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Crystal structures of three substituted 3-aryl-2-phenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-ones

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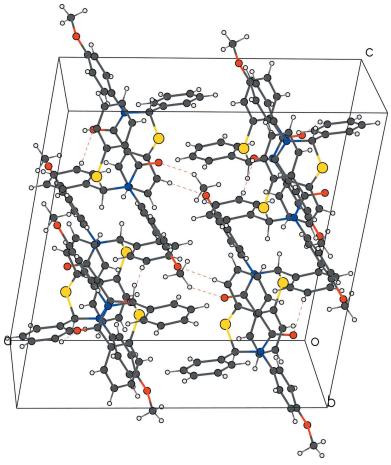
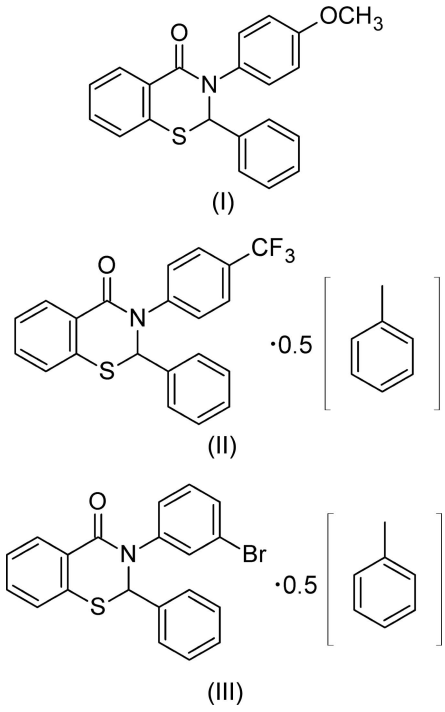
Three ring-substituted 3-aryl analogs of 2-phenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one, namely 3-(4-methoxyphenyl)-2-phenyl-4*H*-1,3-benzothiazin-4-one, $C_{21}H_{17}NO_2S$, (I), 2-phenyl-3-[4-(trifluoromethyl)phenyl]-2,3-dihydro-4*H*-1,3-benzothiazin-4-one toluene hemisolvate, $C_{21}H_{14}F_3NOS{\cdot}0.5C_7H_8$, (II), and 3-(3-bromophenyl)-2-phenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one toluene hemisolvate, $C_{20}H_{14}BrNOS{\cdot}0.5C_7H_8$, (III), were synthesized and their crystal structures determined. The hemisolvates differ in that in (II), the asymmetric unit comprises two molecules of the benzothiazinone compound and a toluene solvent molecule, whereas in (III), the unit comprises one benzothiazinone molecule and a half-occupancy toluene solvent molecule. All crystals are of racemic mixtures of the chiral 2-C atom of the thiazine moiety, which in all structures has a screw-boat puckering, with the puckering amplitude values within the range 0.575–0.603 Å. In all three structures, the benzene plane of the benzothiazine system makes a dihedral angle in the range 78.60 (5) to 98.40 (5)° with the unsubstituted benzene plane and in the range 70.50 (1) to 121.00 (5)° with the substituted benzene plane. The CF_3 substituent group in one of the molecules of (II) shows positional disorder, with an occupancy ratio of 0.57 (3):0.43 (3). In the crystals of (I) and (II), weak intermolecular C—H···O interactions are present, giving in (I), molecules arranged in a plane parallel to (010), and in (II), chains along *a*. In addition, all three structures show weak C—H···π interactions involving various aromatic rings.

1. Chemical context

We have previously reported the crystal structures of 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one (Yennawar *et al.*, 2014) and three 2-aryl-3-phenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-ones (Yennawar *et al.*, 2013, 2015). In the present communication, we report the synthesis and crystal structures of three ring-substituted 3-aryl-2-phenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-ones, namely the 4-methoxyphenyl compound, (I), the 4-(trifluoromethyl)phenyl compound as the toluene hemisolvate, (II), and the 4-bromophenyl compound as the toluene hemisolvate, (III). However, (II) and (III) differ in that the asymmetric unit of (II) comprises two independent benzothiazinone molecules and one toluene solvent molecule, while that of (III) comprises one benzothiazinone molecule and a half-occupancy toluene solvent molecule. Each compound has been synthesized using the same T3P/pyridine (T3P is 2,4,6-tripropyl-1,3,5,2,4,6-trioxatriphosphorinane 2,4,6-trioxide) method that was used for the preparation of the previously mentioned analogous compounds (Yennawar *et al.*, 2013, 2014, 2015).





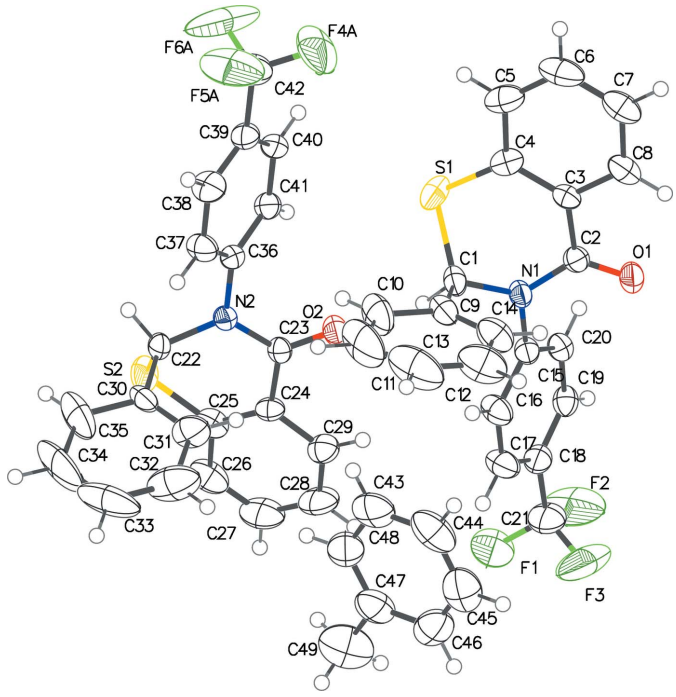


Figure 2
The molecular conformation and atom-numbering scheme for (II), with non-H atoms shown as 50% probability displacement ellipsoids. The minor component of the disordered CF_3 group is not shown.

2. Structural commentary

The three benzothiazinones (Figs. 1–3) exhibit fairly similar conformations. In all three, the thiazine ring pucker is screw-boat, with θ between 63.0 and 67.1°, and puckering amplitudes within the range 0.575–0.603 Å. The interplanar angle

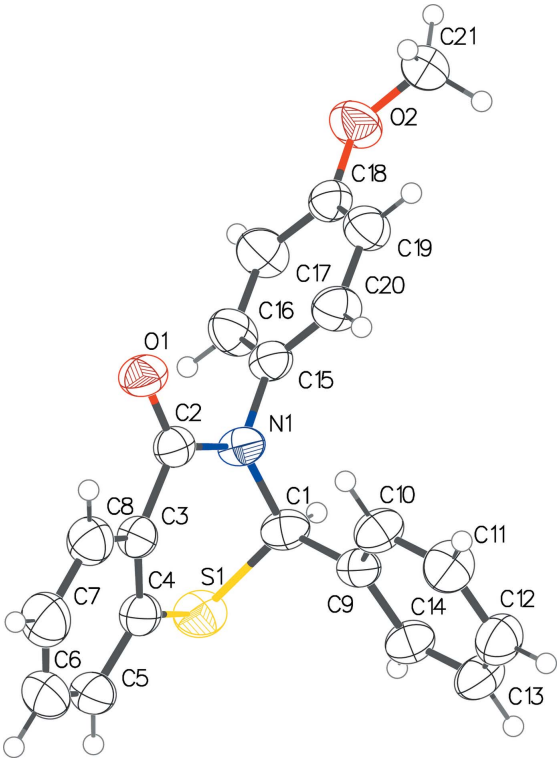


Figure 1
The molecular conformation and atom-numbering scheme for (I), with non-H atoms shown as 50% probability displacement ellipsoids.

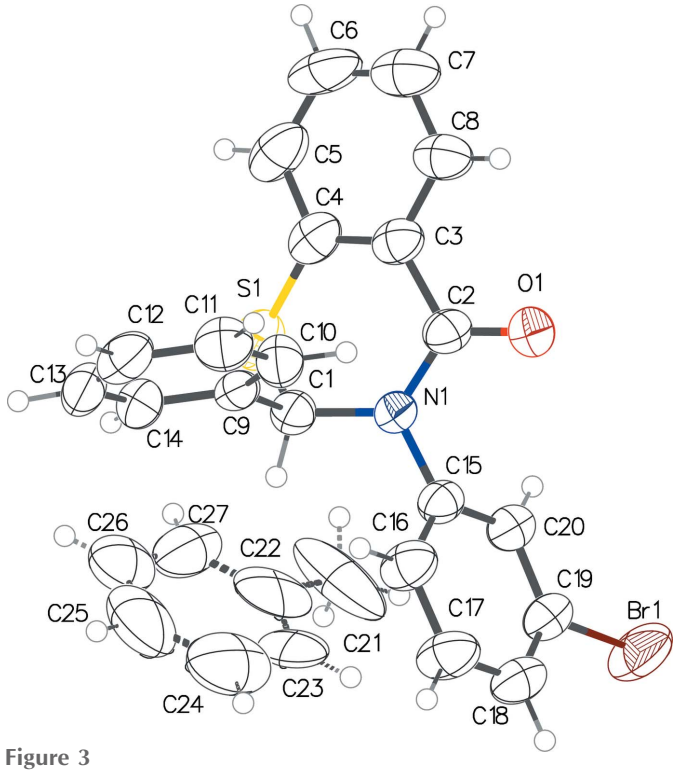


Figure 3
The molecular conformation and atom-numbering scheme for (III), with non-H atoms shown as 50% probability ellipsoids. The partial-occupancy disordered toluene solvent molecule has a site occupancy of 0.50.

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

CgX = center of gravity of ring *X*; *D*—H···*CgX* = angle of the *D*—H bond with the π plane

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1^{i}	0.93	2.59	3.447 (2)	154
C5—H5···O2^{ii}	0.93	2.46	3.387 (2)	173
C21—H21*A*···*Cg*4^{iii}	0.96	2.96	3.8068 (10)	148

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

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

CgX = center of gravity of ring *X*; *D*—H···*CgX* = angle of the *D*—H bond with the π plane

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2	0.98	2.25	3.2053 (7)	165
C22—H22···O1^{i}	0.98	2.34	3.3140 (7)	171
C17—H17···*Cg*9	0.93	2.79	3.5916 (7)	145
C38—H38···*Cg*9^{ii}	0.93	2.78	3.6009 (7)	147

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

between the benzene ring of the benzothiazine system and the substituent benzene rings at the 2-position are 82.68 (6)° in (I), 95.69 (5) and 78.10 (5)° in (II), and 98.37 (1)° in (III). Those with the benzene rings at the 3-position are 59.10 (6)° in (I), 70.56 (5) and 72.26 (5)° in (II), and 78.66 (1)° in (III). The CF_3 substituent group in one of the molecules of (II) shows positional disorder, with an occupancy ratio of 0.57 (3):0.43 (3).

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

CgX = center of gravity of ring *X*; *D*—H···*CgX* = angle of the *D*—H bond with the π plane

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···*Cg*5	0.98	2.66	3.5802 (6)	156
C6—H6···*Cg*3^{i}	0.93	2.83	3.6823 (6)	153

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

3. Supramolecular features

In (I) and (II), weak intermolecular C—H···O interactions are observed (Tables 1 and 2, respectively), giving in (I), molecules arranged in a plane parallel to (010) (Fig. 4), and in (II), chains along the *a*-axis direction (Fig. 5). The crystals also feature T-type C—H···π interactions (Tables 1–3), as

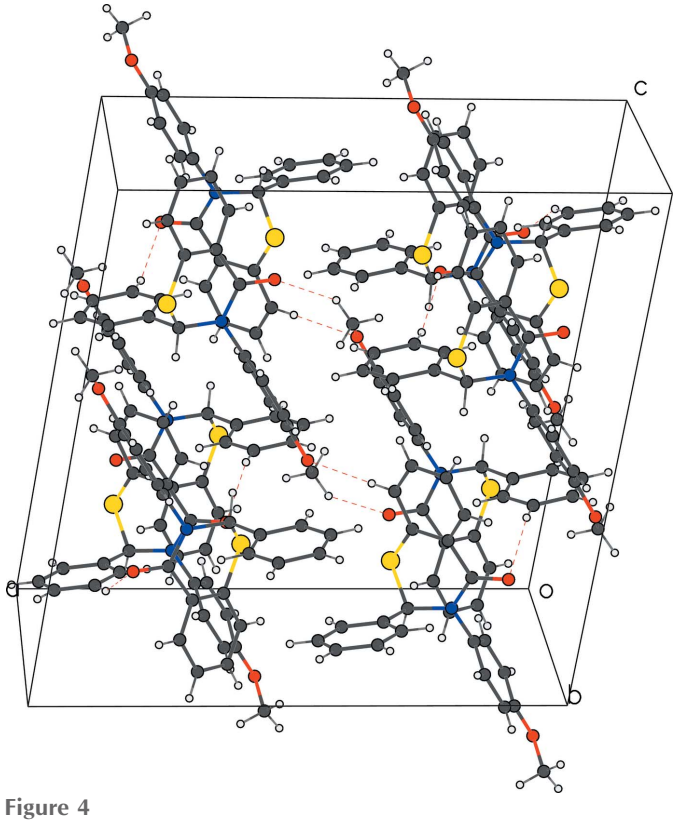


Figure 4
The crystal packing of (I) in the unit cell, viewed along *b*, showing C—H···O hydrogen bonds as dashed lines.

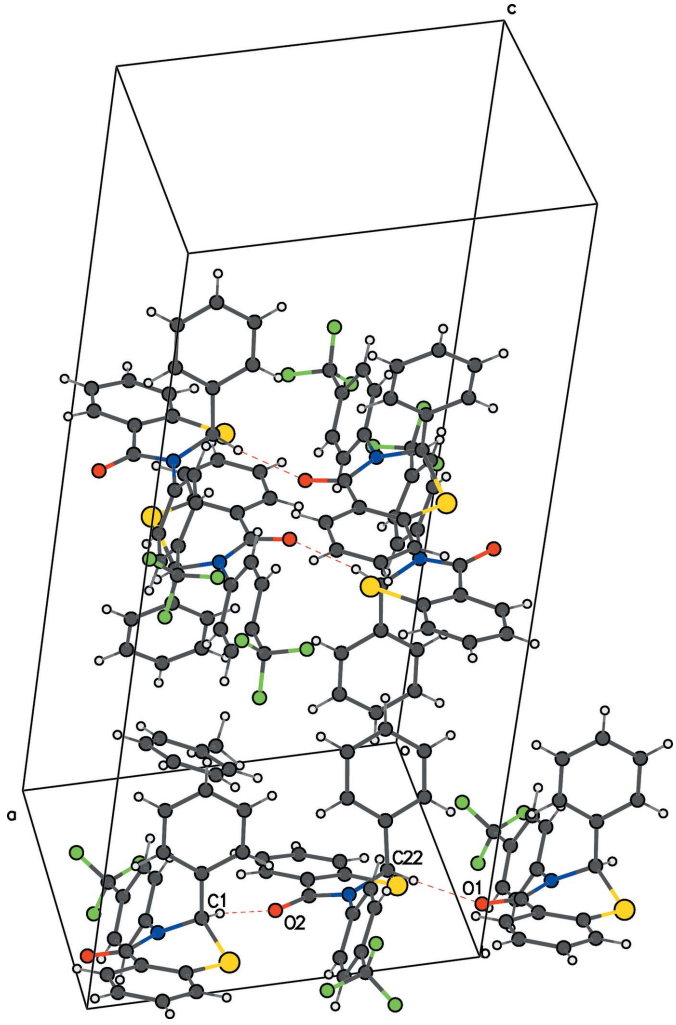


Figure 5
The crystal packing of (II) in the unit cell, viewed along *b*, showing C—H···O hydrogen bonds as dashed lines.

analyzed using *PLATON* (Spek, 2009). In (I), a weak C—H···*Cg*(π ring) interaction of 3.8068 (10) Å is present with an interacting angle of 148°. In (II), the toluene molecule participates in tilted-T-type interactions by placing itself obliquely between phenyl rings of the two enantiomers, with C—H···*Cg*(toluene) distances of 3.5916 (7) and 3.6009 (7) Å, with interacting angles of 145 and 147°, respectively. In (III), two C—H···π interactions, one between the thiazine ring and the toluene solvent molecule and the other between the fused benzene ring and the 2-phenyl ring, have C—H···*Cg* distances of 3.5802 (6) and 3.6823 (6) Å, with interacting angles of 156 and 153°, respectively (Fig. 6). Structure (I) also shows a very weak parallel-displaced π–π interaction between symmetry-related benzene rings, with an inter-centroid (*Cg*···*Cg*) distance of 3.977 (1) Å and an interplanar angle of 8°.

4. Database survey

The three structures reported here and four previously reported analogous structures (Yennawar *et al.*, 2013, 2014, 2015) have very similar screw-boat puckering for the thiazine ring. Among the seven crystal structures, the variation in the interplanar angles between the benzene ring of the benzothiazine moiety and the two substituent benzene rings at positions 2 and 3 lie within 26 and 30°, respectively. A structure for 2-(5-methylthiophen-2-yl)-3-phenyl-2,3-dihydroquinazolin-4(1*H*)-one has been reported in a patent application (Atwood *et al.*, 2015).

5. Synthesis and crystallization

A two-necked 25 ml round-bottomed flask was oven-dried, cooled under N_2 and charged with a stir bar and an *N*-aryl-*C*-phenylimine (6 mmol). Tetrahydrofuran or 2-methyltetrahydrofuran (2.3 ml) was added, the solid dissolved and the solution stirred. Pyridine (1.95 ml, 24 mmol) was added, followed by thiosalicylic acid (0.93 g, 6 mmol). Finally, 2,4,6-tripropyl-1,3,5,2,4,6-trioxatriphosphorinane 2,4,6-trioxide (T3P) in 2-methyltetrahydrofuran (50 wt%, 7.3 ml, 12 mmol) was added. The mixture was stirred at room temperature and the reaction was followed using thin-layer chromatography. The mixture was then poured into a separatory funnel and dichloromethane and distilled water were added. The layers were separated and the aqueous layer was then extracted twice with dichloromethane. The organics were combined and washed with saturated sodium bicarbonate and then saturated sodium chloride. The organic extract was dried over sodium sulfate and concentrated under vacuum. The crude product was chromatographed on 30 g of flash silica gel using mixtures of ethyl acetate and hexanes, and then further purified as indicated below.

Compound (I) was recrystallized from ethanol solution to give yellow crystals (yield 0.72 g, 34.6%; m.p. 365–369 K). R_F = 0.52 (50% ethyl acetate/hexanes). Colorless block-shaped crystals suitable for the X-ray analysis were grown by slow evaporation from ethanol solution.

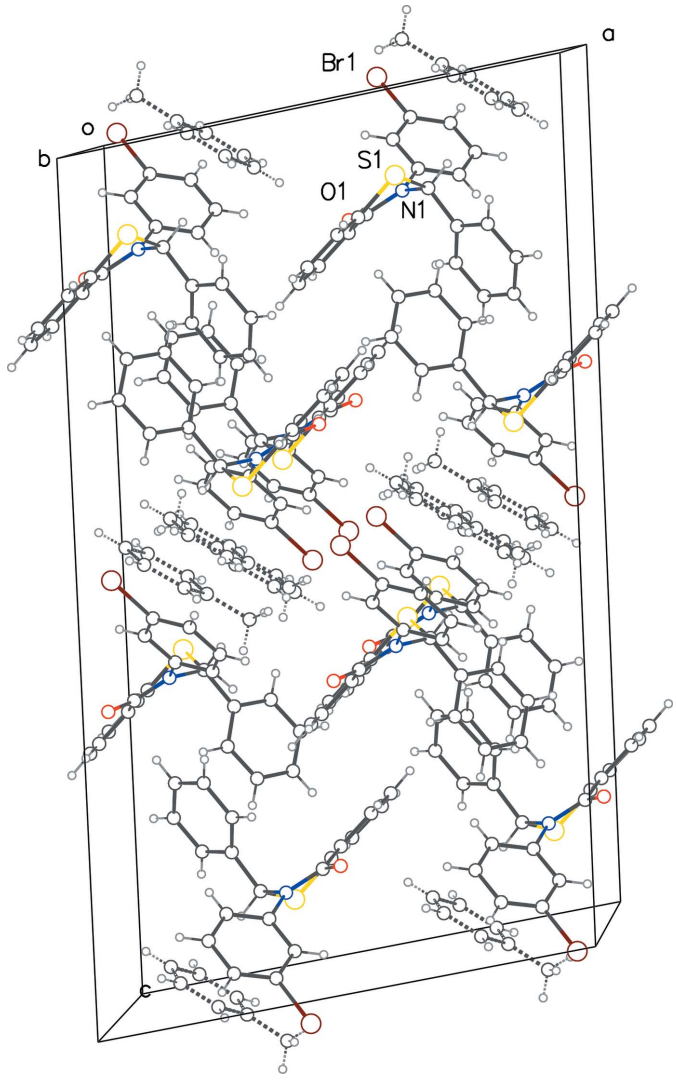


Figure 6
A perspective view of the crystal packing of (III), with the half-occupancy toluene solvent molecules shown as dashed bonds.

Compound (II) was recrystallized from methylene chloride/hexanes to give yellow crystals (yield 0.5639 g, 24.4%; m.p. 404–406 K). R_F = 0.56 (30% ethyl acetate/hexanes solution). Colorless needle-shaped crystals suitable for the X-ray analysis were grown by slow evaporation from toluene solution.

Compound (III) was triturated with hexanes solution to give a solid (0.7242 g) and then recrystallized from toluene/hexanes to give white crystals (yield 0.3544 g, 14.5%; m.p.: 358–359 K). R_F = 0.39 (20% ethyl acetate/hexanes). A second crop of 0.30 g (12.7%) was obtained by slow evaporation of the mother liquor, giving colorless blocks suitable for the X-ray analysis.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. In the refinement of (II), the two

Table 4
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{21}H_{17}NO_2S$	$2C_{21}H_{14}F_3NOS{\cdot}C_7H_8$	$2C_{20}H_{14}BrNOS{\cdot}C_7H_8$
M_r	347.42	862.92	884.72
Crystal system, space group	Monoclinic, $C2/c$	Monoclinic, $P2_1/c$	Monoclinic, $C2/c$
Temperature (K)	298	298	298
a, b, c (Å)	17.820 (4), 11.016 (3), 17.890 (4)	11.953 (2), 14.516 (3), 24.546 (5)	15.736 (2), 9.3530 (15), 27.259 (4)
β (°)	98.385 (5)	101.024 (4)	99.560 (3)
V (Å^3)	3474.4 (15)	4180.5 (14)	3956.2 (10)
Z	8	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.20	0.20	2.20
Crystal size (mm)	0.26 × 0.24 × 0.12	0.29 × 0.09 × 0.07	0.21 × 0.17 × 0.10
Data collection			
Diffractometer	Bruker CCD area-detector	Bruker CCD area-detector	Bruker CCD area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2001)	Multi-scan (*SADABS*; Bruker, 2001)	Multi-scan (*SADABS*; Bruker, 2001)
T_{min}, T_{max}	0.940, 0.986	0.592, 0.920	0.103, 0.901
No. of measured, independent and observed reflections	15005, 4284, 3414 [$I > 2\sigma(I)$]	39335, 10359, 8329 [$I > 2\sigma(I)$]	18289, 4903, 2424 [$I > 2\sigma(I)$]
R_{int}	0.024	0.052	0.064
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.666	0.668	0.667
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.125, 1.05	0.133, 0.240, 1.31	0.049, 0.153, 0.79
No. of reflections	4284	10359	4903
No. of parameters	227	580	266
No. of restraints	0	74	186
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.22, −0.37	0.48, −0.34	0.73, −0.71

Computer programs: *SMART* and *SAINT* (Bruker, 2001), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

molecules in the asymmetric unit were restrained using the SAME command in *SHELXL2014* (Sheldrick, 2015). One of the molecules shows positional disorder in the –CF_3 group, with the occupancy ratio refining to 0.57 (3):0.43 (3). We tried to address the high R values (relative to R_{int}) by looking for twinning and using restraints but we have had no success in achieving respectable R values. In (III), the disordered partial toluene molecule was refined with a site-occupancy factor determined as 0.50 and with positional constraints (AFIX 6). In all three compounds, the H atoms were placed geometrically and allowed to ride on the C atoms during refinement, with C—H distances of 0.98 (methine), 0.96 (methyl) or 0.93 Å (aromatic) and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) otherwise.

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supporting information

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Crystal structures of three substituted 3-aryl-2-phenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-ones

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Computing details

For all compounds, data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008). Program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015) for (I); *SHELXL97* (Sheldrick, 2008) for (II); *SHELXL014* (Sheldrick, 2015) for (III). For all compounds, molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

(I) 3-(4-Methoxyphenyl)-2-phenyl-4*H*-1,3-benzothiazin-4-one

Crystal data

$C_{21}H_{17}NO_2S$
M_r = 347.42
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 17.820 (4) Å
b = 11.016 (3) Å
c = 17.890 (4) Å
β = 98.385 (5)°
V = 3474.4 (15) Å^3
Z = 8
F(000) = 1456
D_x = 1.328 Mg m^{-3}
Melting point = 365–369 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4847 reflections
θ = 2.3–28.3°
μ = 0.20 mm^{-1}
T = 298 K
Block, colorless
0.26 × 0.24 × 0.12 mm

Data collection

Bruker CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
T_{min} = 0.940, T_{max} = 0.986
15005 measured reflections
4284 independent reflections
3414 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.3°, θ_{min} = 2.2°
h = −20→23
k = −14→14
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.125
S = 1.05
4284 reflections
227 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.7957P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Special details

Experimental. Absorption correction: SADABS (Bruker, 2001) was used for absorption correction. R(int) was 0.0415 before and 0.0233 after correction. The $\lambda/2$ correction factor is 0.0015.

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.17986 (7)	0.37162 (13)	0.33545 (8)	0.0429 (3)
H1	0.1812	0.3332	0.3850	0.052*
C2	0.28205 (7)	0.42680 (12)	0.26124 (7)	0.0387 (3)
C3	0.22572 (8)	0.41320 (12)	0.19132 (8)	0.0411 (3)
C4	0.16382 (8)	0.33411 (13)	0.18637 (8)	0.0453 (3)
C5	0.11974 (9)	0.31295 (16)	0.11634 (10)	0.0604 (4)
H5	0.0793	0.2588	0.1126	0.072*
C6	0.13587 (11)	0.3717 (2)	0.05331 (10)	0.0709 (5)
H6	0.1068	0.3565	0.0068	0.085*
C7	0.19525 (11)	0.4538 (2)	0.05825 (10)	0.0689 (5)
H7	0.2047	0.4957	0.0155	0.083*
C8	0.24026 (9)	0.47316 (15)	0.12662 (8)	0.0529 (4)
H8	0.2808	0.5270	0.1295	0.063*
C9	0.12871 (7)	0.48211 (12)	0.33619 (7)	0.0395 (3)
C10	0.15308 (9)	0.59940 (14)	0.32618 (9)	0.0491 (3)
H10	0.2023	0.6131	0.3168	0.059*
C11	0.10443 (10)	0.69663 (15)	0.33011 (10)	0.0571 (4)
H11	0.1213	0.7752	0.3232	0.068*
C12	0.03146 (10)	0.67779 (16)	0.34414 (10)	0.0578 (4)
H12	−0.0010	0.7433	0.3469	0.069*
C13	0.00683 (9)	0.56148 (16)	0.35406 (10)	0.0562 (4)
H13	−0.0425	0.5484	0.3635	0.067*
C14	0.05456 (8)	0.46423 (15)	0.35005 (8)	0.0484 (3)
H14	0.0372	0.3859	0.3567	0.058*
C15	0.31352 (7)	0.39619 (13)	0.39612 (7)	0.0405 (3)
C16	0.34533 (10)	0.28627 (14)	0.42046 (9)	0.0530 (4)
H16	0.3329	0.2165	0.3921	0.064*
C17	0.39550 (10)	0.27956 (15)	0.48685 (9)	0.0568 (4)
H17	0.4167	0.2053	0.5031	0.068*
C18	0.41438 (8)	0.38303 (14)	0.52936 (7)	0.0441 (3)

C19	0.38413 (9)	0.49301 (14)	0.50441 (9)	0.0499 (4)
H19	0.3975	0.5632	0.5320	0.060*
C20	0.33346 (8)	0.49867 (13)	0.43761 (9)	0.0492 (4)
H20	0.3128	0.5731	0.4209	0.059*
C21	0.48531 (10)	0.46879 (18)	0.64109 (9)	0.0625 (5)
H21A	0.5066	0.5295	0.6119	0.094*
H21B	0.5224	0.4448	0.6829	0.094*
H21C	0.4416	0.5014	0.6598	0.094*
N1	0.25868 (6)	0.39985 (11)	0.32831 (6)	0.0424 (3)
O1	0.34743 (5)	0.45779 (10)	0.25787 (6)	0.0511 (3)
O2	0.46342 (7)	0.36572 (11)	0.59465 (6)	0.0601 (3)
S1	0.14215 (2)	0.25763 (3)	0.26647 (3)	0.05493 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0365 (7)	0.0455 (7)	0.0465 (7)	−0.0048 (6)	0.0049 (6)	0.0087 (6)
C2	0.0370 (7)	0.0353 (6)	0.0438 (7)	0.0003 (5)	0.0057 (5)	−0.0008 (5)
C3	0.0392 (7)	0.0404 (7)	0.0431 (7)	0.0045 (6)	0.0043 (5)	−0.0041 (5)
C4	0.0393 (7)	0.0398 (7)	0.0558 (8)	0.0048 (6)	0.0040 (6)	−0.0100 (6)
C5	0.0439 (8)	0.0628 (10)	0.0712 (11)	0.0028 (8)	−0.0025 (7)	−0.0260 (9)
C6	0.0614 (11)	0.0961 (15)	0.0507 (9)	0.0139 (10)	−0.0065 (8)	−0.0204 (9)
C7	0.0690 (12)	0.0926 (14)	0.0445 (8)	0.0150 (11)	0.0065 (8)	0.0023 (9)
C8	0.0531 (9)	0.0593 (9)	0.0465 (8)	0.0037 (7)	0.0083 (7)	0.0012 (7)
C9	0.0349 (6)	0.0451 (7)	0.0379 (6)	−0.0037 (6)	0.0031 (5)	0.0028 (5)
C10	0.0403 (7)	0.0490 (8)	0.0594 (8)	−0.0051 (6)	0.0116 (6)	0.0050 (7)
C11	0.0588 (10)	0.0440 (8)	0.0702 (10)	−0.0028 (7)	0.0151 (8)	0.0016 (7)
C12	0.0535 (9)	0.0549 (9)	0.0662 (10)	0.0077 (8)	0.0126 (8)	−0.0030 (8)
C13	0.0381 (8)	0.0645 (10)	0.0678 (10)	−0.0003 (7)	0.0135 (7)	0.0004 (8)
C14	0.0399 (7)	0.0506 (8)	0.0556 (8)	−0.0065 (6)	0.0097 (6)	0.0043 (6)
C15	0.0350 (7)	0.0462 (7)	0.0398 (6)	0.0015 (6)	0.0040 (5)	0.0035 (5)
C16	0.0606 (10)	0.0422 (8)	0.0521 (8)	0.0055 (7)	−0.0059 (7)	−0.0043 (6)
C17	0.0659 (10)	0.0439 (8)	0.0553 (9)	0.0126 (7)	−0.0089 (8)	0.0021 (7)
C18	0.0383 (7)	0.0537 (8)	0.0397 (6)	0.0043 (6)	0.0039 (5)	0.0015 (6)
C19	0.0502 (8)	0.0452 (8)	0.0521 (8)	0.0014 (6)	−0.0001 (7)	−0.0062 (6)
C20	0.0476 (8)	0.0415 (8)	0.0558 (8)	0.0054 (6)	−0.0015 (7)	0.0037 (6)
C21	0.0509 (9)	0.0859 (13)	0.0483 (8)	0.0029 (9)	−0.0013 (7)	−0.0147 (8)
N1	0.0325 (6)	0.0506 (7)	0.0432 (6)	−0.0013 (5)	0.0028 (4)	0.0065 (5)
O1	0.0384 (5)	0.0636 (7)	0.0520 (6)	−0.0094 (5)	0.0083 (4)	−0.0003 (5)
O2	0.0631 (7)	0.0654 (7)	0.0461 (6)	0.0108 (6)	−0.0111 (5)	−0.0018 (5)
S1	0.0508 (2)	0.0384 (2)	0.0756 (3)	−0.00997 (16)	0.00913 (19)	−0.00040 (17)

Geometric parameters (Å, º)

C1—N1	1.4622 (17)	C11—H11	0.9300
C1—C9	1.522 (2)	C12—C13	1.374 (2)
C1—S1	1.8192 (15)	C12—H12	0.9300
C1—H1	0.9800	C13—C14	1.376 (2)

C2—O1	1.2240 (16)	C13—H13	0.9300
C2—N1	1.3591 (18)	C14—H14	0.9300
C2—C3	1.4929 (18)	C15—C20	1.369 (2)
C3—C8	1.389 (2)	C15—C16	1.380 (2)
C3—C4	1.398 (2)	C15—N1	1.4432 (17)
C4—C5	1.398 (2)	C16—C17	1.380 (2)
C4—S1	1.7533 (16)	C16—H16	0.9300
C5—C6	1.367 (3)	C17—C18	1.384 (2)
C5—H5	0.9300	C17—H17	0.9300
C6—C7	1.385 (3)	C18—O2	1.3661 (17)
C6—H6	0.9300	C18—C19	1.374 (2)
C7—C8	1.378 (2)	C19—C20	1.390 (2)
C7—H7	0.9300	C19—H19	0.9300
C8—H8	0.9300	C20—H20	0.9300
C9—C10	1.383 (2)	C21—O2	1.427 (2)
C9—C14	1.3935 (19)	C21—H21A	0.9600
C10—C11	1.386 (2)	C21—H21B	0.9600
C10—H10	0.9300	C21—H21C	0.9600
C11—C12	1.376 (2)		

N1—C1—C9	114.51 (11)	C13—C12—H12	120.2
N1—C1—S1	110.61 (10)	C11—C12—H12	120.2
C9—C1—S1	113.29 (9)	C12—C13—C14	120.51 (15)
N1—C1—H1	105.9	C12—C13—H13	119.7
C9—C1—H1	105.9	C14—C13—H13	119.7
S1—C1—H1	105.9	C13—C14—C9	120.56 (15)
O1—C2—N1	121.40 (12)	C13—C14—H14	119.7
O1—C2—C3	121.02 (12)	C9—C14—H14	119.7
N1—C2—C3	117.56 (12)	C20—C15—C16	119.35 (13)
C8—C3—C4	118.99 (13)	C20—C15—N1	121.54 (12)
C8—C3—C2	117.78 (13)	C16—C15—N1	119.10 (12)
C4—C3—C2	122.92 (13)	C15—C16—C17	120.20 (14)
C5—C4—C3	119.71 (15)	C15—C16—H16	119.9
C5—C4—S1	119.37 (13)	C17—C16—H16	119.9
C3—C4—S1	120.89 (11)	C16—C17—C18	120.24 (14)
C6—C5—C4	120.15 (17)	C16—C17—H17	119.9
C6—C5—H5	119.9	C18—C17—H17	119.9
C4—C5—H5	119.9	O2—C18—C19	124.93 (14)
C5—C6—C7	120.48 (16)	O2—C18—C17	115.38 (13)
C5—C6—H6	119.8	C19—C18—C17	119.68 (13)
C7—C6—H6	119.8	C18—C19—C20	119.59 (14)
C8—C7—C6	119.88 (17)	C18—C19—H19	120.2
C8—C7—H7	120.1	C20—C19—H19	120.2
C6—C7—H7	120.1	C15—C20—C19	120.91 (14)
C7—C8—C3	120.71 (16)	C15—C20—H20	119.5
C7—C8—H8	119.6	C19—C20—H20	119.5
C3—C8—H8	119.6	O2—C21—H21A	109.5
C10—C9—C14	118.62 (14)	O2—C21—H21B	109.5

C10—C9—C1	123.11 (12)	H21A—C21—H21B	109.5
C14—C9—C1	118.24 (13)	O2—C21—H21C	109.5
C9—C10—C11	120.31 (14)	H21A—C21—H21C	109.5
C9—C10—H10	119.8	H21B—C21—H21C	109.5
C11—C10—H10	119.8	C2—N1—C15	119.41 (11)
C12—C11—C10	120.50 (15)	C2—N1—C1	123.07 (11)
C12—C11—H11	119.8	C15—N1—C1	117.51 (10)
C10—C11—H11	119.8	C18—O2—C21	118.24 (13)
C13—C12—C11	119.50 (16)	C4—S1—C1	96.84 (7)

O1—C2—C3—C8	−20.9 (2)	N1—C15—C16—C17	−177.27 (15)
N1—C2—C3—C8	161.10 (13)	C15—C16—C17—C18	−0.1 (3)
O1—C2—C3—C4	152.55 (14)	C16—C17—C18—O2	178.86 (16)
N1—C2—C3—C4	−25.42 (19)	C16—C17—C18—C19	−1.3 (3)
C8—C3—C4—C5	2.6 (2)	O2—C18—C19—C20	−178.67 (14)
C2—C3—C4—C5	−170.80 (13)	C17—C18—C19—C20	1.5 (2)
C8—C3—C4—S1	−179.16 (11)	C16—C15—C20—C19	−1.2 (2)
C2—C3—C4—S1	7.43 (18)	N1—C15—C20—C19	177.45 (13)
C3—C4—C5—C6	−1.7 (2)	C18—C19—C20—C15	−0.3 (2)
S1—C4—C5—C6	−179.94 (13)	O1—C2—N1—C15	−6.1 (2)
C4—C5—C6—C7	−0.8 (3)	C3—C2—N1—C15	171.89 (12)
C5—C6—C7—C8	2.4 (3)	O1—C2—N1—C1	175.13 (13)
C6—C7—C8—C3	−1.5 (3)	C3—C2—N1—C1	−6.91 (19)
C4—C3—C8—C7	−1.0 (2)	C20—C15—N1—C2	86.36 (17)
C2—C3—C8—C7	172.71 (14)	C16—C15—N1—C2	−95.04 (17)
N1—C1—C9—C10	3.89 (19)	C20—C15—N1—C1	−94.78 (16)
S1—C1—C9—C10	−124.24 (13)	C16—C15—N1—C1	83.83 (17)
N1—C1—C9—C14	−173.97 (12)	C9—C1—N1—C2	−80.00 (16)
S1—C1—C9—C14	57.90 (15)	S1—C1—N1—C2	49.48 (16)
C14—C9—C10—C11	0.1 (2)	C9—C1—N1—C15	101.18 (14)
C1—C9—C10—C11	−177.71 (14)	S1—C1—N1—C15	−129.34 (11)
C9—C10—C11—C12	0.1 (3)	C19—C18—O2—C21	0.1 (2)
C10—C11—C12—C13	−0.2 (3)	C17—C18—O2—C21	179.91 (15)
C11—C12—C13—C14	0.0 (3)	C5—C4—S1—C1	−152.63 (12)
C12—C13—C14—C9	0.3 (2)	C3—C4—S1—C1	29.14 (12)
C10—C9—C14—C13	−0.3 (2)	N1—C1—S1—C4	−54.14 (10)
C1—C9—C14—C13	177.63 (13)	C9—C1—S1—C4	75.99 (10)
C20—C15—C16—C17	1.4 (2)		

Hydrogen-bond geometry (Å, º)

Cg(X) = center of gravity of ring (X); D—H···*Cg*X = angle of the D—H bond with the π plane

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1^{i}	0.93	2.59	3.447 (2)	154
C21—H21*B*···O1^{ii}	0.96	2.63	3.352 (2)	132

C5—H5···O2^{iii}	0.93	2.46	3.387 (2)	173
C21—H21*A*···*Cg*4^{iv}	0.96	2.96	3.8068 (10)	148

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

(II) 2-Phenyl-3-[4-(trifluoromethyl)phenyl]-2,3-dihydro-4*H*-1,3-benzothiazin-4-one toluene hemisolvate

Crystal data

$2C_{21}H_{14}F_3NOS·C_7H_8$
M_r = 862.92
Monoclinic, $P2_1/c$
a = 11.953 (2) Å
b = 14.516 (3) Å
c = 24.546 (5) Å
β = 101.024 (4)°
V = 4180.5 (14) Å^3
Z = 4
F(000) = 1784
D_x = 1.371 Mg m^{-3}
Melting point = 404–406 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6665 reflections
θ = 2.2–28.2°
μ = 0.20 mm^{-1}
T = 298 K
Needle, colourless
0.29 × 0.09 × 0.07 mm

Data collection

Bruker CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
T_{min} = 0.592, T_{max} = 0.920
39335 measured reflections
10359 independent reflections
8329 reflections with $I > 2\tilde{}I$)
R_{int} = 0.052
θ_{max} = 28.4°, θ_{min} = 1.6°
h = −15→15
k = −19→19
l = −32→32

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.133
$wR(F^2)$ = 0.240
S = 1.31
10359 reflections
580 parameters
74 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 8.2026P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0015 (2)

Special details

Experimental. Absorption correction: SADABS (Bruker, 2001) was used for absorption correction. R(int) was 0.0709 before and 0.0303 after correction. The Ratio of minimum to maximum transmission is 0.5920. The *λ*/2 correction factor is 0.0015.
The data collection nominally covered a full sphere of reciprocal space by a combination of 4 sets of *ω* scans each set at different *φ* and/or 2*θ* angles and each scan (10 s exposure) covering -0.300° degrees in *ω*. The crystal to detector distance was 5.82 cm.
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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.7415 (3)	1.0190 (3)	0.11278 (17)	0.0326 (9)	
H1	0.6834	0.9719	0.1005	0.039*	
C2	0.9318 (3)	1.0494 (3)	0.09051 (15)	0.0292 (8)	
C3	0.9144 (3)	1.1495 (3)	0.10152 (15)	0.0296 (9)	
C4	0.8087 (4)	1.1904 (3)	0.09618 (16)	0.0347 (9)	
C5	0.8013 (5)	1.2859 (3)	0.10036 (19)	0.0498 (12)	
H5	0.7301	1.3140	0.0959	0.060*	
C6	0.8979 (5)	1.3387 (3)	0.1109 (2)	0.0597 (15)	
H6	0.8919	1.4024	0.1134	0.072*	
C7	1.0031 (5)	1.2988 (3)	0.1178 (2)	0.0549 (14)	
H7	1.0684	1.3350	0.1257	0.066*	
C8	1.0120 (4)	1.2042 (3)	0.11295 (18)	0.0417 (11)	
H8	1.0836	1.1770	0.1173	0.050*	
C9	0.7581 (4)	1.0210 (3)	0.17549 (17)	0.0374 (10)	
C10	0.6620 (5)	1.0249 (4)	0.1991 (2)	0.0641 (16)	
H10	0.5901	1.0276	0.1765	0.077*	
C11	0.6725 (8)	1.0247 (5)	0.2565 (3)	0.094 (3)	
H11	0.6076	1.0267	0.2721	0.113*	
C12	0.7777 (8)	1.0216 (5)	0.2900 (3)	0.095 (3)	
H12	0.7841	1.0226	0.3284	0.114*	
C13	0.8724 (6)	1.0169 (4)	0.2676 (2)	0.0735 (18)	
H13	0.9440	1.0139	0.2904	0.088*	
C14	0.8622 (5)	1.0166 (4)	0.21027 (19)	0.0520 (13)	
H14	0.9277	1.0134	0.1951	0.062*	
C15	0.8522 (3)	0.8954 (3)	0.07876 (15)	0.0287 (8)	
C16	0.8250 (4)	0.8292 (3)	0.11498 (18)	0.0410 (11)	
H16	0.7999	0.8472	0.1470	0.049*	
C17	0.8350 (4)	0.7373 (3)	0.10367 (19)	0.0449 (11)	
H17	0.8168	0.6931	0.1280	0.054*	
C18	0.8718 (4)	0.7104 (3)	0.05646 (18)	0.0368 (10)	
C19	0.8959 (3)	0.7756 (3)	0.01947 (17)	0.0325 (9)	
H19	0.9187	0.7571	−0.0130	0.039*	
C20	0.8862 (3)	0.8678 (3)	0.03046 (15)	0.0307 (9)	
H20	0.9024	0.9117	0.0054	0.037*	
C21	0.8898 (5)	0.6098 (3)	0.0469 (2)	0.0543 (13)	
F1	0.8004 (3)	0.5593 (2)	0.05201 (19)	0.0934 (13)	
F2	0.9121 (5)	0.5909 (2)	−0.00173 (17)	0.1125 (17)	
F3	0.9745 (3)	0.5749 (2)	0.08345 (19)	0.1020 (15)	
N1	0.8416 (3)	0.9909 (2)	0.09121 (13)	0.0287 (7)	
O1	1.0226 (2)	1.0219 (2)	0.08167 (14)	0.0426 (8)	

S1	0.68361 (9)	1.12577 (9)	0.08114 (5)	0.0443 (3)	
C22	0.2359 (3)	0.8724 (3)	0.09757 (16)	0.0312 (9)	
H22	0.1793	0.9219	0.0944	0.037*	
C23	0.4274 (3)	0.8614 (3)	0.07246 (14)	0.0259 (8)	
C24	0.4087 (3)	0.7606 (3)	0.06289 (15)	0.0288 (8)	
C25	0.3015 (4)	0.7208 (3)	0.05029 (16)	0.0361 (10)	
C26	0.2912 (5)	0.6259 (3)	0.0385 (2)	0.0514 (13)	
H26	0.2197	0.5985	0.0300	0.062*	
C27	0.3874 (5)	0.5744 (3)	0.0397 (2)	0.0595 (15)	
H27	0.3805	0.5118	0.0317	0.071*	
C28	0.4935 (5)	0.6129 (3)	0.0526 (2)	0.0548 (13)	
H28	0.5580	0.5766	0.0534	0.066*	
C29	0.5046 (4)	0.7060 (3)	0.06431 (18)	0.0397 (10)	
H29	0.5767	0.7323	0.0732	0.048*	
C30	0.2498 (4)	0.8357 (3)	0.15572 (18)	0.0370 (10)	
C35	0.1522 (5)	0.8182 (5)	0.1762 (3)	0.0771 (19)	
H35	0.0811	0.8304	0.1544	0.093*	
C34	0.1596 (8)	0.7825 (6)	0.2291 (4)	0.110 (3)	
H34	0.0937	0.7719	0.2431	0.132*	
C33	0.2635 (9)	0.7628 (5)	0.2606 (3)	0.102 (3)	
H33	0.2683	0.7372	0.2957	0.123*	
C32	0.3592 (7)	0.7803 (5)	0.2412 (2)	0.084 (2)	
H32	0.4299	0.7672	0.2631	0.101*	
C31	0.3528 (5)	0.8178 (4)	0.18866 (19)	0.0548 (14)	
H31	0.4194	0.8308	0.1758	0.066*	
C36	0.3509 (3)	1.0108 (3)	0.08862 (15)	0.0273 (8)	
C37	0.3283 (4)	1.0558 (3)	0.13476 (17)	0.0393 (10)	
H37	0.3061	1.0225	0.1633	0.047*	
C38	0.3386 (4)	1.1501 (3)	0.13847 (19)	0.0435 (11)	
H38	0.3236	1.1804	0.1697	0.052*	
C39	0.3709 (3)	1.1997 (3)	0.09650 (17)	0.0341 (9)	
C40	0.3922 (3)	1.1551 (3)	0.04987 (16)	0.0328 (9)	
H40	0.4134	1.1888	0.0213	0.039*	
C41	0.3820 (3)	1.0614 (3)	0.04573 (16)	0.0305 (9)	
H41	0.3959	1.0315	0.0142	0.037*	
C42	0.3831 (5)	1.3022 (3)	0.1012 (2)	0.0516 (13)	
F6A	0.308 (2)	1.3467 (7)	0.0659 (11)	0.155 (13)	0.57 (3)
F5A	0.378 (3)	1.3360 (6)	0.1506 (5)	0.104 (7)	0.57 (3)
F4A	0.4828 (13)	1.3323 (8)	0.0936 (12)	0.112 (9)	0.57 (3)
N2	0.3391 (3)	0.9132 (2)	0.08403 (13)	0.0271 (7)	
O2	0.5195 (2)	0.89607 (19)	0.06948 (12)	0.0364 (7)	
S2	0.17800 (9)	0.78640 (9)	0.04611 (5)	0.0457 (3)	
F4B	0.474 (2)	1.3253 (11)	0.1315 (15)	0.138 (16)	0.43 (3)
F5B	0.383 (3)	1.3413 (10)	0.0544 (8)	0.109 (12)	0.43 (3)
F6B	0.301 (2)	1.3415 (7)	0.1170 (15)	0.106 (10)	0.43 (3)
C43	0.6833 (6)	0.7489 (4)	0.2230 (2)	0.0640 (16)	
H43	0.6441	0.8046	0.2200	0.077*	
C44	0.7974 (6)	0.7450 (5)	0.2477 (2)	0.0703 (18)	

H44	0.8358	0.7984	0.2613	0.084*
C45	0.8531 (6)	0.6628 (5)	0.2521 (2)	0.0708 (17)
H45	0.9297	0.6606	0.2689	0.085*
C46	0.7985 (5)	0.5832 (5)	0.2322 (2)	0.0657 (16)
H46	0.8378	0.5276	0.2355	0.079*
C47	0.6844 (5)	0.5861 (4)	0.2072 (2)	0.0599 (15)
C48	0.6283 (5)	0.6680 (4)	0.2028 (2)	0.0582 (14)
H48	0.5517	0.6699	0.1860	0.070*
C49	0.6264 (8)	0.4992 (5)	0.1851 (4)	0.112 (3)
H49A	0.6040	0.4655	0.2149	0.168*
H49B	0.6778	0.4625	0.1686	0.168*
H49C	0.5602	0.5137	0.1577	0.168*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.026 (2)	0.030 (2)	0.044 (2)	−0.0063 (17)	0.0144 (17)	−0.0091 (18)
C2	0.031 (2)	0.031 (2)	0.0272 (19)	0.0012 (17)	0.0082 (16)	0.0014 (16)
C3	0.036 (2)	0.027 (2)	0.0263 (19)	−0.0033 (17)	0.0092 (16)	0.0029 (16)
C4	0.044 (2)	0.031 (2)	0.027 (2)	0.0055 (19)	0.0029 (18)	−0.0022 (17)
C5	0.070 (3)	0.036 (3)	0.045 (3)	0.015 (3)	0.013 (2)	0.003 (2)
C6	0.098 (5)	0.030 (3)	0.055 (3)	−0.008 (3)	0.025 (3)	0.001 (2)
C7	0.068 (4)	0.038 (3)	0.059 (3)	−0.021 (3)	0.014 (3)	−0.002 (2)
C8	0.048 (3)	0.040 (3)	0.037 (2)	−0.012 (2)	0.008 (2)	0.006 (2)
C9	0.046 (3)	0.029 (2)	0.044 (2)	−0.0076 (19)	0.024 (2)	−0.0083 (19)
C10	0.067 (4)	0.060 (4)	0.079 (4)	−0.008 (3)	0.048 (3)	−0.013 (3)
C11	0.129 (7)	0.082 (5)	0.097 (6)	−0.012 (5)	0.088 (5)	−0.012 (4)
C12	0.163 (8)	0.078 (5)	0.060 (4)	−0.021 (6)	0.060 (5)	−0.006 (4)
C13	0.105 (5)	0.072 (4)	0.042 (3)	−0.015 (4)	0.011 (3)	0.005 (3)
C14	0.063 (3)	0.056 (3)	0.039 (3)	−0.011 (3)	0.014 (2)	0.004 (2)
C15	0.0273 (19)	0.028 (2)	0.032 (2)	−0.0021 (16)	0.0086 (16)	−0.0029 (16)
C16	0.055 (3)	0.034 (2)	0.040 (2)	−0.005 (2)	0.025 (2)	−0.0040 (19)
C17	0.057 (3)	0.032 (2)	0.049 (3)	−0.006 (2)	0.019 (2)	0.003 (2)
C18	0.033 (2)	0.029 (2)	0.048 (3)	−0.0012 (18)	0.0060 (19)	−0.0052 (19)
C19	0.029 (2)	0.035 (2)	0.035 (2)	−0.0013 (18)	0.0089 (17)	−0.0079 (18)
C20	0.030 (2)	0.034 (2)	0.0288 (19)	−0.0029 (18)	0.0076 (16)	0.0001 (17)
C21	0.061 (3)	0.031 (3)	0.069 (4)	0.000 (2)	0.011 (3)	−0.005 (2)
F1	0.080 (3)	0.0350 (17)	0.166 (4)	−0.0179 (17)	0.025 (3)	−0.019 (2)
F2	0.206 (5)	0.047 (2)	0.101 (3)	0.028 (3)	0.071 (3)	−0.014 (2)
F3	0.095 (3)	0.0451 (19)	0.144 (4)	0.0244 (19)	−0.032 (3)	−0.003 (2)
N1	0.0263 (17)	0.0288 (17)	0.0338 (17)	−0.0029 (14)	0.0126 (14)	−0.0020 (14)
O1	0.0307 (16)	0.0365 (17)	0.065 (2)	−0.0004 (13)	0.0208 (15)	0.0002 (15)
S1	0.0308 (6)	0.0465 (7)	0.0526 (7)	0.0113 (5)	0.0007 (5)	−0.0057 (6)
C22	0.026 (2)	0.028 (2)	0.043 (2)	0.0014 (17)	0.0153 (17)	−0.0001 (18)
C23	0.0272 (19)	0.030 (2)	0.0219 (17)	−0.0002 (16)	0.0078 (15)	−0.0006 (15)
C24	0.037 (2)	0.0222 (19)	0.0273 (19)	0.0016 (17)	0.0065 (16)	−0.0013 (15)
C25	0.044 (2)	0.034 (2)	0.029 (2)	−0.009 (2)	0.0050 (18)	−0.0046 (18)
C26	0.065 (3)	0.041 (3)	0.047 (3)	−0.022 (3)	0.007 (2)	−0.009 (2)

C27	0.092 (5)	0.030 (3)	0.058 (3)	−0.003 (3)	0.019 (3)	−0.009 (2)
C28	0.076 (4)	0.031 (3)	0.061 (3)	0.015 (3)	0.021 (3)	0.000 (2)
C29	0.046 (3)	0.036 (2)	0.039 (2)	0.006 (2)	0.014 (2)	−0.0002 (19)
C30	0.047 (3)	0.026 (2)	0.046 (2)	−0.0053 (19)	0.028 (2)	0.0014 (18)
C35	0.074 (4)	0.089 (5)	0.082 (4)	−0.026 (4)	0.049 (4)	−0.003 (4)
C34	0.146 (8)	0.101 (6)	0.113 (7)	−0.037 (6)	0.101 (6)	0.005 (5)
C33	0.215 (11)	0.047 (4)	0.067 (5)	−0.005 (5)	0.083 (6)	0.009 (3)
C32	0.133 (6)	0.077 (4)	0.047 (3)	0.038 (4)	0.028 (4)	0.017 (3)
C31	0.071 (4)	0.060 (3)	0.037 (3)	0.016 (3)	0.021 (2)	0.013 (2)
C36	0.0263 (19)	0.027 (2)	0.0291 (19)	0.0026 (16)	0.0066 (15)	0.0038 (16)
C37	0.054 (3)	0.034 (2)	0.036 (2)	0.003 (2)	0.023 (2)	0.0026 (18)
C38	0.060 (3)	0.033 (2)	0.043 (2)	0.004 (2)	0.022 (2)	−0.010 (2)
C39	0.035 (2)	0.026 (2)	0.042 (2)	0.0013 (18)	0.0074 (18)	−0.0025 (18)
C40	0.034 (2)	0.032 (2)	0.034 (2)	0.0029 (18)	0.0098 (17)	0.0094 (17)
C41	0.034 (2)	0.031 (2)	0.0288 (19)	0.0031 (17)	0.0105 (17)	−0.0001 (16)
C42	0.063 (4)	0.035 (3)	0.057 (3)	−0.005 (3)	0.010 (3)	−0.003 (2)
F6A	0.166 (17)	0.036 (4)	0.20 (2)	0.001 (9)	−0.125 (16)	0.018 (11)
F5A	0.20 (2)	0.038 (4)	0.084 (7)	−0.002 (8)	0.062 (10)	−0.020 (4)
F4A	0.096 (12)	0.053 (6)	0.21 (2)	−0.034 (7)	0.078 (15)	−0.037 (11)
N2	0.0268 (16)	0.0257 (16)	0.0306 (17)	0.0005 (14)	0.0098 (13)	0.0017 (13)
O2	0.0318 (15)	0.0311 (15)	0.0500 (18)	−0.0044 (13)	0.0170 (13)	−0.0068 (13)
S2	0.0273 (5)	0.0535 (7)	0.0526 (7)	−0.0094 (5)	−0.0015 (5)	−0.0055 (6)
F4B	0.14 (2)	0.048 (6)	0.18 (3)	−0.020 (11)	−0.085 (19)	−0.028 (14)
F5B	0.22 (3)	0.029 (5)	0.086 (12)	−0.007 (13)	0.060 (17)	0.007 (6)
F6B	0.117 (15)	0.037 (5)	0.18 (3)	0.028 (7)	0.081 (16)	−0.016 (11)
C43	0.087 (4)	0.069 (4)	0.040 (3)	0.004 (3)	0.021 (3)	0.016 (3)
C44	0.090 (5)	0.080 (5)	0.041 (3)	−0.032 (4)	0.015 (3)	0.002 (3)
C45	0.066 (4)	0.098 (5)	0.048 (3)	−0.010 (4)	0.008 (3)	0.008 (3)
C46	0.072 (4)	0.079 (4)	0.048 (3)	0.019 (3)	0.016 (3)	0.017 (3)
C47	0.072 (4)	0.061 (4)	0.048 (3)	−0.008 (3)	0.013 (3)	0.014 (3)
C48	0.056 (3)	0.072 (4)	0.046 (3)	0.004 (3)	0.007 (2)	0.018 (3)
C49	0.131 (7)	0.080 (5)	0.124 (7)	−0.012 (5)	0.026 (6)	0.010 (5)

Geometric parameters (Å, º)

C1—H1	0.9800	C25—C26	1.407 (6)
C1—C9	1.514 (6)	C25—S2	1.743 (5)
C1—N1	1.456 (5)	C26—H26	0.9300
C1—S1	1.811 (4)	C26—C27	1.367 (7)
C2—C3	1.501 (5)	C27—H27	0.9300
C2—N1	1.375 (5)	C27—C28	1.367 (7)
C2—O1	1.215 (4)	C28—H28	0.9300
C3—C4	1.379 (5)	C28—C29	1.384 (6)
C3—C8	1.395 (6)	C29—H29	0.9300
C4—C5	1.394 (6)	C30—C35	1.379 (6)
C4—S1	1.744 (4)	C30—C31	1.362 (6)
C5—H5	0.9300	C35—H35	0.9300
C5—C6	1.369 (7)	C35—C34	1.386 (9)

C6—H6	0.9300	C34—H34	0.9300
C6—C7	1.365 (7)	C34—C33	1.362 (10)
C7—H7	0.9300	C33—H33	0.9300
C7—C8	1.384 (6)	C33—C32	1.344 (10)
C8—H8	0.9300	C32—H32	0.9300
C9—C10	1.383 (6)	C32—C31	1.389 (7)
C9—C14	1.369 (6)	C31—H31	0.9300
C10—H10	0.9300	C36—C37	1.378 (5)
C10—C11	1.390 (8)	C36—C41	1.391 (5)
C11—H11	0.9300	C36—N2	1.425 (5)
C11—C12	1.365 (10)	C37—H37	0.9300
C12—H12	0.9300	C37—C38	1.376 (6)
C12—C13	1.353 (9)	C38—H38	0.9300
C13—H13	0.9300	C38—C39	1.371 (6)
C13—C14	1.388 (7)	C39—C40	1.380 (5)
C14—H14	0.9300	C39—C42	1.497 (6)
C15—C16	1.390 (5)	C40—H40	0.9300
C15—C20	1.383 (5)	C40—C41	1.368 (5)
C15—N1	1.430 (5)	C41—H41	0.9300
C16—H16	0.9300	C42—F6A	1.292 (9)
C16—C17	1.373 (6)	C42—F5A	1.321 (9)
C17—H17	0.9300	C42—F4A	1.316 (10)
C17—C18	1.372 (6)	C42—F4B	1.239 (15)
C18—C19	1.379 (6)	C42—F5B	1.283 (15)
C18—C21	1.501 (6)	C42—F6B	1.262 (12)
C19—H19	0.9300	C43—H43	0.9300
C19—C20	1.375 (5)	C43—C44	1.383 (9)
C20—H20	0.9300	C43—C48	1.390 (8)
C21—F1	1.321 (6)	C44—H44	0.9300
C21—F2	1.301 (6)	C44—C45	1.360 (9)
C21—F3	1.318 (6)	C45—H45	0.9300
C22—H22	0.9800	C45—C46	1.371 (9)
C22—C30	1.503 (5)	C46—H46	0.9300
C22—N2	1.463 (5)	C46—C47	1.385 (8)
C22—S2	1.816 (4)	C47—C48	1.358 (8)
C23—C24	1.493 (5)	C47—C49	1.491 (9)
C23—N2	1.370 (5)	C48—H48	0.9300
C23—O2	1.225 (4)	C49—H49A	0.9600
C24—C25	1.386 (5)	C49—H49B	0.9600
C24—C29	1.388 (6)	C49—H49C	0.9600

C9—C1—H1	106.0	C25—C26—H26	120.3
C9—C1—S1	112.6 (3)	C27—C26—C25	119.4 (5)
N1—C1—H1	106.0	C27—C26—H26	120.3
N1—C1—C9	114.7 (3)	C26—C27—H27	119.3
N1—C1—S1	110.9 (3)	C28—C27—C26	121.4 (5)
S1—C1—H1	106.0	C28—C27—H27	119.3
N1—C2—C3	117.4 (3)	C27—C28—H28	120.2

O1—C2—C3	120.6 (4)	C27—C28—C29	119.6 (5)
O1—C2—N1	122.0 (4)	C29—C28—H28	120.2
C4—C3—C2	123.8 (4)	C24—C29—H29	119.8
C4—C3—C8	119.4 (4)	C28—C29—C24	120.5 (5)
C8—C3—C2	116.6 (4)	C28—C29—H29	119.8
C3—C4—C5	119.4 (4)	C35—C30—C22	117.6 (5)
C3—C4—S1	121.4 (3)	C31—C30—C22	123.7 (4)
C5—C4—S1	119.1 (4)	C31—C30—C35	118.7 (5)
C4—C5—H5	119.8	C30—C35—H35	119.9
C6—C5—C4	120.5 (5)	C30—C35—C34	120.2 (7)
C6—C5—H5	119.8	C34—C35—H35	119.9
C5—C6—H6	119.7	C35—C34—H34	120.0
C7—C6—C5	120.7 (5)	C33—C34—C35	119.9 (6)
C7—C6—H6	119.7	C33—C34—H34	120.0
C6—C7—H7	120.2	C34—C33—H33	119.8
C6—C7—C8	119.7 (5)	C32—C33—C34	120.3 (6)
C8—C7—H7	120.2	C32—C33—H33	119.8
C3—C8—H8	119.8	C33—C32—H32	119.9
C7—C8—C3	120.4 (5)	C33—C32—C31	120.2 (7)
C7—C8—H8	119.8	C31—C32—H32	119.9
C10—C9—C1	117.9 (4)	C30—C31—C32	120.6 (5)
C14—C9—C1	124.1 (4)	C30—C31—H31	119.7
C14—C9—C10	118.0 (5)	C32—C31—H31	119.7
C9—C10—H10	119.9	C37—C36—C41	119.6 (4)
C9—C10—C11	120.1 (6)	C37—C36—N2	120.3 (3)
C11—C10—H10	119.9	C41—C36—N2	120.1 (3)
C10—C11—H11	119.8	C36—C37—H37	120.1
C12—C11—C10	120.4 (6)	C38—C37—C36	119.8 (4)
C12—C11—H11	119.8	C38—C37—H37	120.1
C11—C12—H12	120.0	C37—C38—H38	119.8
C13—C12—C11	120.1 (6)	C39—C38—C37	120.5 (4)
C13—C12—H12	120.0	C39—C38—H38	119.8
C12—C13—H13	120.2	C38—C39—C40	120.0 (4)
C12—C13—C14	119.6 (6)	C38—C39—C42	120.1 (4)
C14—C13—H13	120.2	C40—C39—C42	120.0 (4)
C9—C14—C13	121.7 (5)	C39—C40—H40	120.0
C9—C14—H14	119.2	C41—C40—C39	120.0 (4)
C13—C14—H14	119.2	C41—C40—H40	120.0
C16—C15—N1	119.5 (3)	C36—C41—H41	119.9
C20—C15—C16	119.4 (4)	C40—C41—C36	120.1 (4)
C20—C15—N1	121.1 (3)	C40—C41—H41	119.9
C15—C16—H16	119.9	F6A—C42—C39	113.6 (6)
C17—C16—C15	120.2 (4)	F6A—C42—F5A	106.2 (10)
C17—C16—H16	119.9	F6A—C42—F4A	105.4 (10)
C16—C17—H17	120.0	F5A—C42—C39	114.8 (6)
C18—C17—C16	120.1 (4)	F4A—C42—C39	113.4 (7)
C18—C17—H17	120.0	F4A—C42—F5A	102.3 (8)
C17—C18—C19	120.1 (4)	F4B—C42—C39	112.2 (9)

C17—C18—C21	119.1 (4)	F4B—C42—F5B	105.4 (14)
C19—C18—C21	120.7 (4)	F4B—C42—F6B	109.8 (13)
C18—C19—H19	119.9	F5B—C42—C39	112.8 (8)
C20—C19—C18	120.2 (4)	F6B—C42—C39	113.8 (7)
C20—C19—H19	119.9	F6B—C42—F5B	102.2 (11)
C15—C20—H20	120.0	C23—N2—C22	122.8 (3)
C19—C20—C15	120.0 (4)	C23—N2—C36	119.6 (3)
C19—C20—H20	120.0	C36—N2—C22	117.3 (3)
F1—C21—C18	112.5 (4)	C25—S2—C22	97.41 (18)
F2—C21—C18	114.1 (4)	C44—C43—H43	120.6
F2—C21—F1	106.3 (5)	C44—C43—C48	118.7 (6)
F2—C21—F3	106.2 (5)	C48—C43—H43	120.6
F3—C21—C18	112.5 (4)	C43—C44—H44	120.1
F3—C21—F1	104.6 (5)	C45—C44—C43	119.9 (6)
C2—N1—C1	122.2 (3)	C45—C44—H44	120.1
C2—N1—C15	119.6 (3)	C44—C45—H45	119.4
C15—N1—C1	117.5 (3)	C44—C45—C46	121.2 (6)
C4—S1—C1	97.37 (19)	C46—C45—H45	119.4
C30—C22—H22	106.5	C45—C46—H46	120.2
C30—C22—S2	112.0 (3)	C45—C46—C47	119.6 (6)
N2—C22—H22	106.5	C47—C46—H46	120.2
N2—C22—C30	114.5 (3)	C46—C47—C49	119.0 (6)
N2—C22—S2	110.3 (3)	C48—C47—C46	119.4 (6)
S2—C22—H22	106.5	C48—C47—C49	121.6 (6)
N2—C23—C24	118.2 (3)	C43—C48—H48	119.4
O2—C23—C24	120.2 (3)	C47—C48—C43	121.2 (6)
O2—C23—N2	121.6 (3)	C47—C48—H48	119.4
C25—C24—C23	123.3 (4)	C47—C49—H49A	109.5
C25—C24—C29	119.4 (4)	C47—C49—H49B	109.5
C29—C24—C23	117.2 (4)	C47—C49—H49C	109.5
C24—C25—C26	119.6 (4)	H49A—C49—H49B	109.5
C24—C25—S2	121.4 (3)	H49A—C49—H49C	109.5
C26—C25—S2	118.9 (4)	H49B—C49—H49C	109.5

C1—C9—C10—C11	178.5 (5)	C24—C25—C26—C27	0.1 (7)
C1—C9—C14—C13	−178.7 (5)	C24—C25—S2—C22	−31.4 (4)
C2—C3—C4—C5	−171.8 (4)	C25—C24—C29—C28	0.8 (6)
C2—C3—C4—S1	5.2 (5)	C25—C26—C27—C28	0.5 (8)
C2—C3—C8—C7	173.2 (4)	C26—C25—S2—C22	151.0 (4)
C3—C2—N1—C1	−12.6 (5)	C26—C27—C28—C29	−0.4 (8)
C3—C2—N1—C15	177.5 (3)	C27—C28—C29—C24	−0.3 (7)
C3—C4—C5—C6	−1.5 (7)	C29—C24—C25—C26	−0.7 (6)
C3—C4—S1—C1	28.5 (4)	C29—C24—C25—S2	−178.3 (3)
C4—C3—C8—C7	−1.3 (6)	C30—C22—N2—C23	77.0 (4)
C4—C5—C6—C7	−0.3 (8)	C30—C22—N2—C36	−96.8 (4)
C5—C4—S1—C1	−154.5 (4)	C30—C22—S2—C25	−75.1 (3)
C5—C6—C7—C8	1.3 (8)	C30—C35—C34—C33	−1.4 (12)
C6—C7—C8—C3	−0.5 (7)	C35—C30—C31—C32	1.7 (8)

C8—C3—C4—C5	2.2 (6)	C35—C34—C33—C32	1.8 (13)
C8—C3—C4—S1	179.3 (3)	C34—C33—C32—C31	−0.5 (11)
C9—C1—N1—C2	−77.0 (5)	C33—C32—C31—C30	−1.3 (9)
C9—C1—N1—C15	93.1 (4)	C31—C30—C35—C34	−0.4 (9)
C9—C1—S1—C4	76.4 (3)	C36—C37—C38—C39	0.2 (7)
C9—C10—C11—C12	0.6 (10)	C37—C36—C41—C40	1.3 (6)
C10—C9—C14—C13	−0.6 (8)	C37—C36—N2—C22	46.0 (5)
C10—C11—C12—C13	−1.2 (12)	C37—C36—N2—C23	−128.0 (4)
C11—C12—C13—C14	0.9 (11)	C37—C38—C39—C40	0.6 (7)
C12—C13—C14—C9	0.0 (9)	C37—C38—C39—C42	−179.3 (5)
C14—C9—C10—C11	0.3 (8)	C38—C39—C40—C41	−0.5 (6)
C15—C16—C17—C18	−0.1 (7)	C38—C39—C42—F6A	−113 (2)
C16—C15—C20—C19	−1.9 (6)	C38—C39—C42—F5A	9.7 (16)
C16—C15—N1—C1	−41.8 (5)	C38—C39—C42—F4A	126.8 (15)
C16—C15—N1—C2	128.6 (4)	C38—C39—C42—F4B	79 (2)
C16—C17—C18—C19	−1.8 (7)	C38—C39—C42—F5B	−162.5 (19)
C16—C17—C18—C21	175.8 (5)	C38—C39—C42—F6B	−47 (2)
C17—C18—C19—C20	1.8 (6)	C39—C40—C41—C36	−0.4 (6)
C17—C18—C21—F1	51.4 (6)	C40—C39—C42—F6A	67 (2)
C17—C18—C21—F2	172.5 (5)	C40—C39—C42—F5A	−170.3 (15)
C17—C18—C21—F3	−66.5 (6)	C40—C39—C42—F4A	−53.1 (15)
C18—C19—C20—C15	0.0 (6)	C40—C39—C42—F4B	−101 (2)
C19—C18—C21—F1	−131.0 (5)	C40—C39—C42—F5B	17.5 (19)
C19—C18—C21—F2	−9.9 (7)	C40—C39—C42—F6B	133 (2)
C19—C18—C21—F3	111.2 (5)	C41—C36—C37—C38	−1.2 (7)
C20—C15—C16—C17	1.9 (7)	C41—C36—N2—C22	−132.0 (4)
C20—C15—N1—C1	136.4 (4)	C41—C36—N2—C23	54.0 (5)
C20—C15—N1—C2	−53.2 (5)	C42—C39—C40—C41	179.4 (4)
C21—C18—C19—C20	−175.7 (4)	N2—C22—C30—C35	163.7 (4)
N1—C1—C9—C10	−164.6 (4)	N2—C22—C30—C31	−17.2 (6)
N1—C1—C9—C14	13.5 (6)	N2—C22—S2—C25	53.7 (3)
N1—C1—S1—C4	−53.7 (3)	N2—C23—C24—C25	18.0 (5)
N1—C2—C3—C4	−19.7 (6)	N2—C23—C24—C29	−165.2 (3)
N1—C2—C3—C8	166.1 (3)	N2—C36—C37—C38	−179.2 (4)
N1—C15—C16—C17	−179.9 (4)	N2—C36—C41—C40	179.3 (4)
N1—C15—C20—C19	179.9 (4)	O2—C23—C24—C25	−161.5 (4)
O1—C2—C3—C4	160.7 (4)	O2—C23—C24—C29	15.4 (5)
O1—C2—C3—C8	−13.5 (6)	O2—C23—N2—C22	−168.8 (4)
O1—C2—N1—C1	167.0 (4)	O2—C23—N2—C36	4.9 (5)
O1—C2—N1—C15	−2.9 (6)	S2—C22—C30—C35	−69.8 (5)
S1—C1—C9—C10	67.3 (5)	S2—C22—C30—C31	109.3 (4)
S1—C1—C9—C14	−114.6 (4)	S2—C22—N2—C23	−50.4 (4)
S1—C1—N1—C2	51.9 (4)	S2—C22—N2—C36	135.8 (3)
S1—C1—N1—C15	−138.0 (3)	S2—C25—C26—C27	177.7 (4)
S1—C4—C5—C6	−178.6 (4)	C43—C44—C45—C46	0.4 (9)
C22—C30—C35—C34	178.6 (6)	C44—C43—C48—C47	0.3 (8)
C22—C30—C31—C32	−177.3 (5)	C44—C45—C46—C47	−0.1 (9)
C23—C24—C25—C26	176.1 (4)	C45—C46—C47—C48	−0.1 (8)

C23—C24—C25—S2	−1.5 (5)	C45—C46—C47—C49	179.3 (6)
C23—C24—C29—C28	−176.2 (4)	C46—C47—C48—C43	0.0 (8)
C24—C23—N2—C22	11.7 (5)	C48—C43—C44—C45	−0.5 (8)
C24—C23—N2—C36	−174.6 (3)	C49—C47—C48—C43	−179.4 (6)

Hydrogen-bond geometry (Å, º)

Cg(X) = center of gravity of ring (X); D—H···*Cg*X = angle of the D—H bond with the π plane

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2	0.98	2.25	3.2053 (7)	165
C22—H22···O1^{i}	0.98	2.34	3.3140 (7)	171
C17—H17···*Cg*9	0.93	2.79	3.5916 (7)	145
C38—H38···*Cg*9^{ii}	0.93	2.78	3.6009 (7)	147

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

(III) 3-(3-Bromophenyl)-2-phenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one toluene hemisolvate

Crystal data

$2C_{20}H_{14}BrNOS·C_7H_8$
M_r = 884.72
Monoclinic, *C*2/*c*
a = 15.736 (2) Å
b = 9.3530 (15) Å
c = 27.259 (4) Å
β = 99.560 (3)°
V = 3956.2 (10) Å^3
Z = 4
F(000) = 1800
D_x = 1.485 Mg m^{-3}
Melting point = 358–359 K
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 3326 reflections
θ = 2.5–25.0°
μ = 2.20 mm^{-1}
T = 298 K
Block, colorless
0.21 × 0.17 × 0.10 mm

Data collection

Bruker CCD area-detector diffractometer
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
T_{min} = 0.103, T_{max} = 0.901
18289 measured reflections
4903 independent reflections
2424 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 28.3°, θ_{min} = 2.5°
h = −20→20
k = −12→12
l = −36→36

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.153
S = 0.79
4903 reflections
266 parameters
186 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

Special details

Experimental. Absorption correction: SADABS was used for absorption correction. R(int) was 0.2680 before and 0.0355 after correction. The Ratio of minimum to maximum transmission is 0.1027. The $\lambda/2$ correction factor is 0.0015.
The data collection nominally covered a full sphere of reciprocal space by a combination of 4 sets of ω scans each set at different φ and/or 2θ angles and each scan (10 s exposure) covering -0.300° degrees in ω. The crystal to detector distance was 5.82 cm.

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)
Br1	0.45074 (3)	0.27453 (5)	0.01191 (2)	0.0874 (2)	
C1	0.3189 (2)	−0.2500 (3)	−0.12554 (12)	0.0471 (8)	
H1	0.2798	−0.2262	−0.1023	0.057*	
C2	0.4549 (2)	−0.1391 (3)	−0.14162 (11)	0.0479 (8)	
C3	0.4821 (2)	−0.2827 (3)	−0.15744 (12)	0.0506 (8)	
C4	0.4518 (2)	−0.4113 (3)	−0.14088 (11)	0.0540 (9)	
C5	0.4858 (3)	−0.5405 (4)	−0.15463 (15)	0.0736 (12)	
H5	0.4662	−0.6269	−0.1438	0.088*	
C6	0.5487 (3)	−0.5386 (5)	−0.18431 (16)	0.0840 (14)	
H6	0.5723	−0.6246	−0.1927	0.101*	
C7	0.5774 (3)	−0.4129 (5)	−0.20176 (15)	0.0766 (12)	
H7	0.6178	−0.4141	−0.2231	0.092*	
C8	0.5453 (2)	−0.2859 (4)	−0.18817 (13)	0.0613 (9)	
H8	0.5657	−0.2006	−0.1994	0.074*	
C9	0.2622 (2)	−0.2733 (3)	−0.17542 (11)	0.0441 (7)	
C10	0.2728 (2)	−0.1978 (4)	−0.21777 (12)	0.0551 (9)	
H10	0.3176	−0.1322	−0.2164	0.066*	
C11	0.2169 (3)	−0.2193 (4)	−0.26239 (14)	0.0650 (10)	
H11	0.2250	−0.1686	−0.2906	0.078*	
C12	0.1505 (3)	−0.3137 (4)	−0.26511 (14)	0.0653 (10)	
H12	0.1139	−0.3288	−0.2951	0.078*	
C13	0.1381 (2)	−0.3865 (3)	−0.22331 (15)	0.0646 (10)	
H13	0.0915	−0.4485	−0.2247	0.078*	
C14	0.1942 (2)	−0.3683 (3)	−0.17905 (13)	0.0565 (9)	
H14	0.1861	−0.4209	−0.1512	0.068*	
C15	0.3547 (2)	0.0010 (3)	−0.10271 (10)	0.0444 (7)	
C16	0.2748 (2)	0.0582 (4)	−0.11998 (12)	0.0605 (9)	
H16	0.2388	0.0144	−0.1462	0.073*	
C17	0.2477 (3)	0.1802 (4)	−0.09865 (14)	0.0684 (11)	
H17	0.1940	0.2192	−0.1107	0.082*	
C18	0.3005 (3)	0.2443 (3)	−0.05941 (14)	0.0602 (10)	
H18	0.2825	0.3262	−0.0447	0.072*	
C19	0.3790 (2)	0.1866 (3)	−0.04242 (12)	0.0519 (8)	
C20	0.4081 (2)	0.0654 (3)	−0.06351 (11)	0.0498 (8)	

H20	0.4624	0.0280	−0.0516	0.060*	
N1	0.37987 (17)	−0.1309 (2)	−0.12306 (9)	0.0436 (6)	
O1	0.49900 (16)	−0.0328 (3)	−0.14536 (9)	0.0642 (7)	
S1	0.37419 (7)	−0.41236 (9)	−0.10170 (3)	0.0632 (3)	
C21	0.3612 (7)	−0.228 (2)	0.0506 (4)	0.146 (15)	0.5
H21A	0.4040	−0.2957	0.0440	0.219*	0.5
H21B	0.3479	−0.2455	0.0832	0.219*	0.5
H21C	0.3831	−0.1330	0.0489	0.219*	0.5
C22	0.2809 (6)	−0.2458 (12)	0.0125 (3)	0.135 (7)	0.5
C23	0.2341 (8)	−0.1264 (10)	−0.0081 (4)	0.123 (8)	0.5
H23	0.2537	−0.0351	0.0015	0.147*	0.5
C24	0.1585 (7)	−0.1422 (11)	−0.0426 (4)	0.145 (9)	0.5
H24	0.1283	−0.0618	−0.0558	0.174*	0.5
C25	0.1282 (5)	−0.2785 (13)	−0.0573 (3)	0.160 (14)	0.5
H25	0.0779	−0.2893	−0.0803	0.192*	0.5
C26	0.1743 (6)	−0.3985 (10)	−0.0374 (3)	0.086 (3)	0.5
H26	0.1546	−0.4896	−0.0470	0.103*	0.5
C27	0.2498 (6)	−0.3820 (10)	−0.0028 (3)	0.105 (6)	0.5
H27	0.2800	−0.4626	0.0102	0.125*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0933 (4)	0.0782 (3)	0.0831 (3)	0.0078 (2)	−0.0077 (2)	−0.0405 (2)
C1	0.054 (2)	0.0439 (17)	0.0465 (18)	−0.0039 (14)	0.0166 (15)	−0.0027 (13)
C2	0.055 (2)	0.0477 (18)	0.0418 (17)	0.0015 (16)	0.0104 (15)	−0.0051 (13)
C3	0.050 (2)	0.0543 (18)	0.0451 (18)	0.0068 (15)	−0.0007 (15)	−0.0095 (14)
C4	0.058 (2)	0.0494 (18)	0.0483 (18)	0.0078 (16)	−0.0085 (15)	−0.0059 (14)
C5	0.077 (3)	0.050 (2)	0.083 (3)	0.0155 (19)	−0.017 (2)	−0.0109 (18)
C6	0.076 (3)	0.082 (3)	0.086 (3)	0.043 (3)	−0.010 (2)	−0.031 (2)
C7	0.063 (3)	0.099 (3)	0.064 (2)	0.026 (2)	−0.0003 (19)	−0.021 (2)
C8	0.050 (2)	0.080 (2)	0.053 (2)	0.0138 (19)	0.0033 (17)	−0.0137 (17)
C9	0.050 (2)	0.0359 (14)	0.0486 (18)	0.0026 (14)	0.0144 (15)	−0.0056 (12)
C10	0.056 (2)	0.0550 (19)	0.055 (2)	−0.0074 (16)	0.0098 (17)	−0.0003 (15)
C11	0.070 (3)	0.070 (2)	0.054 (2)	0.003 (2)	0.0094 (19)	−0.0010 (17)
C12	0.067 (3)	0.056 (2)	0.067 (2)	0.009 (2)	−0.0057 (19)	−0.0172 (18)
C13	0.056 (3)	0.0447 (18)	0.091 (3)	−0.0053 (17)	0.004 (2)	−0.0168 (19)
C14	0.058 (2)	0.0423 (17)	0.070 (2)	−0.0031 (16)	0.0157 (18)	−0.0022 (15)
C15	0.056 (2)	0.0394 (15)	0.0404 (16)	−0.0004 (14)	0.0144 (15)	−0.0006 (12)
C16	0.062 (2)	0.062 (2)	0.054 (2)	0.0100 (18)	−0.0033 (17)	−0.0125 (16)
C17	0.065 (3)	0.066 (2)	0.069 (2)	0.024 (2)	−0.0027 (19)	−0.0134 (19)
C18	0.075 (3)	0.0435 (18)	0.064 (2)	0.0102 (17)	0.015 (2)	−0.0054 (15)
C19	0.063 (2)	0.0442 (16)	0.0478 (18)	0.0006 (16)	0.0085 (16)	−0.0081 (14)
C20	0.054 (2)	0.0458 (17)	0.0496 (18)	0.0016 (15)	0.0098 (15)	−0.0043 (14)
N1	0.0467 (17)	0.0403 (13)	0.0457 (14)	−0.0042 (11)	0.0132 (12)	−0.0079 (11)
O1	0.0639 (17)	0.0552 (14)	0.0797 (16)	−0.0133 (12)	0.0304 (13)	−0.0105 (12)
S1	0.0802 (7)	0.0477 (5)	0.0597 (5)	−0.0038 (4)	0.0061 (5)	0.0101 (4)
C21	0.062 (11)	0.31 (5)	0.074 (10)	−0.006 (17)	0.029 (9)	0.029 (15)

C22	0.131 (17)	0.196 (15)	0.104 (13)	−0.034 (15)	0.096 (13)	−0.052 (12)
C23	0.147 (16)	0.109 (14)	0.143 (13)	−0.016 (13)	0.118 (12)	−0.049 (13)
C24	0.19 (2)	0.097 (10)	0.18 (2)	0.028 (11)	0.128 (17)	0.041 (11)
C25	0.120 (17)	0.26 (4)	0.123 (15)	−0.085 (18)	0.076 (12)	−0.080 (18)
C26	0.088 (8)	0.094 (7)	0.084 (7)	0.004 (6)	0.040 (6)	0.014 (6)
C27	0.174 (18)	0.056 (8)	0.106 (10)	0.002 (9)	0.088 (11)	0.002 (6)

Geometric parameters (Å, º)

Br1—C19	1.895 (3)	C13—C14	1.383 (5)
C1—H1	0.9800	C14—H14	0.9300
C1—C9	1.513 (5)	C15—C16	1.376 (4)
C1—N1	1.464 (4)	C15—C20	1.382 (4)
C1—S1	1.815 (3)	C15—N1	1.435 (4)
C2—C3	1.494 (4)	C16—H16	0.9300
C2—N1	1.362 (4)	C16—C17	1.380 (5)
C2—O1	1.227 (4)	C17—H17	0.9300
C3—C4	1.396 (5)	C17—C18	1.377 (5)
C3—C8	1.404 (5)	C18—H18	0.9300
C4—C5	1.398 (5)	C18—C19	1.357 (5)
C4—S1	1.750 (4)	C19—C20	1.384 (4)
C5—H5	0.9300	C20—H20	0.9300
C5—C6	1.379 (6)	C21—H21A	0.9600
C6—H6	0.9300	C21—H21B	0.9600
C6—C7	1.372 (6)	C21—H21C	0.9600
C7—H7	0.9297	C21—C22	1.5069
C7—C8	1.365 (5)	C22—C23	1.4022
C8—H8	0.9300	C22—C27	1.4024
C9—C10	1.387 (5)	C23—H23	0.9300
C9—C14	1.382 (4)	C23—C24	1.3963
C10—H10	0.9300	C24—H24	0.9300
C10—C11	1.392 (5)	C24—C25	1.3964
C11—H11	0.9300	C25—H25	0.9300
C11—C12	1.361 (5)	C25—C26	1.3966
C12—H12	0.9300	C26—H26	0.9300
C12—C13	1.369 (5)	C26—C27	1.3963
C13—H13	0.9300	C27—H27	0.9300
C9—C1—H1	105.9	C16—C15—N1	119.8 (3)
C9—C1—S1	112.2 (2)	C20—C15—N1	120.1 (3)
N1—C1—H1	105.9	C15—C16—H16	119.8
N1—C1—C9	115.8 (2)	C15—C16—C17	120.4 (3)
N1—C1—S1	110.4 (2)	C17—C16—H16	119.8
S1—C1—H1	105.9	C16—C17—H17	120.0
N1—C2—C3	117.7 (3)	C18—C17—C16	119.9 (3)
O1—C2—C3	120.8 (3)	C18—C17—H17	120.0
O1—C2—N1	121.5 (3)	C17—C18—H18	120.4
C4—C3—C2	123.5 (3)	C19—C18—C17	119.3 (3)

C4—C3—C8	119.3 (3)	C19—C18—H18	120.4
C8—C3—C2	117.1 (3)	C18—C19—Br1	119.0 (2)
C3—C4—C5	119.4 (4)	C18—C19—C20	122.0 (3)
C3—C4—S1	120.8 (2)	C20—C19—Br1	119.0 (3)
C5—C4—S1	119.7 (3)	C15—C20—C19	118.5 (3)
C4—C5—H5	120.3	C15—C20—H20	120.8
C6—C5—C4	119.4 (4)	C19—C20—H20	120.8
C6—C5—H5	120.3	C2—N1—C1	122.8 (2)
C5—C6—H6	119.2	C2—N1—C15	120.2 (2)
C7—C6—C5	121.6 (4)	C15—N1—C1	116.9 (2)
C7—C6—H6	119.2	C4—S1—C1	96.77 (15)
C6—C7—H7	120.3	H21A—C21—H21B	109.5
C8—C7—C6	119.6 (4)	H21A—C21—H21C	109.5
C8—C7—H7	120.1	H21B—C21—H21C	109.5
C3—C8—H8	119.6	C22—C21—H21A	109.5
C7—C8—C3	120.7 (4)	C22—C21—H21B	109.5
C7—C8—H8	119.6	C22—C21—H21C	109.5
C10—C9—C1	122.7 (3)	C23—C22—C21	121.0
C14—C9—C1	119.4 (3)	C23—C22—C27	118.1
C14—C9—C10	117.8 (3)	C27—C22—C21	120.9
C9—C10—H10	119.7	C22—C23—H23	119.4
C9—C10—C11	120.5 (3)	C24—C23—C22	121.1
C11—C10—H10	119.7	C24—C23—H23	119.4
C10—C11—H11	119.7	C23—C24—H24	119.9
C12—C11—C10	120.6 (4)	C23—C24—C25	120.1
C12—C11—H11	119.7	C25—C24—H24	119.9
C11—C12—H12	120.3	C24—C25—H25	120.3
C11—C12—C13	119.5 (3)	C24—C25—C26	119.4
C13—C12—H12	120.3	C26—C25—H25	120.3
C12—C13—H13	119.8	C25—C26—H26	119.9
C12—C13—C14	120.4 (3)	C27—C26—C25	120.1
C14—C13—H13	119.8	C27—C26—H26	119.9
C9—C14—C13	121.1 (3)	C22—C27—H27	119.5
C9—C14—H14	119.4	C26—C27—C22	121.1
C13—C14—H14	119.4	C26—C27—H27	119.5
C16—C15—C20	119.9 (3)		

Br1—C19—C20—C15	179.2 (2)	C17—C18—C19—Br1	−179.5 (3)
C1—C9—C10—C11	−177.6 (3)	C17—C18—C19—C20	0.4 (6)
C1—C9—C14—C13	176.3 (3)	C18—C19—C20—C15	−0.7 (5)
C2—C3—C4—C5	−175.0 (3)	C20—C15—C16—C17	0.5 (5)
C2—C3—C4—S1	3.0 (5)	C20—C15—N1—C1	125.6 (3)
C2—C3—C8—C7	176.0 (3)	C20—C15—N1—C2	−58.2 (4)
C3—C2—N1—C1	−10.2 (4)	N1—C1—C9—C10	6.7 (4)
C3—C2—N1—C15	173.8 (3)	N1—C1—C9—C14	−170.0 (3)
C3—C4—C5—C6	0.0 (5)	N1—C1—S1—C4	−54.9 (2)
C3—C4—S1—C1	31.4 (3)	N1—C2—C3—C4	−20.5 (5)
C4—C3—C8—C7	−0.2 (5)	N1—C2—C3—C8	163.5 (3)

C4—C5—C6—C7	−1.6 (6)	N1—C15—C16—C17	176.0 (3)
C5—C4—S1—C1	−150.5 (3)	N1—C15—C20—C19	−175.2 (3)
C5—C6—C7—C8	2.3 (6)	O1—C2—C3—C4	159.5 (3)
C6—C7—C8—C3	−1.4 (6)	O1—C2—C3—C8	−16.5 (5)
C8—C3—C4—C5	0.9 (5)	O1—C2—N1—C1	169.7 (3)
C8—C3—C4—S1	179.0 (2)	O1—C2—N1—C15	−6.2 (4)
C9—C1—N1—C2	−77.8 (4)	S1—C1—C9—C10	−121.2 (3)
C9—C1—N1—C15	98.2 (3)	S1—C1—C9—C14	62.0 (4)
C9—C1—S1—C4	75.8 (3)	S1—C1—N1—C2	51.0 (3)
C9—C10—C11—C12	0.6 (5)	S1—C1—N1—C15	−132.9 (2)
C10—C9—C14—C13	−0.6 (5)	S1—C4—C5—C6	−178.1 (3)
C10—C11—C12—C13	1.0 (6)	C21—C22—C23—C24	−178.5
C11—C12—C13—C14	−2.4 (5)	C21—C22—C27—C26	178.5
C12—C13—C14—C9	2.2 (5)	C22—C23—C24—C25	−0.1
C14—C9—C10—C11	−0.8 (5)	C23—C22—C27—C26	−0.3
C15—C16—C17—C18	−0.9 (6)	C23—C24—C25—C26	−0.1
C16—C15—C20—C19	0.3 (5)	C24—C25—C26—C27	0.1
C16—C15—N1—C1	−49.8 (4)	C25—C26—C27—C22	0.1
C16—C15—N1—C2	126.4 (3)	C27—C22—C23—C24	0.3
C16—C17—C18—C19	0.4 (6)		

Hydrogen-bond geometry (Å, º)

Cg(X) = center of gravity of ring (X); D—H···Cg(X) = angle of the D—H bond with the π plane

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···*Cg*5	0.98	2.66	3.5802 (6)	156
C6—H6···*Cg*3^{i}	0.93	2.83	3.6823 (6)	153

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