235	−179 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O137	0.86	2.06	2.70 (2)	131
N21—H21···O237	0.86	2.12	2.71 (6)	126

C114—H114···*Cg*1^{i}	0.93	2.82	3.565 (12)	138
C214—H214···O237^{ii}	0.93	2.24	2.96 (4)	134

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

N-[3-(2-Chlorobenzoyl)-5-ethylthiophen-2-yl]-2-iodobenzamide (IV)

Crystal data

$C_{20}H_{15}ClINO_2S$
M_r = 495.74
Monoclinic, $P2_1/n$
a = 8.1813 (6) Å
b = 11.8276 (7) Å
c = 20.1347 (12) Å
β = 94.388 (2)°
V = 1942.6 (2) Å^3
Z = 4
$F(000)$ = 976
D_x = 1.695 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3860 reflections
θ = 2.6–26.1°
μ = 1.91 mm^{-1}
T = 294 K
Block, yellow
0.16 × 0.14 × 0.11 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} = 0.722, T_{max} = 0.811
20297 measured reflections
3860 independent reflections
2525 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 26.1°, θ_{min} = 2.6°
h = −10→10
k = −14→14
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.128
S = 1.02
3860 reflections
236 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 3.2004P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3131 (6)	0.3030 (4)	0.3690 (2)	0.0428 (10)
O1	0.2068 (4)	0.2861 (3)	0.40670 (16)	0.0587 (9)
N1	0.4011 (4)	0.4010 (3)	0.37099 (17)	0.0424 (9)
H1	0.4802	0.4057	0.3453	0.051*
C11	0.3569 (5)	0.2212 (4)	0.3164 (2)	0.0408 (10)
C12	0.3616 (5)	0.1046 (4)	0.3269 (2)	0.0465 (11)

I12	0.33344 (6)	0.03002 (4)	0.41855 (2)	0.0856 (2)
C13	0.3960 (7)	0.0328 (4)	0.2758 (3)	0.0624 (15)
H13	0.4012	−0.0447	0.2833	0.075*
C14	0.4224 (7)	0.0743 (6)	0.2143 (3)	0.0708 (16)
H14	0.4454	0.0246	0.1804	0.085*
C15	0.4156 (7)	0.1878 (5)	0.2019 (3)	0.0661 (15)
H15	0.4328	0.2156	0.1598	0.079*
C16	0.3825 (6)	0.2609 (4)	0.2531 (2)	0.0528 (12)
H16	0.3774	0.3382	0.2449	0.063*
S21	0.22237 (15)	0.49676 (10)	0.46480 (6)	0.0452 (3)
C22	0.3745 (5)	0.4928 (4)	0.4103 (2)	0.0391 (10)
C23	0.4582 (5)	0.5937 (4)	0.4090 (2)	0.0404 (10)
C24	0.3951 (6)	0.6743 (4)	0.4536 (2)	0.0459 (11)
H24	0.4363	0.7473	0.4589	0.055*
C25	0.2706 (6)	0.6351 (4)	0.4872 (2)	0.0450 (11)
C26	0.1747 (7)	0.6894 (5)	0.5393 (3)	0.0670 (15)
H26A	0.0592	0.6853	0.5246	0.080*
H26B	0.1913	0.6457	0.5800	0.080*
C27	0.2152 (10)	0.8057 (6)	0.5544 (4)	0.102 (2)
H27A	0.3271	0.8105	0.5724	0.153*
H27B	0.1443	0.8340	0.5864	0.153*
H27C	0.2012	0.8500	0.5144	0.153*
C37	0.5889 (6)	0.6110 (4)	0.3649 (2)	0.0508 (12)
O37	0.6360 (5)	0.5341 (3)	0.3298 (2)	0.0812 (13)
C31	0.6698 (6)	0.7239 (4)	0.3620 (2)	0.0430 (11)
C32	0.6757 (6)	0.7829 (4)	0.3023 (2)	0.0503 (12)
Cl32	0.5699 (2)	0.73426 (18)	0.23000 (7)	0.0917 (5)
C33	0.7588 (7)	0.8843 (5)	0.2998 (3)	0.0625 (15)
H33	0.7586	0.9239	0.2599	0.075*
C34	0.8403 (7)	0.9258 (5)	0.3554 (3)	0.0694 (16)
H34	0.8995	0.9926	0.3533	0.083*
C35	0.8363 (7)	0.8707 (5)	0.4145 (3)	0.0671 (15)
H35	0.8913	0.9007	0.4526	0.081*
C36	0.7518 (6)	0.7711 (4)	0.4182 (2)	0.0533 (12)
H36	0.7495	0.7345	0.4590	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.047 (3)	0.042 (2)	0.041 (2)	−0.002 (2)	0.009 (2)	−0.0023 (19)
O1	0.067 (2)	0.0520 (19)	0.063 (2)	−0.0132 (17)	0.0372 (18)	−0.0111 (16)
N1	0.044 (2)	0.037 (2)	0.049 (2)	−0.0066 (16)	0.0220 (17)	−0.0067 (16)
C11	0.033 (2)	0.047 (2)	0.043 (2)	−0.0056 (19)	0.0060 (19)	−0.005 (2)
C12	0.038 (3)	0.050 (3)	0.051 (3)	−0.008 (2)	0.003 (2)	−0.006 (2)
I12	0.1151 (4)	0.0636 (3)	0.0805 (3)	−0.0053 (2)	0.0237 (3)	0.0179 (2)
C13	0.056 (3)	0.050 (3)	0.081 (4)	−0.005 (2)	0.005 (3)	−0.024 (3)
C14	0.063 (4)	0.087 (4)	0.062 (4)	−0.006 (3)	0.008 (3)	−0.038 (3)
C15	0.062 (3)	0.091 (5)	0.046 (3)	−0.005 (3)	0.013 (3)	−0.015 (3)

C16	0.052 (3)	0.057 (3)	0.051 (3)	−0.002 (2)	0.012 (2)	−0.006 (2)
S21	0.0481 (7)	0.0445 (6)	0.0458 (6)	−0.0059 (5)	0.0213 (5)	−0.0026 (5)
C22	0.043 (3)	0.042 (2)	0.034 (2)	−0.0005 (19)	0.0133 (19)	0.0005 (18)
C23	0.042 (3)	0.039 (2)	0.041 (2)	−0.0024 (19)	0.016 (2)	−0.0030 (19)
C24	0.052 (3)	0.037 (2)	0.050 (3)	−0.005 (2)	0.019 (2)	−0.008 (2)
C25	0.049 (3)	0.045 (3)	0.044 (2)	−0.002 (2)	0.017 (2)	−0.006 (2)
C26	0.072 (4)	0.060 (3)	0.075 (4)	−0.006 (3)	0.043 (3)	−0.018 (3)
C27	0.132 (6)	0.076 (4)	0.106 (5)	−0.014 (4)	0.072 (5)	−0.032 (4)
C37	0.056 (3)	0.046 (3)	0.054 (3)	−0.008 (2)	0.027 (2)	−0.010 (2)
O37	0.094 (3)	0.056 (2)	0.103 (3)	−0.024 (2)	0.071 (3)	−0.032 (2)
C31	0.045 (3)	0.042 (2)	0.044 (3)	−0.004 (2)	0.022 (2)	−0.003 (2)
C32	0.045 (3)	0.063 (3)	0.046 (3)	0.006 (2)	0.017 (2)	0.000 (2)
Cl32	0.0881 (12)	0.1342 (15)	0.0521 (8)	0.0034 (11)	0.0011 (8)	−0.0147 (9)
C33	0.070 (4)	0.057 (3)	0.065 (3)	0.009 (3)	0.036 (3)	0.020 (3)
C34	0.077 (4)	0.043 (3)	0.093 (5)	−0.014 (3)	0.039 (3)	−0.005 (3)
C35	0.074 (4)	0.063 (3)	0.067 (4)	−0.016 (3)	0.020 (3)	−0.019 (3)
C36	0.065 (3)	0.057 (3)	0.040 (3)	−0.009 (3)	0.020 (2)	−0.001 (2)

Geometric parameters (Å, º)

C1—O1	1.214 (5)	C24—H24	0.9300
C1—N1	1.364 (5)	C25—C26	1.501 (6)
C1—C11	1.498 (6)	C26—C27	1.441 (8)
N1—C22	1.370 (5)	C26—H26A	0.9700
N1—H1	0.8600	C26—H26B	0.9700
C11—C16	1.390 (6)	C27—H27A	0.9600
C11—C12	1.395 (6)	C27—H27B	0.9600
C12—C13	1.380 (7)	C27—H27C	0.9600
C12—I12	2.073 (5)	C37—O37	1.232 (5)
C13—C14	1.363 (8)	C37—C31	1.493 (6)
C13—H13	0.9300	C31—C36	1.387 (6)
C14—C15	1.366 (9)	C31—C32	1.393 (6)
C14—H14	0.9300	C32—C33	1.381 (7)
C15—C16	1.389 (7)	C32—Cl32	1.735 (5)
C15—H15	0.9300	C33—C34	1.351 (8)
C16—H16	0.9300	C33—H33	0.9300
S21—C22	1.722 (4)	C34—C35	1.359 (8)
S21—C25	1.734 (5)	C34—H34	0.9300
C22—C23	1.377 (6)	C35—C36	1.371 (7)
C23—C24	1.433 (6)	C35—H35	0.9300
C23—C37	1.455 (6)	C36—H36	0.9300
C24—C25	1.348 (6)		
O1—C1—N1	121.5 (4)	C24—C25—S21	111.1 (3)
O1—C1—C11	123.9 (4)	C26—C25—S21	117.7 (3)
N1—C1—C11	114.6 (4)	C27—C26—C25	115.6 (5)
C1—N1—C22	125.8 (4)	C27—C26—H26A	108.4
C1—N1—H1	117.1	C25—C26—H26A	108.4

C22—N1—H1	117.1	C27—C26—H26B	108.4
C16—C11—C12	118.0 (4)	C25—C26—H26B	108.4
C16—C11—C1	119.4 (4)	H26A—C26—H26B	107.4
C12—C11—C1	122.5 (4)	C26—C27—H27A	109.5
C13—C12—C11	120.0 (5)	C26—C27—H27B	109.5
C13—C12—I12	116.5 (4)	H27A—C27—H27B	109.5
C11—C12—I12	123.5 (3)	C26—C27—H27C	109.5
C14—C13—C12	120.8 (5)	H27A—C27—H27C	109.5
C14—C13—H13	119.6	H27B—C27—H27C	109.5
C12—C13—H13	119.6	O37—C37—C23	121.5 (4)
C13—C14—C15	120.8 (5)	O37—C37—C31	118.6 (4)
C13—C14—H14	119.6	C23—C37—C31	119.9 (4)
C15—C14—H14	119.6	C36—C31—C32	117.1 (4)
C14—C15—C16	118.9 (5)	C36—C31—C37	121.0 (4)
C14—C15—H15	120.5	C32—C31—C37	121.8 (4)
C16—C15—H15	120.5	C33—C32—C31	121.0 (5)
C15—C16—C11	121.5 (5)	C33—C32—Cl32	118.2 (4)
C15—C16—H16	119.3	C31—C32—Cl32	120.6 (4)
C11—C16—H16	119.3	C34—C33—C32	119.9 (5)
C22—S21—C25	91.7 (2)	C34—C33—H33	120.0
N1—C22—C23	125.1 (4)	C32—C33—H33	120.0
N1—C22—S21	122.9 (3)	C33—C34—C35	120.5 (5)
C23—C22—S21	111.9 (3)	C33—C34—H34	119.7
C22—C23—C24	111.2 (4)	C35—C34—H34	119.7
C22—C23—C37	121.5 (4)	C34—C35—C36	120.4 (5)
C24—C23—C37	127.3 (4)	C34—C35—H35	119.8
C25—C24—C23	114.1 (4)	C36—C35—H35	119.8
C25—C24—H24	122.9	C35—C36—C31	121.1 (5)
C23—C24—H24	122.9	C35—C36—H36	119.5
C24—C25—C26	131.2 (4)	C31—C36—H36	119.5

O1—C1—N1—C22	−5.1 (7)	C37—C23—C24—C25	178.5 (5)
C11—C1—N1—C22	173.9 (4)	C23—C24—C25—C26	178.0 (5)
O1—C1—C11—C16	136.1 (5)	C23—C24—C25—S21	−0.8 (5)
N1—C1—C11—C16	−42.8 (6)	C22—S21—C25—C24	0.7 (4)
O1—C1—C11—C12	−39.1 (7)	C22—S21—C25—C26	−178.3 (4)
N1—C1—C11—C12	142.0 (4)	C24—C25—C26—C27	3.5 (10)
C16—C11—C12—C13	2.0 (7)	S21—C25—C26—C27	−177.8 (5)
C1—C11—C12—C13	177.2 (4)	C22—C23—C37—O37	−4.2 (8)
C16—C11—C12—I12	178.0 (3)	C24—C23—C37—O37	178.0 (5)
C1—C11—C12—I12	−6.7 (6)	C22—C23—C37—C31	176.3 (4)
C11—C12—C13—C14	−1.3 (8)	C24—C23—C37—C31	−1.5 (8)
I12—C12—C13—C14	−177.7 (4)	O37—C37—C31—C36	−119.0 (6)
C12—C13—C14—C15	0.0 (9)	C23—C37—C31—C36	60.6 (7)
C13—C14—C15—C16	0.5 (9)	O37—C37—C31—C32	56.7 (7)
C14—C15—C16—C11	0.2 (8)	C23—C37—C31—C32	−123.7 (5)
C12—C11—C16—C15	−1.4 (7)	C36—C31—C32—C33	−0.5 (7)
C1—C11—C16—C15	−176.9 (4)	C37—C31—C32—C33	−176.4 (4)

C1—N1—C22—C23	−176.0 (4)	C36—C31—C32—Cl32	−177.2 (4)
C1—N1—C22—S21	0.5 (6)	C37—C31—C32—Cl32	6.9 (6)
C25—S21—C22—N1	−177.4 (4)	C31—C32—C33—C34	2.1 (8)
C25—S21—C22—C23	−0.4 (4)	Cl32—C32—C33—C34	178.9 (4)
N1—C22—C23—C24	176.9 (4)	C32—C33—C34—C35	−2.4 (8)
S21—C22—C23—C24	0.0 (5)	C33—C34—C35—C36	1.1 (9)
N1—C22—C23—C37	−1.2 (7)	C34—C35—C36—C31	0.5 (8)
S21—C22—C23—C37	−178.1 (4)	C32—C31—C36—C35	−0.8 (7)
C22—C23—C24—C25	0.6 (6)	C37—C31—C36—C35	175.1 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O37	0.86	2.02	2.665 (5)	131
C33—H33···O37^{i}	0.93	2.44	3.322 (7)	159
C15—H15···*Cg*2^{ii}	0.93	2.92	3.453 (6)	118

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

N-[3-(2-Chlorobenzoyl)-5-ethylthiophen-2-yl]-2-methoxybenzamide (V)

Crystal data

$C_{21}H_{18}ClNO_3S$
M_r = 399.87
Triclinic, $P\overline{1}$
a = 7.4798 (3) Å
b = 11.4237 (5) Å
c = 12.0933 (5) Å
α = 105.814 (2)°
β = 101.959 (3)°
γ = 99.187 (3)°
V = 946.66 (7) Å^3

Z = 2
$F(000)$ = 416
D_x = 1.403 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5014 reflections
θ = 1.8–29.2°
μ = 0.33 mm^{-1}
T = 294 K
Block, colourless
0.25 × 0.20 × 0.15 mm

Data collection

Bruker Kappa APEXII diffractometer
Radiation source: fine focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.908, T_{max} = 0.951
20919 measured reflections

3959 independent reflections
2406 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 26.6°, θ_{min} = 2.2°
h = −9→9
k = −14→14
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.121
S = 1.02
3959 reflections
246 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.2991P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0867 (3)	0.3228 (2)	0.2731 (2)	0.0441 (6)
O1	0.1139 (3)	0.41653 (16)	0.24242 (15)	0.0582 (5)
N1	0.1477 (3)	0.33071 (18)	0.38926 (17)	0.0457 (5)
H1	0.1372	0.2618	0.4060	0.055*
C11	−0.0209 (3)	0.1991 (2)	0.1865 (2)	0.0449 (6)
C12	−0.0288 (3)	0.0833 (2)	0.2063 (2)	0.0481 (6)
C13	−0.1416 (4)	−0.0247 (3)	0.1210 (2)	0.0627 (8)
H13	−0.1458	−0.1013	0.1345	0.075*
C14	−0.2470 (4)	−0.0186 (3)	0.0164 (3)	0.0708 (8)
H14	−0.3245	−0.0913	−0.0400	0.085*
C15	−0.2401 (4)	0.0934 (3)	−0.0064 (2)	0.0686 (8)
H15	−0.3104	0.0965	−0.0782	0.082*
C16	−0.1279 (4)	0.2010 (3)	0.0786 (2)	0.0557 (7)
H16	−0.1236	0.2768	0.0635	0.067*
O12	0.0804 (3)	0.08329 (16)	0.31140 (16)	0.0631 (5)
C121	0.0715 (5)	−0.0305 (3)	0.3394 (3)	0.0893 (11)
H12A	−0.0553	−0.0647	0.3376	0.134*
H12B	0.1518	−0.0144	0.4175	0.134*
H12C	0.1121	−0.0891	0.2820	0.134*
S21	0.27094 (9)	0.58442 (6)	0.46466 (6)	0.0500 (2)
C22	0.2251 (3)	0.4400 (2)	0.4826 (2)	0.0414 (6)
C23	0.2691 (3)	0.4464 (2)	0.6010 (2)	0.0460 (6)
C24	0.3393 (3)	0.5733 (2)	0.6764 (2)	0.0502 (6)
H24	0.3743	0.5957	0.7591	0.060*
C25	0.3506 (3)	0.6574 (2)	0.6180 (2)	0.0484 (6)
C26	0.4251 (4)	0.7971 (2)	0.6691 (3)	0.0673 (8)
H26A	0.3860	0.8266	0.7414	0.081*
H26B	0.5611	0.8143	0.6914	0.081*
C27	0.3657 (6)	0.8705 (3)	0.5901 (3)	0.1093 (14)
H27A	0.4188	0.8513	0.5233	0.164*
H27B	0.4086	0.9581	0.6336	0.164*
H27C	0.2312	0.8497	0.5621	0.164*
C37	0.2486 (4)	0.3369 (2)	0.6398 (2)	0.0565 (7)
O37	0.2028 (4)	0.23012 (18)	0.56934 (17)	0.0930 (8)
C31	0.2781 (4)	0.3534 (2)	0.7703 (2)	0.0488 (6)
C32	0.4281 (4)	0.3240 (2)	0.8355 (2)	0.0472 (6)
Cl32	0.60371 (13)	0.27880 (9)	0.77183 (8)	0.0882 (3)
C33	0.4446 (4)	0.3309 (2)	0.9529 (2)	0.0529 (7)
H33	0.5477	0.3113	0.9958	0.064*

C34	0.3090 (4)	0.3666 (2)	1.0064 (2)	0.0590 (7)
H34	0.3188	0.3708	1.0854	0.071*
C35	0.1598 (5)	0.3958 (3)	0.9431 (3)	0.0698 (8)
H35	0.0671	0.4194	0.9792	0.084*
C36	0.1436 (4)	0.3910 (3)	0.8266 (3)	0.0655 (8)
H36	0.0419	0.4132	0.7853	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0465 (14)	0.0477 (15)	0.0463 (14)	0.0150 (12)	0.0155 (12)	0.0234 (12)
O1	0.0830 (13)	0.0478 (11)	0.0503 (10)	0.0142 (9)	0.0193 (9)	0.0250 (9)
N1	0.0554 (12)	0.0386 (12)	0.0451 (12)	0.0061 (10)	0.0084 (10)	0.0226 (9)
C11	0.0461 (14)	0.0490 (15)	0.0428 (14)	0.0119 (12)	0.0137 (11)	0.0175 (11)
C12	0.0484 (15)	0.0478 (16)	0.0499 (15)	0.0097 (12)	0.0125 (12)	0.0194 (12)
C13	0.0682 (19)	0.0495 (17)	0.0632 (18)	0.0043 (14)	0.0122 (15)	0.0154 (14)
C14	0.070 (2)	0.063 (2)	0.0643 (19)	0.0005 (15)	0.0074 (16)	0.0104 (15)
C15	0.0669 (19)	0.079 (2)	0.0498 (16)	0.0110 (16)	0.0009 (14)	0.0176 (16)
C16	0.0595 (17)	0.0605 (18)	0.0522 (16)	0.0161 (14)	0.0140 (13)	0.0253 (14)
O12	0.0825 (13)	0.0408 (11)	0.0614 (11)	0.0091 (9)	0.0015 (10)	0.0248 (9)
C121	0.133 (3)	0.0502 (19)	0.082 (2)	0.0174 (19)	0.006 (2)	0.0364 (17)
S21	0.0600 (4)	0.0435 (4)	0.0528 (4)	0.0102 (3)	0.0181 (3)	0.0240 (3)
C22	0.0394 (13)	0.0431 (14)	0.0465 (14)	0.0106 (11)	0.0105 (11)	0.0218 (11)
C23	0.0487 (14)	0.0449 (15)	0.0465 (14)	0.0104 (12)	0.0083 (11)	0.0213 (12)
C24	0.0533 (15)	0.0507 (16)	0.0464 (14)	0.0091 (12)	0.0095 (12)	0.0194 (12)
C25	0.0488 (15)	0.0436 (15)	0.0538 (15)	0.0085 (12)	0.0155 (12)	0.0168 (12)
C26	0.075 (2)	0.0486 (17)	0.077 (2)	0.0071 (15)	0.0270 (16)	0.0169 (15)
C27	0.166 (4)	0.053 (2)	0.102 (3)	0.014 (2)	0.017 (3)	0.033 (2)
C37	0.0731 (19)	0.0435 (16)	0.0501 (15)	0.0072 (14)	0.0057 (14)	0.0218 (13)
O37	0.168 (2)	0.0439 (13)	0.0499 (11)	0.0041 (13)	0.0005 (13)	0.0204 (10)
C31	0.0626 (17)	0.0388 (14)	0.0467 (14)	0.0091 (12)	0.0097 (13)	0.0212 (11)
C32	0.0539 (15)	0.0459 (15)	0.0469 (14)	0.0119 (12)	0.0154 (12)	0.0210 (11)
Cl32	0.0863 (6)	0.1248 (8)	0.0851 (6)	0.0477 (5)	0.0455 (5)	0.0531 (5)
C33	0.0552 (16)	0.0541 (16)	0.0487 (15)	0.0070 (13)	0.0060 (13)	0.0238 (12)
C34	0.084 (2)	0.0474 (16)	0.0500 (16)	0.0122 (15)	0.0233 (16)	0.0201 (13)
C35	0.092 (2)	0.0614 (19)	0.078 (2)	0.0336 (17)	0.0466 (19)	0.0299 (16)
C36	0.0693 (19)	0.0625 (19)	0.076 (2)	0.0286 (16)	0.0170 (16)	0.0339 (16)

Geometric parameters (Å, º)

C1—O1	1.226 (3)	C23—C37	1.449 (3)
C1—N1	1.357 (3)	C24—C25	1.340 (3)
C1—C11	1.491 (3)	C24—H24	0.9300
N1—C22	1.378 (3)	C25—C26	1.508 (4)
N1—H1	0.8600	C26—C27	1.481 (4)
C11—C16	1.390 (3)	C26—H26A	0.9700
C11—C12	1.401 (3)	C26—H26B	0.9700
C12—O12	1.361 (3)	C27—H27A	0.9600

C12—C13	1.383 (4)	C27—H27B	0.9600
C13—C14	1.370 (4)	C27—H27C	0.9600
C13—H13	0.9300	C37—O37	1.229 (3)
C14—C15	1.376 (4)	C37—C31	1.501 (3)
C14—H14	0.9300	C31—C32	1.376 (3)
C15—C16	1.377 (4)	C31—C36	1.387 (4)
C15—H15	0.9300	C32—C33	1.380 (3)
C16—H16	0.9300	C32—Cl32	1.733 (3)
O12—C121	1.425 (3)	C33—C34	1.369 (4)
C121—H12A	0.9600	C33—H33	0.9300
C121—H12B	0.9600	C34—C35	1.359 (4)
C121—H12C	0.9600	C34—H34	0.9300
S21—C22	1.712 (2)	C35—C36	1.373 (4)
S21—C25	1.741 (3)	C35—H35	0.9300
C22—C23	1.379 (3)	C36—H36	0.9300
C23—C24	1.431 (3)		

O1—C1—N1	120.2 (2)	C25—C24—H24	122.8
O1—C1—C11	121.7 (2)	C23—C24—H24	122.8
N1—C1—C11	118.0 (2)	C24—C25—C26	128.3 (2)
C1—N1—C22	125.3 (2)	C24—C25—S21	110.88 (19)
C1—N1—H1	117.3	C26—C25—S21	120.8 (2)
C22—N1—H1	117.3	C27—C26—C25	116.0 (3)
C16—C11—C12	117.9 (2)	C27—C26—H26A	108.3
C16—C11—C1	116.2 (2)	C25—C26—H26A	108.3
C12—C11—C1	125.9 (2)	C27—C26—H26B	108.3
O12—C12—C13	122.6 (2)	C25—C26—H26B	108.3
O12—C12—C11	116.9 (2)	H26A—C26—H26B	107.4
C13—C12—C11	120.5 (2)	C26—C27—H27A	109.5
C14—C13—C12	119.9 (3)	C26—C27—H27B	109.5
C14—C13—H13	120.1	H27A—C27—H27B	109.5
C12—C13—H13	120.1	C26—C27—H27C	109.5
C13—C14—C15	121.0 (3)	H27A—C27—H27C	109.5
C13—C14—H14	119.5	H27B—C27—H27C	109.5
C15—C14—H14	119.5	O37—C37—C23	122.3 (2)
C14—C15—C16	119.1 (3)	O37—C37—C31	118.4 (2)
C14—C15—H15	120.4	C23—C37—C31	119.2 (2)
C16—C15—H15	120.4	C32—C31—C36	117.8 (2)
C15—C16—C11	121.7 (3)	C32—C31—C37	122.4 (2)
C15—C16—H16	119.2	C36—C31—C37	119.6 (2)
C11—C16—H16	119.2	C31—C32—C33	121.4 (2)
C12—O12—C121	119.6 (2)	C31—C32—Cl32	120.44 (19)
O12—C121—H12A	109.5	C33—C32—Cl32	118.2 (2)
O12—C121—H12B	109.5	C34—C33—C32	119.8 (2)
H12A—C121—H12B	109.5	C34—C33—H33	120.1
O12—C121—H12C	109.5	C32—C33—H33	120.1
H12A—C121—H12C	109.5	C35—C34—C33	119.5 (3)
H12B—C121—H12C	109.5	C35—C34—H34	120.3

C22—S21—C25	91.63 (12)	C33—C34—H34	120.3
N1—C22—C23	124.2 (2)	C34—C35—C36	121.1 (3)
N1—C22—S21	123.58 (17)	C34—C35—H35	119.5
C23—C22—S21	112.23 (18)	C36—C35—H35	119.5
C22—C23—C24	110.8 (2)	C35—C36—C31	120.4 (3)
C22—C23—C37	123.0 (2)	C35—C36—H36	119.8
C24—C23—C37	126.2 (2)	C31—C36—H36	119.8
C25—C24—C23	114.5 (2)		

O1—C1—N1—C22	6.8 (4)	C22—C23—C24—C25	−1.0 (3)
C11—C1—N1—C22	−170.4 (2)	C37—C23—C24—C25	177.7 (2)
O1—C1—C11—C16	−15.9 (3)	C23—C24—C25—C26	−177.3 (2)
N1—C1—C11—C16	161.3 (2)	C23—C24—C25—S21	0.9 (3)
O1—C1—C11—C12	166.8 (2)	C22—S21—C25—C24	−0.4 (2)
N1—C1—C11—C12	−16.1 (4)	C22—S21—C25—C26	177.9 (2)
C16—C11—C12—O12	178.5 (2)	C24—C25—C26—C27	−160.9 (3)
C1—C11—C12—O12	−4.2 (4)	S21—C25—C26—C27	21.1 (4)
C16—C11—C12—C13	−0.6 (4)	C22—C23—C37—O37	5.2 (4)
C1—C11—C12—C13	176.7 (2)	C24—C23—C37—O37	−173.4 (3)
O12—C12—C13—C14	−179.4 (3)	C22—C23—C37—C31	−172.8 (2)
C11—C12—C13—C14	−0.3 (4)	C24—C23—C37—C31	8.6 (4)
C12—C13—C14—C15	1.3 (5)	O37—C37—C31—C32	70.3 (4)
C13—C14—C15—C16	−1.3 (5)	C23—C37—C31—C32	−111.7 (3)
C14—C15—C16—C11	0.3 (4)	O37—C37—C31—C36	−104.9 (3)
C12—C11—C16—C15	0.6 (4)	C23—C37—C31—C36	73.2 (3)
C1—C11—C16—C15	−177.0 (2)	C36—C31—C32—C33	0.3 (4)
C13—C12—O12—C121	−4.0 (4)	C37—C31—C32—C33	−174.9 (2)
C11—C12—O12—C121	176.9 (3)	C36—C31—C32—Cl32	−178.9 (2)
C1—N1—C22—C23	173.4 (2)	C37—C31—C32—Cl32	5.9 (3)
C1—N1—C22—S21	−4.5 (3)	C31—C32—C33—C34	0.6 (4)
C25—S21—C22—N1	178.0 (2)	Cl32—C32—C33—C34	179.8 (2)
C25—S21—C22—C23	−0.16 (19)	C32—C33—C34—C35	−0.5 (4)
N1—C22—C23—C24	−177.4 (2)	C33—C34—C35—C36	−0.5 (4)
S21—C22—C23—C24	0.7 (3)	C34—C35—C36—C31	1.4 (4)
N1—C22—C23—C37	3.8 (4)	C32—C31—C36—C35	−1.3 (4)
S21—C22—C23—C37	−178.1 (2)	C37—C31—C36—C35	174.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O12	0.86	1.97	2.651 (3)	135
N1—H1···O37	0.86	2.08	2.716 (3)	130
C13—H13···*Cg*2^{i}	0.93	2.90	3.782 (4)	158
C36—H36···O1^{ii}	0.93	2.48	3.306 (4)	147

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

N-[3-(2-Chlorobenzoyl)-5-ethylthiophen-2-yl]-2,6-difluorobenzamide (VI)

Crystal data

$C_{20}H_{14}ClF_2NO_2S$
$M_r = 405.83$
Monoclinic, $C2/c$
$a = 58.0164$ (10) Å
$b = 7.8002$ (2) Å
$c = 16.1365$ (3) Å
$\beta = 92.105$ (1)°
$V = 7297.5$ (3) Å^3
$Z = 16$
$F(000) = 3328$
$D_x = 1.478$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 6468 reflections
$\theta = 3.1$–$66.7°$
$\mu = 3.24$ mm^{-1}
$T = 294$ K
Block, green
0.15 × 0.15 × 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)
$T_{min} = 0.601$, $T_{max} = 0.753$
42600 measured reflections
6468 independent reflections
3979 reflections with $I > 2\sigma(I)$
$R_{int} = 0.109$
$\theta_{max} = 66.7°$, $\theta_{min} = 3.1°$
$h = -68 \rightarrow 66$
$k = -8 \rightarrow 9$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.159$
$S = 1.03$
6468 reflections
572 parameters
70 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2 + 13.9466P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ 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)
C11	0.62024 (6)	0.3614 (5)	0.6424 (2)	0.0425 (10)	
O11	0.61145 (4)	0.3990 (4)	0.70712 (17)	0.0629 (9)	
N11	0.64277 (5)	0.3177 (5)	0.63965 (18)	0.0474 (9)	
H11	0.6482	0.2985	0.5917	0.057*	
C111	0.60684 (6)	0.3554 (5)	0.5617 (2)	0.0420 (9)	
C112	0.58473 (7)	0.2861 (6)	0.5570 (2)	0.0489 (10)	
F112	0.57624 (4)	0.2166 (4)	0.62578 (16)	0.0782 (9)	
C113	0.57151 (7)	0.2797 (6)	0.4849 (3)	0.0615 (13)	
H113	0.5569	0.2302	0.4841	0.074*	
C114	0.58006 (7)	0.3469 (6)	0.4143 (3)	0.0614 (13)	

H114	0.5711	0.3443	0.3652	0.074*	
C115	0.60171 (7)	0.4184 (6)	0.4149 (3)	0.0585 (12)	
H115	0.6076	0.4648	0.3669	0.070*	
C116	0.61442 (6)	0.4194 (6)	0.4880 (3)	0.0502 (11)	
F116	0.63565 (4)	0.4904 (4)	0.48860 (16)	0.0785 (9)	
S121	0.64917 (2)	0.32607 (15)	0.80796 (6)	0.0471 (3)	
C122	0.65768 (6)	0.3016 (5)	0.7079 (2)	0.0396 (9)	
C123	0.68075 (6)	0.2583 (5)	0.7043 (2)	0.0407 (9)	
C124	0.69108 (6)	0.2443 (5)	0.7861 (2)	0.0424 (10)	
H124	0.7065	0.2159	0.7961	0.051*	
C125	0.67654 (6)	0.2759 (6)	0.8468 (2)	0.0456 (10)	
C126	0.68120 (7)	0.2728 (7)	0.9397 (2)	0.0628 (13)	
H12A	0.6829	0.3896	0.9596	0.075*	
H12B	0.6680	0.2230	0.9660	0.075*	
C127	0.70239 (8)	0.1727 (7)	0.9648 (3)	0.0711 (14)	
H12C	0.7157	0.2284	0.9438	0.107*	
H12D	0.7012	0.0589	0.9424	0.107*	
H12E	0.7038	0.1668	1.0242	0.107*	
C137	0.69234 (6)	0.2357 (6)	0.6271 (2)	0.0470 (10)	
O137	0.68288 (5)	0.2540 (5)	0.55904 (16)	0.0701 (10)	
C131	0.71751 (7)	0.1829 (6)	0.6344 (2)	0.0480 (11)	
C132	0.73497 (8)	0.2898 (6)	0.6141 (3)	0.0636 (13)	
Cl12	0.72898 (3)	0.5018 (2)	0.58879 (12)	0.1114 (6)	
C133	0.75778 (7)	0.2361 (8)	0.6161 (3)	0.0778 (16)	
H133	0.7695	0.3119	0.6030	0.093*	
C134	0.76281 (8)	0.0730 (7)	0.6371 (3)	0.0754 (15)	
H134	0.7779	0.0342	0.6355	0.090*	
C135	0.74558 (9)	−0.0378 (7)	0.6612 (3)	0.0765 (15)	
H135	0.7494	−0.1485	0.6782	0.092*	
C136	0.72347 (7)	0.0126 (7)	0.6604 (3)	0.0575 (12)	
H136	0.7121	−0.0627	0.6767	0.069*	
C21	0.63105 (7)	0.8579 (9)	0.6285 (3)	0.0422 (10)	0.916 (3)
O21	0.63979 (5)	0.9189 (5)	0.5677 (3)	0.0560 (12)	0.916 (3)
N21	0.60841 (8)	0.8128 (11)	0.6287 (3)	0.0473 (15)	0.916 (3)
H21	0.6030	0.7771	0.6745	0.057*	0.916 (3)
C211	0.64465 (7)	0.8237 (11)	0.7074 (3)	0.0404 (11)	0.916 (3)
C212	0.66623 (10)	0.7482 (11)	0.7052 (3)	0.0452 (15)	0.916 (3)
F212	0.67381 (7)	0.6941 (5)	0.6325 (2)	0.0700 (15)	0.916 (3)
C213	0.67994 (12)	0.7200 (14)	0.7750 (3)	0.052 (2)	0.916 (3)
H213	0.6943	0.6684	0.7712	0.062*	0.916 (3)
C214	0.67207 (14)	0.7691 (19)	0.8504 (3)	0.058 (2)	0.916 (3)
H214	0.6811	0.7490	0.8982	0.069*	0.916 (3)
C215	0.65095 (11)	0.848 (2)	0.8566 (3)	0.0600 (15)	0.916 (3)
H215	0.6457	0.8843	0.9075	0.072*	0.916 (3)
C216	0.63786 (8)	0.8702 (12)	0.7846 (3)	0.0513 (13)	0.916 (3)
F216	0.61691 (5)	0.9467 (8)	0.7899 (3)	0.0806 (16)	0.916 (3)
S221	0.60210 (3)	0.8752 (3)	0.46262 (13)	0.0489 (4)	0.916 (3)
C222	0.59333 (8)	0.8206 (13)	0.5594 (3)	0.0420 (11)	0.916 (3)

C223	0.57043 (7)	0.7760 (14)	0.5602 (3)	0.0444 (11)	0.916 (3)
C224	0.56012 (7)	0.7857 (9)	0.4785 (2)	0.0471 (14)	0.916 (3)
H224	0.5447	0.7589	0.4667	0.056*	0.916 (3)
C225	0.57460 (7)	0.8368 (7)	0.4200 (2)	0.0500 (13)	0.916 (3)
C226	0.57012 (8)	0.8649 (9)	0.3285 (3)	0.0736 (18)	0.916 (3)
H22A	0.5752	0.9796	0.3145	0.088*	0.916 (3)
H22B	0.5794	0.7845	0.2983	0.088*	0.916 (3)
C227	0.54616 (10)	0.8451 (12)	0.3008 (3)	0.111 (3)	0.916 (3)
H22C	0.5366	0.9150	0.3344	0.166*	0.916 (3)
H22D	0.5417	0.7272	0.3059	0.166*	0.916 (3)
H22E	0.5444	0.8801	0.2439	0.166*	0.916 (3)
C237	0.55882 (7)	0.7266 (7)	0.6341 (3)	0.0490 (11)	0.916 (3)
O237	0.56891 (13)	0.712 (3)	0.7020 (3)	0.0730 (11)	0.916 (3)
C231	0.53372 (8)	0.6783 (6)	0.6271 (3)	0.0488 (12)	0.916 (3)
C232	0.51612 (7)	0.7917 (6)	0.6099 (3)	0.0512 (13)	0.916 (3)
Cl22	0.52193 (2)	1.0065 (2)	0.59302 (11)	0.0765 (5)	0.916 (3)
C233	0.49327 (8)	0.7397 (8)	0.6065 (4)	0.0714 (18)	0.916 (3)
H233	0.4816	0.8192	0.5960	0.086*	0.916 (3)
C234	0.48803 (10)	0.5731 (9)	0.6185 (6)	0.097 (3)	0.916 (3)
H234	0.4727	0.5373	0.6149	0.116*	0.916 (3)
C235	0.50504 (11)	0.4573 (9)	0.6360 (15)	0.112 (3)	0.916 (3)
H235	0.5013	0.3432	0.6456	0.134*	0.916 (3)
C236	0.52772 (10)	0.5089 (8)	0.6395 (10)	0.085 (2)	0.916 (3)
H236	0.5393	0.4283	0.6504	0.102*	0.916 (3)
C31	0.6279 (5)	0.862 (9)	0.629 (2)	0.0422 (10)	0.084 (3)
O31	0.6340 (7)	0.957 (7)	0.574 (3)	0.0560 (12)	0.084 (3)
N31	0.6070 (6)	0.784 (13)	0.627 (3)	0.0473 (15)	0.084 (3)
H31	0.6038	0.7172	0.6670	0.057*	0.084 (3)
C311	0.6419 (5)	0.841 (11)	0.7086 (19)	0.0404 (11)	0.084 (3)
C312	0.6643 (8)	0.779 (14)	0.707 (2)	0.0452 (15)	0.084 (3)
F312	0.6739 (9)	0.757 (9)	0.634 (3)	0.0700 (15)	0.084 (3)
C313	0.6779 (11)	0.76 (2)	0.778 (3)	0.052 (2)	0.084 (3)
H313	0.6929	0.7168	0.7747	0.062*	0.084 (3)
C314	0.6691 (13)	0.80 (2)	0.853 (2)	0.058 (2)	0.084 (3)
H314	0.6785	0.7910	0.9008	0.069*	0.084 (3)
C315	0.6465 (13)	0.85 (3)	0.859 (2)	0.0600 (15)	0.084 (3)
H315	0.6402	0.8709	0.9098	0.072*	0.084 (3)
C316	0.6338 (7)	0.872 (15)	0.786 (2)	0.0600 (15)	0.084 (3)
F316	0.6110 (6)	0.910 (11)	0.791 (3)	0.0806 (16)	0.084 (3)
S321	0.5965 (4)	0.889 (4)	0.4680 (16)	0.0489 (4)	0.084 (3)
C322	0.5904 (5)	0.804 (15)	0.563 (3)	0.0420 (11)	0.084 (3)
C323	0.5673 (5)	0.772 (15)	0.570 (3)	0.0444 (11)	0.084 (3)
C324	0.5550 (4)	0.802 (11)	0.493 (2)	0.0471 (14)	0.084 (3)
H324	0.5392	0.7823	0.4859	0.056*	0.084 (3)
C325	0.5681 (5)	0.862 (7)	0.4326 (15)	0.0500 (13)	0.084 (3)
C326	0.5625 (7)	0.878 (7)	0.3409 (17)	0.0736 (18)	0.084 (3)
H32A	0.5490	0.9499	0.3328	0.088*	0.084 (3)
H32B	0.5753	0.9340	0.3148	0.088*	0.084 (3)

C327	0.5581 (13)	0.715 (9)	0.300 (3)	0.111 (3)	0.084 (3)
H32C	0.5524	0.7350	0.2438	0.166*	0.084 (3)
H32D	0.5469	0.6510	0.3291	0.166*	0.084 (3)
H32E	0.5722	0.6503	0.2983	0.166*	0.084 (3)
C337	0.5576 (4)	0.704 (4)	0.645 (2)	0.0490 (11)	0.084 (3)
O337	0.5681 (14)	0.70 (3)	0.712 (3)	0.0730 (11)	0.084 (3)
C331	0.5326 (5)	0.652 (2)	0.642 (3)	0.0488 (12)	0.084 (3)
C332	0.5145 (4)	0.766 (2)	0.643 (3)	0.0512 (13)	0.084 (3)
Cl32	0.5198 (3)	0.9818 (19)	0.6615 (13)	0.0765 (5)	0.084 (3)
C333	0.4919 (4)	0.710 (3)	0.644 (4)	0.0714 (18)	0.084 (3)
H333	0.4799	0.7891	0.6475	0.086*	0.084 (3)
C334	0.4872 (6)	0.541 (4)	0.641 (9)	0.097 (3)	0.084 (3)
H334	0.4720	0.5033	0.6376	0.116*	0.084 (3)
C335	0.5047 (8)	0.425 (3)	0.643 (18)	0.112 (3)	0.084 (3)
H335	0.5014	0.3078	0.6409	0.134*	0.084 (3)
C336	0.5272 (7)	0.480 (3)	0.648 (13)	0.085 (2)	0.084 (3)
H336	0.5390	0.4004	0.6561	0.102*	0.084 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.031 (2)	0.052 (3)	0.044 (2)	0.0019 (18)	0.0009 (17)	−0.0024 (19)
O11	0.0433 (16)	0.094 (3)	0.0514 (17)	0.0078 (16)	0.0052 (13)	−0.0090 (17)
N11	0.0327 (17)	0.073 (3)	0.0367 (16)	0.0038 (16)	0.0006 (13)	−0.0050 (16)
C111	0.033 (2)	0.046 (3)	0.046 (2)	0.0029 (17)	0.0002 (17)	−0.0033 (19)
C112	0.040 (2)	0.060 (3)	0.047 (2)	0.000 (2)	0.0017 (18)	0.003 (2)
F112	0.0516 (15)	0.115 (2)	0.0684 (17)	−0.0265 (15)	0.0043 (13)	0.0145 (16)
C113	0.037 (2)	0.080 (4)	0.067 (3)	−0.004 (2)	−0.007 (2)	−0.006 (3)
C114	0.053 (3)	0.079 (4)	0.051 (3)	0.015 (2)	−0.017 (2)	−0.004 (2)
C115	0.046 (3)	0.081 (4)	0.048 (2)	0.005 (2)	−0.002 (2)	0.005 (2)
C116	0.034 (2)	0.060 (3)	0.057 (3)	−0.0057 (19)	−0.0027 (19)	0.003 (2)
F116	0.0486 (14)	0.119 (3)	0.0671 (16)	−0.0282 (15)	−0.0029 (12)	0.0277 (16)
S121	0.0398 (5)	0.0651 (8)	0.0367 (5)	0.0007 (5)	0.0064 (4)	−0.0035 (5)
C122	0.036 (2)	0.049 (3)	0.0334 (18)	−0.0019 (18)	−0.0012 (15)	−0.0015 (17)
C123	0.035 (2)	0.053 (3)	0.0338 (18)	−0.0017 (17)	0.0026 (15)	−0.0039 (18)
C124	0.036 (2)	0.053 (3)	0.0379 (19)	0.0023 (18)	−0.0025 (16)	−0.0036 (18)
C125	0.043 (2)	0.061 (3)	0.0321 (18)	0.0013 (19)	−0.0022 (16)	0.0027 (19)
C126	0.060 (3)	0.096 (4)	0.033 (2)	0.007 (3)	0.0049 (19)	−0.002 (2)
C127	0.091 (4)	0.078 (4)	0.043 (2)	0.008 (3)	−0.013 (2)	0.002 (2)
C137	0.037 (2)	0.070 (3)	0.0337 (19)	0.0052 (19)	0.0007 (16)	−0.006 (2)
O137	0.0469 (17)	0.131 (3)	0.0322 (14)	0.0169 (18)	−0.0028 (12)	−0.0050 (17)
C131	0.040 (2)	0.071 (3)	0.0329 (19)	0.000 (2)	0.0050 (17)	−0.015 (2)
C132	0.053 (3)	0.065 (4)	0.072 (3)	0.006 (2)	0.001 (2)	−0.006 (3)
Cl12	0.0925 (11)	0.0792 (12)	0.1628 (17)	0.0073 (9)	0.0097 (10)	0.0280 (11)
C133	0.034 (3)	0.087 (5)	0.113 (4)	0.003 (2)	0.004 (3)	−0.010 (3)
C134	0.047 (3)	0.076 (4)	0.102 (4)	0.011 (3)	−0.009 (3)	−0.024 (3)
C135	0.075 (4)	0.066 (4)	0.088 (4)	0.009 (3)	−0.019 (3)	−0.008 (3)
C136	0.038 (2)	0.073 (4)	0.061 (3)	−0.001 (2)	−0.003 (2)	−0.018 (2)

C21	0.038 (2)	0.042 (3)	0.046 (2)	0.003 (2)	0.0000 (18)	0.0006 (19)
O21	0.040 (2)	0.077 (3)	0.0511 (18)	−0.0001 (18)	0.0032 (17)	0.0132 (19)
N21	0.0347 (19)	0.065 (4)	0.0423 (18)	−0.0035 (17)	−0.0001 (14)	0.009 (2)
C211	0.033 (2)	0.044 (3)	0.044 (2)	−0.003 (2)	−0.0016 (17)	−0.0009 (18)
C212	0.037 (2)	0.053 (5)	0.045 (2)	0.000 (2)	0.0047 (18)	0.002 (2)
F212	0.0557 (15)	0.100 (4)	0.0553 (15)	0.027 (2)	0.0127 (12)	0.001 (2)
C213	0.039 (3)	0.057 (7)	0.060 (3)	0.004 (2)	−0.004 (2)	0.006 (3)
C214	0.054 (3)	0.063 (7)	0.055 (3)	−0.011 (3)	−0.017 (2)	0.006 (2)
C215	0.055 (4)	0.081 (4)	0.044 (2)	−0.008 (5)	−0.001 (2)	−0.013 (2)
C216	0.037 (3)	0.061 (3)	0.056 (3)	0.003 (3)	0.003 (2)	−0.009 (2)
F216	0.050 (2)	0.126 (4)	0.0659 (17)	0.023 (3)	0.0009 (17)	−0.034 (2)
S221	0.0362 (10)	0.0709 (10)	0.0400 (6)	−0.0003 (9)	0.0058 (7)	0.0067 (6)
C222	0.039 (2)	0.052 (4)	0.0343 (19)	0.002 (2)	−0.0004 (18)	0.0022 (18)
C223	0.038 (2)	0.060 (3)	0.036 (2)	−0.001 (2)	0.0042 (18)	0.005 (2)
C224	0.036 (2)	0.072 (4)	0.033 (2)	−0.006 (3)	−0.0013 (18)	0.001 (2)
C225	0.045 (3)	0.070 (4)	0.036 (2)	0.000 (2)	−0.0013 (19)	0.000 (2)
C226	0.057 (3)	0.126 (5)	0.038 (2)	−0.016 (3)	0.003 (2)	0.010 (3)
C227	0.082 (5)	0.204 (9)	0.044 (3)	−0.018 (5)	−0.016 (3)	0.019 (4)
C237	0.039 (2)	0.068 (3)	0.039 (2)	−0.002 (2)	0.0002 (18)	0.009 (2)
O237	0.0525 (19)	0.127 (4)	0.039 (2)	−0.0064 (19)	−0.0044 (18)	0.022 (3)
C231	0.041 (2)	0.061 (3)	0.044 (3)	−0.001 (2)	0.0021 (18)	0.013 (2)
C232	0.045 (3)	0.056 (3)	0.053 (3)	−0.008 (2)	−0.001 (2)	0.002 (2)
Cl22	0.0661 (8)	0.0607 (9)	0.1017 (13)	−0.0047 (7)	−0.0091 (8)	−0.0010 (8)
C233	0.044 (3)	0.071 (4)	0.098 (5)	0.003 (3)	−0.009 (3)	0.012 (3)
C234	0.041 (3)	0.083 (5)	0.166 (9)	−0.007 (3)	0.007 (4)	0.034 (5)
C235	0.066 (4)	0.065 (5)	0.206 (9)	−0.015 (3)	0.033 (4)	0.041 (7)
C236	0.057 (3)	0.071 (4)	0.129 (7)	0.012 (3)	0.029 (3)	0.043 (5)
C31	0.038 (2)	0.042 (3)	0.046 (2)	0.003 (2)	0.0000 (18)	0.0006 (19)
O31	0.040 (2)	0.077 (3)	0.0511 (18)	−0.0001 (18)	0.0032 (17)	0.0132 (19)
N31	0.0347 (19)	0.065 (4)	0.0423 (18)	−0.0035 (17)	−0.0001 (14)	0.009 (2)
C311	0.033 (2)	0.044 (3)	0.044 (2)	−0.003 (2)	−0.0016 (17)	−0.0009 (18)
C312	0.037 (2)	0.053 (5)	0.045 (2)	0.000 (2)	0.0047 (18)	0.002 (2)
F312	0.0557 (15)	0.100 (4)	0.0553 (15)	0.027 (2)	0.0127 (12)	0.001 (2)
C313	0.039 (3)	0.057 (7)	0.060 (3)	0.004 (2)	−0.004 (2)	0.006 (3)
C314	0.054 (3)	0.063 (7)	0.055 (3)	−0.011 (3)	−0.017 (2)	0.006 (2)
C315	0.055 (4)	0.081 (4)	0.044 (2)	−0.008 (5)	−0.001 (2)	−0.013 (2)
C316	0.055 (4)	0.081 (4)	0.044 (2)	−0.008 (5)	−0.001 (2)	−0.013 (2)
F316	0.050 (2)	0.126 (4)	0.0659 (17)	0.023 (3)	0.0009 (17)	−0.034 (2)
S321	0.0362 (10)	0.0709 (10)	0.0400 (6)	−0.0003 (9)	0.0058 (7)	0.0067 (6)
C322	0.039 (2)	0.052 (4)	0.0343 (19)	0.002 (2)	−0.0004 (18)	0.0022 (18)
C323	0.038 (2)	0.060 (3)	0.036 (2)	−0.001 (2)	0.0042 (18)	0.005 (2)
C324	0.036 (2)	0.072 (4)	0.033 (2)	−0.006 (3)	−0.0013 (18)	0.001 (2)
C325	0.045 (3)	0.070 (4)	0.036 (2)	0.000 (2)	−0.0013 (19)	0.000 (2)
C326	0.057 (3)	0.126 (5)	0.038 (2)	−0.016 (3)	0.003 (2)	0.010 (3)
C327	0.082 (5)	0.204 (9)	0.044 (3)	−0.018 (5)	−0.016 (3)	0.019 (4)
C337	0.039 (2)	0.068 (3)	0.039 (2)	−0.002 (2)	0.0002 (18)	0.009 (2)
O337	0.0525 (19)	0.127 (4)	0.039 (2)	−0.0064 (19)	−0.0044 (18)	0.022 (3)
C331	0.041 (2)	0.061 (3)	0.044 (3)	−0.001 (2)	0.0021 (18)	0.013 (2)

C332	0.045 (3)	0.056 (3)	0.053 (3)	−0.008 (2)	−0.001 (2)	0.002 (2)
Cl32	0.0661 (8)	0.0607 (9)	0.1017 (13)	−0.0047 (7)	−0.0091 (8)	−0.0010 (8)
C333	0.044 (3)	0.071 (4)	0.098 (5)	0.003 (3)	−0.009 (3)	0.012 (3)
C334	0.041 (3)	0.083 (5)	0.166 (9)	−0.007 (3)	0.007 (4)	0.034 (5)
C335	0.066 (4)	0.065 (5)	0.206 (9)	−0.015 (3)	0.033 (4)	0.041 (7)
C336	0.057 (3)	0.071 (4)	0.129 (7)	0.012 (3)	0.029 (3)	0.043 (5)

Geometric parameters (Å, º)

C11—O11	1.215 (4)	C224—H224	0.9300
C11—N11	1.354 (4)	C225—C226	1.507 (6)
C11—C111	1.493 (5)	C226—C227	1.452 (7)
N11—C122	1.381 (4)	C226—H22A	0.9700
N11—H11	0.8600	C226—H22B	0.9700
C111—C116	1.376 (5)	C227—H22C	0.9600
C111—C112	1.391 (5)	C227—H22D	0.9600
C112—F112	1.345 (5)	C227—H22E	0.9600
C112—C113	1.371 (6)	C237—O237	1.228 (5)
C113—C114	1.364 (6)	C237—C231	1.505 (5)
C113—H113	0.9300	C231—C232	1.372 (6)
C114—C115	1.374 (6)	C231—C236	1.383 (7)
C114—H114	0.9300	C232—C233	1.386 (6)
C115—C116	1.369 (6)	C232—Cl22	1.732 (5)
C115—H115	0.9300	C233—C234	1.350 (8)
C116—F116	1.350 (4)	C233—H233	0.9300
S121—C122	1.716 (3)	C234—C235	1.359 (8)
S121—C125	1.730 (4)	C234—H234	0.9300
C122—C123	1.384 (5)	C235—C236	1.375 (7)
C123—C124	1.433 (5)	C235—H235	0.9300
C123—C137	1.448 (5)	C236—H236	0.9300
C124—C125	1.339 (5)	C31—O31	1.218 (7)
C124—H124	0.9300	C31—N31	1.360 (7)
C125—C126	1.513 (5)	C31—C311	1.498 (7)
C126—C127	1.499 (6)	N31—C322	1.395 (6)
C126—H12A	0.9700	N31—H31	0.8600
C126—H12B	0.9700	C311—C316	1.369 (7)
C127—H12C	0.9600	C311—C312	1.385 (7)
C127—H12D	0.9600	C312—F312	1.337 (7)
C127—H12E	0.9600	C312—C313	1.374 (7)
C137—O137	1.218 (4)	C313—C314	1.370 (8)
C137—C131	1.518 (5)	C313—H313	0.9300
C131—C132	1.361 (6)	C314—C315	1.377 (8)
C131—C136	1.432 (6)	C314—H314	0.9300
C132—C133	1.387 (6)	C315—C316	1.376 (7)
C132—Cl12	1.736 (5)	C315—H315	0.9300
C133—C134	1.346 (7)	C316—F316	1.360 (7)
C133—H133	0.9300	S321—C322	1.714 (6)
C134—C135	1.388 (7)	S321—C325	1.740 (6)

C134—H134	0.9300	C322—C323	1.374 (7)
C135—C136	1.341 (6)	C323—C324	1.431 (7)
C135—H135	0.9300	C323—C337	1.443 (7)
C136—H136	0.9300	C324—C325	1.346 (7)
C21—O21	1.217 (5)	C324—H324	0.9300
C21—N21	1.360 (5)	C325—C326	1.509 (7)
C21—C211	1.497 (5)	C326—C327	1.453 (8)
N21—C222	1.395 (5)	C326—H32A	0.9700
N21—H21	0.8600	C326—H32B	0.9700
C211—C216	1.369 (5)	C327—H32C	0.9700
C211—C212	1.385 (5)	C327—H32D	0.9600
C212—F212	1.336 (4)	C327—H32E	0.9600
C212—C213	1.373 (5)	C337—O337	1.229 (7)
C213—C214	1.370 (6)	C337—C331	1.505 (7)
C213—H213	0.9300	C331—C332	1.373 (7)
C214—C215	1.378 (7)	C331—C336	1.383 (8)
C214—H214	0.9300	C332—C333	1.386 (8)
C215—C216	1.376 (6)	C332—Cl32	1.733 (7)
C215—H215	0.9300	C333—C334	1.350 (9)
C216—F216	1.359 (5)	C333—H333	0.9300
S221—C222	1.714 (4)	C334—C335	1.360 (9)
S221—C225	1.740 (4)	C334—H334	0.9300
C222—C223	1.374 (5)	C335—C336	1.375 (8)
C223—C224	1.430 (5)	C335—H335	0.9300
C223—C237	1.443 (5)	C336—H336	0.9300
C224—C225	1.346 (6)		

O11—C11—N11	121.7 (3)	C224—C225—S221	111.2 (3)
O11—C11—C111	122.4 (3)	C226—C225—S221	119.0 (3)
N11—C11—C111	115.8 (3)	C227—C226—C225	114.5 (4)
C11—N11—C122	125.0 (3)	C227—C226—H22A	108.6
C11—N11—H11	117.5	C225—C226—H22A	108.6
C122—N11—H11	117.5	C227—C226—H22B	108.6
C116—C111—C112	114.7 (3)	C225—C226—H22B	108.6
C116—C111—C11	124.7 (3)	H22A—C226—H22B	107.6
C112—C111—C11	120.6 (3)	C226—C227—H22C	109.5
F112—C112—C113	118.4 (4)	C226—C227—H22D	109.5
F112—C112—C111	118.5 (3)	H22C—C227—H22D	109.5
C113—C112—C111	123.1 (4)	C226—C227—H22E	109.5
C114—C113—C112	119.0 (4)	H22C—C227—H22E	109.5
C114—C113—H113	120.5	H22D—C227—H22E	109.5
C112—C113—H113	120.5	O237—C237—C223	122.7 (4)
C113—C114—C115	120.7 (4)	O237—C237—C231	118.2 (4)
C113—C114—H114	119.6	C223—C237—C231	119.0 (3)
C115—C114—H114	119.6	C232—C231—C236	117.1 (4)
C116—C115—C114	118.2 (4)	C232—C231—C237	124.4 (4)
C116—C115—H115	120.9	C236—C231—C237	118.4 (4)
C114—C115—H115	120.9	C231—C232—C233	121.5 (5)

F116—C116—C115	118.1 (4)	C231—C232—Cl22	120.5 (3)
F116—C116—C111	117.6 (3)	C233—C232—Cl22	117.9 (4)
C115—C116—C111	124.3 (4)	C234—C233—C232	119.7 (5)
C122—S121—C125	91.36 (17)	C234—C233—H233	120.1
N11—C122—C123	124.5 (3)	C232—C233—H233	120.1
N11—C122—S121	123.2 (3)	C233—C234—C235	120.2 (5)
C123—C122—S121	112.3 (3)	C233—C234—H234	119.9
C122—C123—C124	110.6 (3)	C235—C234—H234	119.9
C122—C123—C137	123.2 (3)	C234—C235—C236	120.0 (6)
C124—C123—C137	126.2 (3)	C234—C235—H235	120.0
C125—C124—C123	114.0 (3)	C236—C235—H235	120.0
C125—C124—H124	123.0	C235—C236—C231	121.3 (5)
C123—C124—H124	123.0	C235—C236—H236	119.4
C124—C125—C126	128.9 (4)	C231—C236—H236	119.4
C124—C125—S121	111.8 (3)	O31—C31—N31	122.0 (9)
C126—C125—S121	119.4 (3)	O31—C31—C311	122.1 (9)
C127—C126—C125	113.0 (3)	N31—C31—C311	115.5 (8)
C127—C126—H12A	109.0	C31—N31—C322	124.4 (10)
C125—C126—H12A	109.0	C31—N31—H31	117.8
C127—C126—H12B	109.0	C322—N31—H31	117.8
C125—C126—H12B	109.0	C316—C311—C312	115.3 (6)
H12A—C126—H12B	107.8	C316—C311—C31	124.5 (8)
C126—C127—H12C	109.5	C312—C311—C31	120.1 (8)
C126—C127—H12D	109.5	F312—C312—C313	117.9 (10)
H12C—C127—H12D	109.5	F312—C312—C311	118.9 (10)
C126—C127—H12E	109.5	C313—C312—C311	122.9 (7)
H12C—C127—H12E	109.5	C314—C313—C312	118.8 (8)
H12D—C127—H12E	109.5	C314—C313—H313	120.6
O137—C137—C123	123.6 (3)	C312—C313—H313	120.6
O137—C137—C131	120.1 (3)	C313—C314—C315	121.0 (9)
C123—C137—C131	116.3 (3)	C313—C314—H314	119.5
C132—C131—C136	117.7 (4)	C315—C314—H314	119.5
C132—C131—C137	122.5 (4)	C316—C315—C314	117.4 (9)
C136—C131—C137	119.7 (4)	C316—C315—H315	121.3
C131—C132—C133	121.8 (5)	C314—C315—H315	121.3
C131—C132—Cl12	119.8 (4)	F316—C316—C311	117.3 (10)
C133—C132—Cl12	118.4 (4)	F316—C316—C315	117.8 (10)
C134—C133—C132	119.3 (5)	C311—C316—C315	124.4 (7)
C134—C133—H133	120.4	C322—S321—C325	91.1 (4)
C132—C133—H133	120.4	C323—C322—N31	124.1 (8)
C133—C134—C135	120.5 (5)	C323—C322—S321	112.9 (7)
C133—C134—H134	119.8	N31—C322—S321	122.8 (7)
C135—C134—H134	119.8	C322—C323—C324	110.3 (7)
C136—C135—C134	120.9 (5)	C322—C323—C337	123.7 (10)
C136—C135—H135	119.6	C324—C323—C337	125.8 (9)
C134—C135—H135	119.6	C325—C324—C323	114.4 (6)
C135—C136—C131	119.7 (4)	C325—C324—H324	122.8
C135—C136—H136	120.2	C323—C324—H324	122.8

C131—C136—H136	120.2	C324—C325—C326	129.4 (10)
O21—C21—N21	122.3 (4)	C324—C325—S321	111.0 (5)
O21—C21—C211	122.2 (4)	C326—C325—S321	118.6 (8)
N21—C21—C211	115.5 (3)	C327—C326—C325	114.1 (9)
C21—N21—C222	124.4 (4)	C327—C326—H32A	108.7
C21—N21—H21	117.8	C325—C326—H32A	108.7
C222—N21—H21	117.8	C327—C326—H32B	108.7
C216—C211—C212	115.3 (4)	C325—C326—H32B	108.7
C216—C211—C21	124.5 (4)	H32A—C326—H32B	107.6
C212—C211—C21	120.1 (3)	C326—C327—H32C	109.5
F212—C212—C213	118.0 (4)	C326—C327—H32D	109.5
F212—C212—C211	119.0 (3)	H32C—C327—H32D	109.5
C213—C212—C211	122.9 (4)	C326—C327—H32E	109.5
C214—C213—C212	118.8 (4)	H32C—C327—H32E	109.5
C214—C213—H213	120.6	H32D—C327—H32E	109.5
C212—C213—H213	120.6	O337—C337—C323	122.6 (12)
C213—C214—C215	121.0 (4)	O337—C337—C331	118.0 (9)
C213—C214—H214	119.5	C323—C337—C331	118.9 (9)
C215—C214—H214	119.5	C332—C331—C336	117.1 (9)
C216—C215—C214	117.4 (4)	C332—C331—C337	124.2 (9)
C216—C215—H215	121.3	C336—C331—C337	118.4 (8)
C214—C215—H215	121.3	C331—C332—C333	121.3 (8)
F216—C216—C211	117.5 (4)	C331—C332—Cl32	120.0 (8)
F216—C216—C215	118.0 (4)	C333—C332—Cl32	117.6 (8)
C211—C216—C215	124.5 (4)	C334—C333—C332	119.8 (9)
C222—S221—C225	91.04 (19)	C334—C333—H333	120.1
C223—C222—N21	124.0 (3)	C332—C333—H333	120.1
C223—C222—S221	113.0 (3)	C333—C334—C335	120.3 (9)
N21—C222—S221	122.8 (3)	C333—C334—H334	119.9
C222—C223—C224	110.5 (3)	C335—C334—H334	119.9
C222—C223—C237	123.8 (4)	C334—C335—C336	119.8 (10)
C224—C223—C237	125.8 (4)	C334—C335—H335	120.1
C225—C224—C223	114.3 (4)	C336—C335—H335	120.1
C225—C224—H224	122.9	C335—C336—C331	121.1 (11)
C223—C224—H224	122.9	C335—C336—H336	119.5
C224—C225—C226	129.8 (4)	C331—C336—H336	119.5

O11—C11—N11—C122	3.8 (7)	C222—C223—C224—C225	−0.7 (10)
C111—C11—N11—C122	−174.5 (4)	C237—C223—C224—C225	179.4 (8)
O11—C11—C111—C116	136.9 (5)	C223—C224—C225—C226	−178.9 (7)
N11—C11—C111—C116	−44.8 (6)	C223—C224—C225—S221	0.3 (8)
O11—C11—C111—C112	−41.1 (6)	C222—S221—C225—C224	0.1 (6)
N11—C11—C111—C112	137.2 (4)	C222—S221—C225—C226	179.4 (6)
C116—C111—C112—F112	178.2 (4)	C224—C225—C226—C227	4.8 (10)
C11—C111—C112—F112	−3.6 (6)	S221—C225—C226—C227	−174.4 (6)
C116—C111—C112—C113	0.8 (6)	C222—C223—C237—O237	−3.8 (16)
C11—C111—C112—C113	179.0 (4)	C224—C223—C237—O237	176.1 (14)
F112—C112—C113—C114	−178.8 (4)	C222—C223—C237—C231	−179.3 (8)

C111—C112—C113—C114	−1.3 (7)
C112—C113—C114—C115	0.7 (7)
C113—C114—C115—C116	0.3 (7)
C114—C115—C116—F116	−179.7 (4)
C114—C115—C116—C111	−0.9 (7)
C112—C111—C116—F116	179.2 (4)
C11—C111—C116—F116	1.1 (6)
C112—C111—C116—C115	0.4 (7)
C11—C111—C116—C115	−177.7 (4)
C11—N11—C122—C123	−179.4 (4)
C11—N11—C122—S121	3.0 (6)
C125—S121—C122—N11	177.2 (4)
C125—S121—C122—C123	−0.7 (3)
N11—C122—C123—C124	−177.3 (4)
S121—C122—C123—C124	0.6 (5)
N11—C122—C123—C137	4.1 (7)
S121—C122—C123—C137	−178.0 (3)
C122—C123—C124—C125	−0.2 (5)
C137—C123—C124—C125	178.4 (4)
C123—C124—C125—C126	179.8 (4)
C123—C124—C125—S121	−0.3 (5)
C122—S121—C125—C124	0.6 (4)
C122—S121—C125—C126	−179.6 (4)
C124—C125—C126—C127	−19.6 (7)
S121—C125—C126—C127	160.5 (4)
C122—C123—C137—O137	0.6 (7)
C124—C123—C137—O137	−177.7 (4)
C122—C123—C137—C131	−178.2 (4)
C124—C123—C137—C131	3.5 (6)
O137—C137—C131—C132	70.4 (6)
C123—C137—C131—C132	−110.7 (5)
O137—C137—C131—C136	−107.0 (5)
C123—C137—C131—C136	71.8 (5)
C136—C131—C132—C133	1.7 (7)
C137—C131—C132—C133	−175.8 (4)
C136—C131—C132—Cl12	−175.8 (3)
C137—C131—C132—Cl12	6.7 (6)
C131—C132—C133—C134	1.2 (8)
Cl12—C132—C133—C134	178.8 (4)
C132—C133—C134—C135	−3.7 (8)
C133—C134—C135—C136	3.1 (8)
C134—C135—C136—C131	−0.1 (7)
C132—C131—C136—C135	−2.3 (6)
C137—C131—C136—C135	175.3 (4)
O21—C21—N21—C222	4.2 (10)
C211—C21—N21—C222	−174.3 (6)
O21—C21—C211—C216	132.4 (8)
N21—C21—C211—C216	−49.1 (10)
C224—C223—C237—C231	0.7 (12)
O237—C237—C231—C232	114.4 (12)
C223—C237—C231—C232	−70.0 (7)
O237—C237—C231—C236	−65.0 (14)
C223—C237—C231—C236	110.6 (10)
C236—C231—C232—C233	1.2 (10)
C237—C231—C232—C233	−178.2 (4)
C236—C231—C232—Cl22	−179.7 (8)
C237—C231—C232—Cl22	0.8 (6)
C231—C232—C233—C234	−1.6 (9)
Cl22—C232—C233—C234	179.4 (6)
C232—C233—C234—C235	1.7 (16)
C233—C234—C235—C236	−2 (2)
C234—C235—C236—C231	1 (2)
C232—C231—C236—C235	−1.1 (15)
C237—C231—C236—C235	178.4 (10)
O31—C31—N31—C322	−1 (11)
C311—C31—N31—C322	172 (6)
O31—C31—C311—C316	122 (9)
N31—C31—C311—C316	−51 (10)
O31—C31—C311—C312	−60 (8)
N31—C31—C311—C312	127 (8)
C316—C311—C312—F312	−175 (9)
C31—C311—C312—F312	7 (11)
C316—C311—C312—C313	−2 (13)
C31—C311—C312—C313	180 (9)
F312—C312—C313—C314	173 (14)
C311—C312—C313—C314	0 (17)
C312—C313—C314—C315	4 (23)
C313—C314—C315—C316	−5 (25)
C312—C311—C316—F316	−171 (9)
C31—C311—C316—F316	7 (11)
C312—C311—C316—C315	1 (13)
C31—C311—C316—C315	179 (10)
C314—C315—C316—F316	174 (16)
C314—C315—C316—C311	2 (20)
C31—N31—C322—C323	−159 (11)
C31—N31—C322—S321	16 (11)
C325—S321—C322—C323	−6 (8)
C325—S321—C322—N31	179 (8)
N31—C322—C323—C324	−179 (9)
S321—C322—C323—C324	6 (11)
N31—C322—C323—C337	−4 (15)
S321—C322—C323—C337	−179 (9)
C322—C323—C324—C325	−3 (12)
C337—C323—C324—C325	−178 (8)
C323—C324—C325—C326	167 (7)
C323—C324—C325—S321	−1 (9)

O21—C21—C211—C212	−44.0 (9)	C322—S321—C325—C324	4 (6)
N21—C21—C211—C212	134.6 (7)	C322—S321—C325—C326	−166 (5)
C216—C211—C212—F212	177.9 (7)	C324—C325—C326—C327	−63 (7)
C21—C211—C212—F212	−5.5 (11)	S321—C325—C326—C327	105 (5)
C216—C211—C212—C213	0.6 (12)	C322—C323—C337—O337	16 (16)
C21—C211—C212—C213	177.2 (7)	C324—C323—C337—O337	−169 (16)
F212—C212—C213—C214	−177.7 (11)	C322—C323—C337—C331	−172 (8)
C211—C212—C213—C214	−0.3 (14)	C324—C323—C337—C331	2 (13)
C212—C213—C214—C215	−0.9 (18)	O337—C337—C331—C332	97 (13)
C213—C214—C215—C216	2 (2)	C323—C337—C331—C332	−75 (6)
C212—C211—C216—F216	179.3 (8)	O337—C337—C331—C336	−77 (16)
C21—C211—C216—F216	2.7 (11)	C323—C337—C331—C336	111 (11)
C212—C211—C216—C215	0.5 (12)	C336—C331—C332—C333	−4 (10)
C21—C211—C216—C215	−176.0 (9)	C337—C331—C332—C333	−177 (4)
C214—C215—C216—F216	179.5 (13)	C336—C331—C332—Cl32	164 (10)
C214—C215—C216—C211	−1.7 (17)	C337—C331—C332—Cl32	−10 (5)
C21—N21—C222—C223	−179.5 (9)	C331—C332—C333—C334	−3 (11)
C21—N21—C222—S221	4.5 (11)	Cl32—C332—C333—C334	−171 (8)
C225—S221—C222—C223	−0.5 (7)	C332—C333—C334—C335	5 (20)
C225—S221—C222—N21	175.9 (7)	C333—C334—C335—C336	0 (25)
N21—C222—C223—C224	−175.6 (8)	C334—C335—C336—C331	−7 (22)
S221—C222—C223—C224	0.7 (10)	C332—C331—C336—C335	9 (15)
N21—C222—C223—C237	4.3 (14)	C337—C331—C336—C335	−177 (11)
S221—C222—C223—C237	−179.3 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O137	0.86	2.13	2.753 (4)	129
N21—H21···O237	0.86	2.10	2.733 (11)	129
N31—H31···O337	0.86	2.22	2.76 (11)	121
N31—H31···O11	0.86	2.60	3.28 (10)	137
C114—H114···O337^{i}	0.93	2.50	3.33 (5)	150
C214—H214···O137^{ii}	0.93	2.59	3.407 (6)	146
C314—H314···O137^{ii}	0.93	2.58	3.42 (4)	150

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

Benzotriazol-1-yl 3,4-dimethoxybenzoate (VII)

Crystal data

$C_{15}H_{13}N_3O_4$
M_r = 299.28
Triclinic, $P\overline{1}$
a = 8.1296 (6) Å
b = 9.5899 (6) Å
c = 10.8824 (8) Å
α = 66.840 (4)°
β = 71.533 (4)°
γ = 71.350 (4)°
V = 721.10 (9) Å^3

Z = 2
F(000) = 312
D_x = 1.378 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3424 reflections
θ = 2.1–27.8°
μ = 0.10 mm^{-1}
T = 294 K
Block, colourless
0.15 × 0.15 × 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} = 0.921, T_{max} = 0.990
15518 measured reflections
2992 independent reflections
1667 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 26.6°, θ_{min} = 2.1°
h = −10→10
k = −12→12
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.156
S = 1.03
2992 reflections
201 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.1923P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.5541 (3)	0.6284 (3)	0.6656 (3)	0.0519 (6)
O1	0.6629 (3)	0.5557 (2)	0.5980 (2)	0.0792 (6)
O2	0.5394 (2)	0.57911 (18)	0.80862 (17)	0.0612 (5)
C11	0.4229 (3)	0.7716 (2)	0.6225 (2)	0.0435 (5)
C12	0.4248 (3)	0.8391 (2)	0.4823 (2)	0.0468 (6)
H12	0.5060	0.7907	0.4210	0.056*
C13	0.3083 (3)	0.9756 (2)	0.4352 (2)	0.0442 (5)
C14	0.1853 (3)	1.0488 (2)	0.5275 (2)	0.0465 (6)
C15	0.1828 (3)	0.9810 (3)	0.6657 (2)	0.0537 (6)
H15	0.1006	1.0284	0.7273	0.064*
C16	0.3013 (3)	0.8433 (2)	0.7129 (2)	0.0499 (6)
H16	0.2991	0.7987	0.8062	0.060*
O13	0.2994 (2)	1.05405 (19)	0.30090 (17)	0.0628 (5)
C131	0.4233 (4)	0.9901 (3)	0.2010 (3)	0.0716 (8)
H13A	0.5420	0.9758	0.2097	0.107*
H13B	0.4094	1.0596	0.1112	0.107*
H13C	0.4026	0.8915	0.2144	0.107*
O14	0.0788 (2)	1.18375 (18)	0.47077 (17)	0.0663 (5)
C141	−0.0360 (4)	1.2734 (3)	0.5554 (3)	0.0776 (9)
H14A	−0.1142	1.2127	0.6277	0.116*
H14B	−0.1051	1.3653	0.5015	0.116*
H14C	0.0337	1.3026	0.5940	0.116*

N21	0.6660 (3)	0.4470 (2)	0.8527 (2)	0.0562 (6)
N22	0.6258 (3)	0.3075 (2)	0.8981 (2)	0.0659 (6)
N23	0.7677 (3)	0.2033 (2)	0.9335 (2)	0.0630 (6)
C23A	0.8983 (3)	0.2793 (3)	0.9080 (2)	0.0485 (6)
C24	1.0717 (3)	0.2208 (3)	0.9269 (3)	0.0599 (7)
H24	1.1163	0.1147	0.9643	0.072*
C25	1.1733 (4)	0.3254 (3)	0.8883 (3)	0.0717 (8)
H25	1.2906	0.2899	0.8982	0.086*
C26	1.1057 (4)	0.4858 (3)	0.8339 (3)	0.0728 (8)
H26	1.1798	0.5533	0.8096	0.087*
C27	0.9358 (4)	0.5470 (3)	0.8151 (3)	0.0624 (7)
H27	0.8912	0.6534	0.7791	0.075*
C27A	0.8342 (3)	0.4379 (3)	0.8540 (2)	0.0481 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0484 (14)	0.0488 (13)	0.0524 (15)	−0.0053 (11)	−0.0135 (11)	−0.0127 (12)
O1	0.0779 (13)	0.0687 (12)	0.0719 (13)	0.0249 (10)	−0.0231 (10)	−0.0307 (10)
O2	0.0555 (10)	0.0566 (10)	0.0559 (11)	0.0054 (8)	−0.0191 (8)	−0.0112 (8)
C11	0.0436 (12)	0.0374 (11)	0.0466 (13)	−0.0065 (10)	−0.0107 (10)	−0.0120 (10)
C12	0.0424 (12)	0.0480 (13)	0.0492 (14)	−0.0045 (10)	−0.0069 (10)	−0.0219 (11)
C13	0.0441 (12)	0.0429 (12)	0.0451 (13)	−0.0068 (10)	−0.0103 (10)	−0.0157 (10)
C14	0.0487 (13)	0.0382 (12)	0.0499 (14)	−0.0055 (10)	−0.0126 (11)	−0.0136 (11)
C15	0.0533 (14)	0.0483 (13)	0.0520 (15)	−0.0029 (11)	−0.0020 (11)	−0.0227 (12)
C16	0.0546 (14)	0.0442 (13)	0.0442 (13)	−0.0083 (11)	−0.0072 (11)	−0.0128 (11)
O13	0.0678 (11)	0.0606 (10)	0.0471 (10)	0.0063 (8)	−0.0163 (8)	−0.0178 (8)
C131	0.0770 (19)	0.0800 (19)	0.0524 (16)	−0.0062 (15)	−0.0084 (14)	−0.0295 (14)
O14	0.0695 (11)	0.0494 (10)	0.0612 (11)	0.0145 (9)	−0.0148 (9)	−0.0205 (9)
C141	0.0727 (18)	0.0560 (16)	0.081 (2)	0.0138 (14)	−0.0045 (15)	−0.0311 (15)
N21	0.0520 (12)	0.0455 (11)	0.0615 (13)	−0.0023 (9)	−0.0222 (10)	−0.0068 (10)
N22	0.0638 (14)	0.0595 (14)	0.0705 (15)	−0.0192 (11)	−0.0243 (12)	−0.0055 (12)
N23	0.0684 (14)	0.0472 (12)	0.0688 (15)	−0.0129 (11)	−0.0283 (12)	−0.0040 (11)
C23A	0.0509 (14)	0.0456 (13)	0.0449 (13)	−0.0093 (11)	−0.0129 (11)	−0.0100 (10)
C24	0.0589 (16)	0.0525 (14)	0.0605 (16)	−0.0017 (12)	−0.0197 (13)	−0.0137 (12)
C25	0.0504 (15)	0.0762 (19)	0.084 (2)	−0.0084 (14)	−0.0193 (14)	−0.0218 (16)
C26	0.0642 (18)	0.0685 (18)	0.084 (2)	−0.0280 (15)	−0.0185 (15)	−0.0111 (16)
C27	0.0713 (18)	0.0425 (13)	0.0661 (17)	−0.0146 (13)	−0.0196 (14)	−0.0050 (12)
C27A	0.0484 (14)	0.0444 (13)	0.0458 (13)	−0.0067 (10)	−0.0113 (10)	−0.0109 (10)

Geometric parameters (Å, º)

C1—O1	1.186 (3)	O14—C141	1.422 (3)
C1—O2	1.414 (3)	C141—H14A	0.9600
C1—C11	1.455 (3)	C141—H14B	0.9600
O2—N21	1.377 (2)	C141—H14C	0.9600
C11—C16	1.378 (3)	N21—N22	1.341 (3)
C11—C12	1.401 (3)	N21—C27A	1.346 (3)

C12—C13	1.363 (3)	N22—N23	1.311 (3)
C12—H12	0.9300	N23—C23A	1.372 (3)
C13—O13	1.370 (3)	C23A—C27A	1.384 (3)
C13—C14	1.404 (3)	C23A—C24	1.390 (3)
C14—O14	1.341 (2)	C24—C25	1.358 (4)
C14—C15	1.380 (3)	C24—H24	0.9300
C15—C16	1.379 (3)	C25—C26	1.403 (4)
C15—H15	0.9300	C25—H25	0.9300
C16—H16	0.9300	C26—C27	1.364 (4)
O13—C131	1.417 (3)	C26—H26	0.9300
C131—H13A	0.9600	C27—C27A	1.392 (3)
C131—H13B	0.9600	C27—H27	0.9300
C131—H13C	0.9600		

O1—C1—O2	120.6 (2)	O14—C141—H14A	109.5
O1—C1—C11	128.8 (2)	O14—C141—H14B	109.5
O2—C1—C11	110.59 (19)	H14A—C141—H14B	109.5
N21—O2—C1	112.32 (17)	O14—C141—H14C	109.5
C16—C11—C12	119.57 (19)	H14A—C141—H14C	109.5
C16—C11—C1	122.8 (2)	H14B—C141—H14C	109.5
C12—C11—C1	117.59 (19)	N22—N21—C27A	113.22 (19)
C13—C12—C11	120.3 (2)	N22—N21—O2	119.18 (19)
C13—C12—H12	119.8	C27A—N21—O2	127.6 (2)
C11—C12—H12	119.8	N23—N22—N21	106.6 (2)
C12—C13—O13	125.7 (2)	N22—N23—C23A	108.39 (19)
C12—C13—C14	120.0 (2)	N23—C23A—C27A	109.3 (2)
O13—C13—C14	114.26 (18)	N23—C23A—C24	130.3 (2)
O14—C14—C15	125.2 (2)	C27A—C23A—C24	120.4 (2)
O14—C14—C13	115.4 (2)	C25—C24—C23A	117.1 (2)
C15—C14—C13	119.44 (19)	C25—C24—H24	121.4
C16—C15—C14	120.5 (2)	C23A—C24—H24	121.4
C16—C15—H15	119.8	C24—C25—C26	121.6 (3)
C14—C15—H15	119.8	C24—C25—H25	119.2
C11—C16—C15	120.2 (2)	C26—C25—H25	119.2
C11—C16—H16	119.9	C27—C26—C25	122.7 (3)
C15—C16—H16	119.9	C27—C26—H26	118.6
C13—O13—C131	117.62 (18)	C25—C26—H26	118.6
O13—C131—H13A	109.5	C26—C27—C27A	114.8 (2)
O13—C131—H13B	109.5	C26—C27—H27	122.6
H13A—C131—H13B	109.5	C27A—C27—H27	122.6
O13—C131—H13C	109.5	N21—C27A—C23A	102.4 (2)
H13A—C131—H13C	109.5	N21—C27A—C27	134.2 (2)
H13B—C131—H13C	109.5	C23A—C27A—C27	123.3 (2)
C14—O14—C141	118.8 (2)		

O1—C1—O2—N21	2.2 (3)	C1—O2—N21—N22	−93.7 (2)
C11—C1—O2—N21	−177.67 (19)	C1—O2—N21—C27A	84.8 (3)
O1—C1—C11—C16	178.9 (3)	C27A—N21—N22—N23	1.5 (3)

O2—C1—C11—C16	−1.2 (3)	O2—N21—N22—N23	−179.79 (19)
O1—C1—C11—C12	−2.4 (4)	N21—N22—N23—C23A	−0.8 (3)
O2—C1—C11—C12	177.50 (19)	N22—N23—C23A—C27A	−0.1 (3)
C16—C11—C12—C13	0.5 (3)	N22—N23—C23A—C24	−179.4 (2)
C1—C11—C12—C13	−178.2 (2)	N23—C23A—C24—C25	178.2 (3)
C11—C12—C13—O13	178.8 (2)	C27A—C23A—C24—C25	−0.9 (4)
C11—C12—C13—C14	−0.2 (3)	C23A—C24—C25—C26	1.0 (4)
C12—C13—C14—O14	178.9 (2)	C24—C25—C26—C27	−0.5 (5)
O13—C13—C14—O14	−0.2 (3)	C25—C26—C27—C27A	−0.1 (4)
C12—C13—C14—C15	−0.5 (3)	N22—N21—C27A—C23A	−1.5 (3)
O13—C13—C14—C15	−179.6 (2)	O2—N21—C27A—C23A	179.9 (2)
O14—C14—C15—C16	−178.5 (2)	N22—N21—C27A—C27	178.4 (3)
C13—C14—C15—C16	0.8 (4)	O2—N21—C27A—C27	−0.2 (4)
C12—C11—C16—C15	−0.3 (3)	N23—C23A—C27A—N21	1.0 (3)
C1—C11—C16—C15	178.4 (2)	C24—C23A—C27A—N21	−179.7 (2)
C14—C15—C16—C11	−0.4 (4)	N23—C23A—C27A—C27	−179.0 (2)
C12—C13—O13—C131	−1.1 (3)	C24—C23A—C27A—C27	0.4 (4)
C14—C13—O13—C131	178.0 (2)	C26—C27—C27A—N21	−179.7 (3)
C15—C14—O14—C141	6.0 (4)	C26—C27—C27A—C23A	0.2 (4)
C13—C14—O14—C141	−173.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C27—H27···O14^{i}	0.93	2.52	3.175 (3)	127

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