organic compounds

{2-[(4-Nitrobenzylidene)amino]-4,5,6,7-tetrahydro-1-benzothiophen-3-yl}-(phenyl)methanone

Manpreet Kaur,[a] Jerry P. Jasinski,[b]* Channappa N. Kavitha,[a] H. S. Yathirajan[a] and K. Byrappa[c]

[a]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, [b]Department of Chemistry, Keene State College, 229 Main Street, Keene, NH 03435-2001, USA, and [c]Materials Science Center, University of Mysore, Vijyana Bhavan Building, Manasagangothri, Mysore 570 006, India
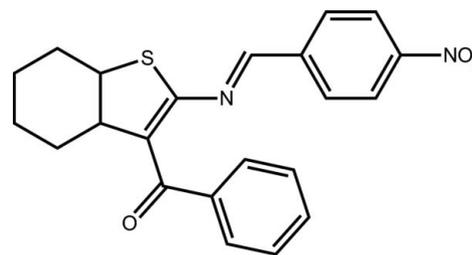Correspondence e-mail: jjasinski@keene.edu

In the title compound, $C_{22}H_{18}N_2O_3S$, disorder is found in the benzoyl group (A and B), as well as for four C atoms of the cyclohexene ring. Two orientations were modeled in a 0.583 (5):0.417 (5) ratio. The cyclohexene ring is in a distorted chair conformation. The dihedral angles between the mean plane of the thiophene ring and the 4-nitrobenzene and phenyl rings are 30.9 (8) and 64.8 (3) (A) and 62.4 (7)° (B). The mean planes of the 4-nitrobenzene and the phenyl rings are almost perpendicular to each other, with dihedral angles of 85.4 (1) (A) and 83.9 (8)° (B). An extensive array of weak C—H···O interactions consolidate molecules into a three-dimensional architecture, forming chains along [001] and [010] and layers parallel to (011).

Related literature

For applications of 2-aminothiophene derivatives in pesticides, dyes and pharmaceuticals, see: Puterová *et al.* (2010). For the biological and industrial importance of Schiff bases, see: Desai *et al.* (2001); Karia & Parsania (1999); Samadhiya & Halve (2001); Singh & Dash (1988). For Schiff bases utilized as starting materials in the synthesis of compounds of industrial and biological interest, see: Aydogan *et al.* (2001); Taggi *et al.* (2002). For related structures, see: Kaur *et al.* (2014*a*,*b*); Kubicki *et al.* (2012). For puckering parameters, see: Cremer & Pople (1975).

Experimental

Crystal data

$C_{22}H_{18}N_2O_3S$	V = 955.39 (4) Å^3
M_r = 390.44	Z = 2
Monoclinic, $P2_1$	Cu $K\alpha$ radiation
a = 4.61595 (13) Å	μ = 1.72 mm^{-1}
b = 17.6844 (4) Å	T = 173 K
c = 11.7068 (3) Å	$0.24 \times 0.18 \times 0.06$ mm
β = 91.285 (3)°	

Data collection

Agilent Eos Gemini diffractometer	6206 measured reflections
Absorption correction: multi-scan (*CrysAlis PRO* and *CrysAlis RED*; Agilent, 2012)	2781 independent reflections
	2610 reflections with $I > 2\sigma(I)$
T_{min} = 0.634, T_{max} = 1.000	R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
$wR(F^2)$ = 0.090	Absolute structure: Flack x determined using 764 quotients $[(I^+)−(I^-)]/[(I^+)+(I^-)]$ (Parsons & Flack, 2004)
S = 1.02	
2781 reflections	
263 parameters	
138 restraints	Absolute structure parameter: 0.028 (16)
H-atom parameters constrained	
$\Delta\rho_{max}$ = 0.21 e Å^{-3}	

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

D—H···A	D—H	H···A	D···A	D—H···A
C4A—H4AA···O1B^i	0.99	2.38	3.15 (4)	135
C4B—H4BA···O1A^i	0.99	2.50	3.45 (4)	162
C4B—H4BA···O1B^i	0.99	2.26	3.18 (4)	154
C7B—H7BB···O2^{ii}	0.99	2.46	3.44 (3)	169
C13B—H13B···O3^{iii}	0.95	2.55	3.371 (13)	145
C21—H21···O1A^{iv}	0.95	2.40	3.127 (18)	133
C21—H21···O1B^{iv}	0.95	2.44	3.13 (3)	129

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

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Agilent, 2012); program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL2012* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5318).

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supporting information

{2-[(4-Nitrobenzylidene)amino]-4,5,6,7-tetrahydro-1-benzothiophen-3-yl} (phenyl)methanone

Manpreet Kaur, Jerry P. Jasinski, Channappa N. Kavitha, H. S. Yathirajan and K. Byrappa

S1. Structural commentary

2-Aminothiophene derivatives have been used in a number of applications, such as in pesticides, dyes and pharmaceuticals. A review on the synthesis and properties of these compounds has been reported (Puterová *et al.*, 2010). Schiff base compounds are an important class of compounds, both synthetically and biologically. These compounds show biological activities including anti-bacterial, anti-fungal, anti-cancer and herbicidal (Desai *et al.*, 2001; Karia & Parsania, 1999; Samadhiya & Halve, 2001; Singh & Dash, 1988). Furthermore, Schiff bases are utilized as starting materials in the synthesis of compounds of industrial (Aydogan *et al.*, 2001) and biological interest such as *β*-lactams (Taggi *et al.*, 2002). Some of the recently reported Schiff base structures of 2-aminothiophenes by our group include {2-[(2-hydroxybenzylidene)amino]-4,5,6,7-tetrahydro-1-benzothiophene-3-yl} (phenyl)methanone (Kaur *et al.*, 2014*a*) and [2-benzylidene-amino)-4,5,6,7-tetrahydro benzo[b]thiophene-3-yl](phenyl) methanone (Kaur *et al.*, 2014*b*). Also, the crystal and molecular structures of two 2-aminothiophenes have been previously reported by our group (Kubicki *et al.*, 2012). In continuation of our work on 2-aminothiophenes and Schiff bases, we report here the crystal structure of the title compound, (I).

In (I), the cyclohexene ring is in a distorted chair conformation with four carbon atoms disordered over two sets of sites with an occupancy ratio of 0.583 (5): 0.417 (5) (Fig. 1). Puckering parameters C3/C4A—C7A/C8: Q and φ = 0.511 (4) Å and 157.387 (6)°, and C3/C4B—C7B/C8: Q and φ = 0.483 (8) Å and 212.306 (8)° (Cremer & Pople, 1975). The disorder extends to the benzoyl residue (A & B). The dihedral angles between the mean plane of the thiophene ring and the 4-nitrophenyl and the phenyl rings is 30.9 (8) and 64.8 (3) (A) and 62.4 (7)° (B), respectively. The mean planes of 4-nitrophenyl and the phenyl rings are almost perpendicular to each other with a dihedral angle of 85.4 (1) (A) and 83.9 (8)° (B). An extensive array of weak C—H···O intermolecular interactions leads to a 3-D architecure, forming chains along [001] and [010] and layers parallel to (011) (Fig. 2).

S2. Synthesis and crystallization

To a solution of (2-amino-4,5,6,7-tetrahydro-benzo[b]thiophen-3-yl)- phenyl-methanone (200 mg, 0.79 mmol) in 10 ml of methanol an equimolar amount of 4-nitrobenzaldehyde (120 mg, 0.79 mmol) was added with constant stirring. The mixture was refluxed for 3 h. A yellow precipitate was obtained. Completion of the reaction was confirmed by TLC. The precipitate was filtered and dried at room temperature overnight. The solid was recrystallized using ethylacetate and the crystals were used as such for x-ray diffraction studies.

S3. Refinement

The H atoms were placed in their calculated positions and then refined using the riding model with Atom—H lengths of 0.93 Å (CH) or 0.97 Å (CH₂), and with U_iso set to 1.2U_eq of the parent atom. Disorder was modeled for C4A—C7A and

C4B—C7B of the cyclohexane ring, C10A—C15A/O1A and C10B—C15B/O1B of the benzoyl group over two positions with an occupancy ratio of 0.583 (5):0.417 (5).
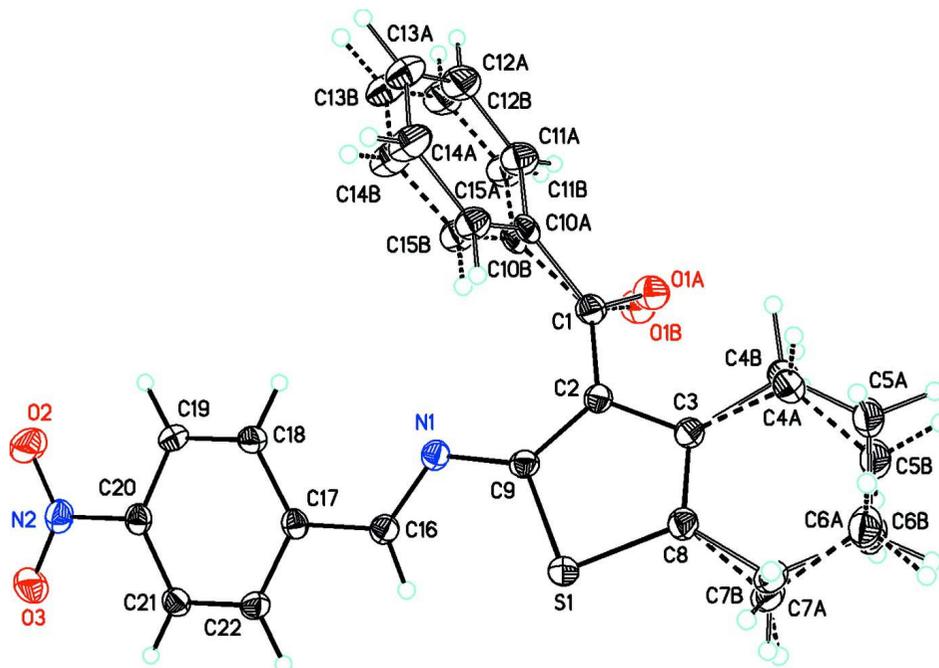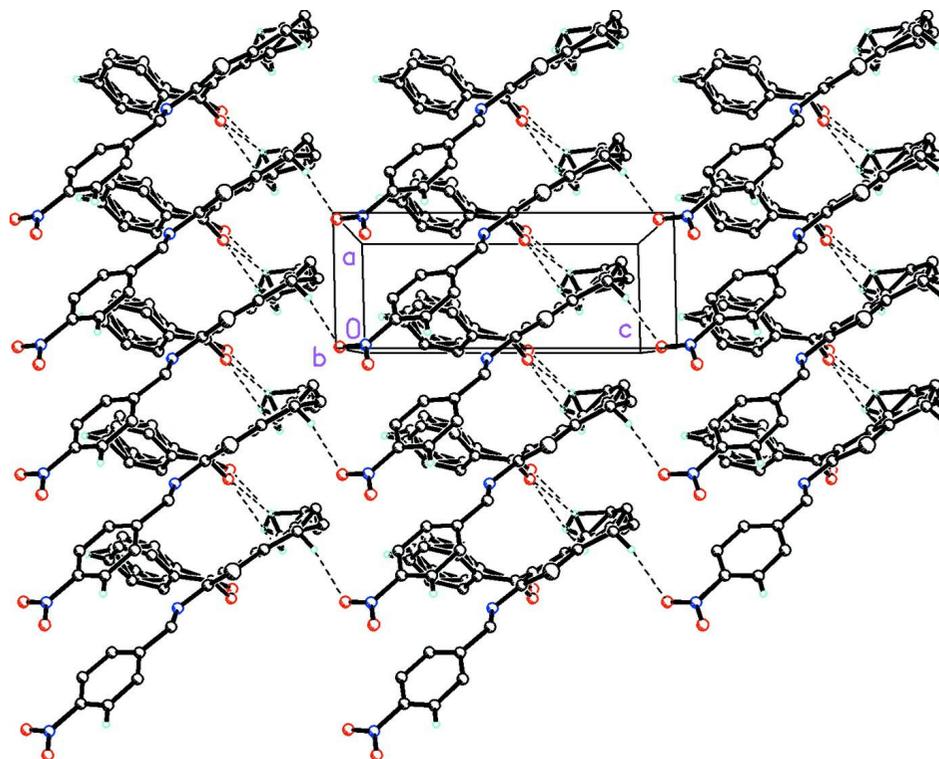

Figure 1

ORTEP drawing of (I) showing the labeling scheme and 30% probability displacement ellipsoids (major and minor components of the disordered atoms in the cyclohexane, benzoyl groups are displayed with dashed lines).

Figure 2

Molecular packing for (I) viewed along the *b* axis. Dashed lines indicate weak C—H···O intermolecular interactions. H atoms not involved in the weak intermolecular interactions have been removed for clarity.

{2-[(4-Nitrobenzylidene)amino]-4,5,6,7-tetrahydro-1-benzothiophen-3-yl}(phenyl)methanone

Crystal data

$C_{22}H_{18}N_2O_3S$	$F(000) = 408$
$M_r = 390.44$	$D_x = 1.357$ Mg m^{-3}
Monoclinic, $P2_1$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$a = 4.61595$ (13) Å	Cell parameters from 3146 reflections
$b = 17.6844$ (4) Å	$\theta = 4.5–71.4°$
$c = 11.7068$ (3) Å	$\mu = 1.72$ mm^{-1}
$\beta = 91.285$ (3)°	$T = 173$ K
$V = 955.39$ (4) Å^3	Irregular, yellow
$Z = 2$	$0.24 \times 0.18 \times 0.06$ mm

Data collection

Agilent Eos Gemini diffractometer	6206 measured reflections
Radiation source: Enhance (Cu) X-ray Source	2781 independent reflections
Detector resolution: 16.0416 pixels mm^{-1}	2610 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.027$
Absorption correction: multi-scan (*CrysAlis PRO* and *CrysAlis RED*; Agilent, 2012)	$\theta_{max} = 71.1°$, $\theta_{min} = 3.8°$
	$h = -5 \rightarrow 5$
	$k = -18 \rightarrow 21$
	$l = -11 \rightarrow 14$
$T_{min} = 0.634$, $T_{max} = 1.000$	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.035$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.090$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.21$ e Å^{-3}
2781 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}
263 parameters	Absolute structure: Flack x determined using
138 restraints	764 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons &
Primary atom site location: structure-invariant	Flack, 2004)
direct methods	Absolute structure parameter: 0.028 (16)
Hydrogen site location: mixed	

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.24441 (14)	0.70387 (3)	0.62913 (5)	0.03214 (17)	
O1A	0.009 (2)	0.4397 (10)	0.5799 (16)	0.041 (2)	0.583 (5)
O1B	−0.071 (4)	0.4474 (15)	0.576 (2)	0.041 (2)	0.417 (5)
O2	−0.9787 (6)	0.75162 (18)	−0.0143 (2)	0.0605 (7)	
O3	−1.1148 (6)	0.84877 (16)	0.0802 (2)	0.0551 (7)	
N1	−0.0687 (5)	0.64901 (14)	0.44225 (18)	0.0293 (5)	
N2	−0.9655 (6)	0.79235 (16)	0.0691 (2)	0.0385 (6)	
C1	0.0964 (6)	0.49027 (16)	0.5199 (2)	0.0338 (6)	
C2	0.1876 (6)	0.56264 (16)	0.5745 (2)	0.0280 (5)	
C3	0.3469 (6)	0.56491 (16)	0.6813 (2)	0.0303 (6)	
C4A	0.439 (6)	0.4943 (15)	0.7449 (16)	0.0392 (15)	0.583 (5)
H4AA	0.5141	0.4559	0.6915	0.047*	0.583 (5)
H4AB	0.2683	0.4731	0.7832	0.047*	0.583 (5)
C5A	0.6700 (13)	0.5148 (4)	0.8363 (5)	0.0438 (11)	0.583 (5)
H5AA	0.7031	0.4720	0.8895	0.053*	0.583 (5)
H5AB	0.8537	0.5248	0.7969	0.053*	0.583 (5)
C6A	0.5854 (15)	0.5850 (4)	0.9033 (5)	0.0428 (12)	0.583 (5)
H6AA	0.3961	0.5767	0.9394	0.051*	0.583 (5)
H6AB	0.7322	0.5949	0.9644	0.051*	0.583 (5)
C7A	0.578 (5)	0.6541 (8)	0.8218 (18)	0.035 (2)	0.583 (5)
H7AA	0.4883	0.6968	0.8628	0.042*	0.583 (5)
H7AB	0.7767	0.6695	0.8023	0.042*	0.583 (5)
C4B	0.464 (8)	0.502 (2)	0.754 (2)	0.0392 (15)	0.417 (5)
H4BA	0.6486	0.4861	0.7197	0.047*	0.417 (5)
H4BB	0.3324	0.4574	0.7522	0.047*	0.417 (5)
C5B	0.5365 (19)	0.5254 (5)	0.8763 (7)	0.0438 (11)	0.417 (5)
H5BA	0.3503	0.5333	0.9149	0.053*	0.417 (5)
H5BB	0.6429	0.4844	0.9169	0.053*	0.417 (5)

C6B	0.717 (2)	0.5966 (5)	0.8813 (8)	0.0428 (12)	0.417 (5)
H6BA	0.7592	0.6094	0.9623	0.051*	0.417 (5)
H6BB	0.9050	0.5874	0.8443	0.051*	0.417 (5)
C7B	0.551 (7)	0.6653 (12)	0.830 (3)	0.035 (2)	0.417 (5)
H7BA	0.6977	0.7031	0.8078	0.042*	0.417 (5)
H7BB	0.4169	0.6895	0.8834	0.042*	0.417 (5)
C8	0.3991 (6)	0.63650 (17)	0.7187 (2)	0.0310 (6)	
C9	0.1092 (6)	0.63358 (15)	0.5361 (2)	0.0276 (6)	
C10A	0.1914 (17)	0.4696 (6)	0.4023 (7)	0.0261 (15)	0.583 (5)
C11A	0.0684 (16)	0.4049 (6)	0.3543 (9)	0.0430 (17)	0.583 (5)
H11A	−0.0653	0.3758	0.3964	0.052*	0.583 (5)
C12A	0.1411 (17)	0.3828 (5)	0.2446 (9)	0.049 (2)	0.583 (5)
H12A	0.0571	0.3386	0.2118	0.059*	0.583 (5)
C13A	0.3367 (16)	0.4254 (6)	0.1830 (7)	0.050 (2)	0.583 (5)
H13A	0.3864	0.4103	0.1081	0.060*	0.583 (5)
C14A	0.4597 (16)	0.4901 (5)	0.2311 (9)	0.051 (2)	0.583 (5)
H14A	0.5934	0.5192	0.1889	0.061*	0.583 (5)
C15A	0.3870 (18)	0.5122 (5)	0.3407 (9)	0.0347 (17)	0.583 (5)
H15A	0.4711	0.5564	0.3735	0.042*	0.583 (5)
C10B	0.127 (3)	0.4756 (10)	0.3952 (11)	0.0261 (15)	0.417 (5)
C11B	−0.001 (3)	0.4119 (9)	0.3461 (13)	0.0430 (17)	0.417 (5)
H11B	−0.1300	0.3819	0.3890	0.052*	0.417 (5)
C12B	0.060 (3)	0.3922 (7)	0.2342 (14)	0.049 (2)	0.417 (5)
H12B	−0.0276	0.3487	0.2007	0.059*	0.417 (5)
C13B	0.249 (3)	0.4360 (9)	0.1715 (11)	0.050 (2)	0.417 (5)
H13B	0.2906	0.4225	0.0950	0.060*	0.417 (5)
C14B	0.377 (3)	0.4997 (8)	0.2206 (13)	0.051 (2)	0.417 (5)
H14B	0.5065	0.5297	0.1777	0.061*	0.417 (5)
C15B	0.316 (3)	0.5194 (8)	0.3324 (14)	0.0347 (17)	0.417 (5)
H15B	0.4041	0.5629	0.3660	0.042*	0.417 (5)
C16	−0.1906 (5)	0.71396 (17)	0.4340 (2)	0.0295 (6)	
H16	−0.1542	0.7504	0.4921	0.035*	
C17	−0.3839 (5)	0.73363 (15)	0.3379 (2)	0.0277 (5)	
C18	−0.4040 (6)	0.68891 (15)	0.2402 (2)	0.0319 (6)	
H18	−0.2870	0.6449	0.2344	0.038*	
C19	−0.5926 (6)	0.70787 (19)	0.1514 (2)	0.0339 (6)	
H19	−0.6052	0.6778	0.0841	0.041*	
C20	−0.7634 (6)	0.77200 (16)	0.1630 (2)	0.0309 (6)	
C21	−0.7499 (6)	0.81762 (16)	0.2582 (2)	0.0310 (6)	
H21	−0.8692	0.8612	0.2637	0.037*	
C22	−0.5568 (6)	0.79807 (16)	0.3461 (2)	0.0316 (6)	
H22	−0.5423	0.8289	0.4124	0.038*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0408 (3)	0.0258 (3)	0.0297 (3)	0.0003 (3)	−0.0032 (2)	−0.0018 (3)
O1A	0.045 (6)	0.033 (4)	0.0448 (18)	−0.007 (5)	0.011 (5)	0.004 (2)

O1B	0.045 (6)	0.033 (4)	0.0448 (18)	−0.007 (5)	0.011 (5)	0.004 (2)
O2	0.0743 (17)	0.0657 (18)	0.0406 (12)	0.0136 (14)	−0.0202 (11)	−0.0143 (12)
O3	0.0636 (15)	0.0471 (14)	0.0538 (14)	0.0174 (12)	−0.0183 (12)	0.0023 (11)
N1	0.0326 (11)	0.0294 (12)	0.0260 (11)	−0.0036 (10)	0.0002 (8)	0.0008 (9)
N2	0.0416 (13)	0.0419 (15)	0.0318 (12)	−0.0038 (12)	−0.0050 (10)	0.0034 (11)
C1	0.0416 (16)	0.0271 (15)	0.0328 (14)	−0.0008 (13)	0.0061 (12)	0.0022 (12)
C2	0.0332 (13)	0.0263 (14)	0.0248 (12)	−0.0002 (10)	0.0061 (10)	0.0016 (10)
C3	0.0331 (13)	0.0318 (15)	0.0262 (12)	0.0040 (11)	0.0050 (10)	0.0013 (11)
C4A	0.047 (4)	0.035 (4)	0.036 (3)	0.009 (3)	0.002 (2)	0.004 (2)
C5A	0.044 (3)	0.052 (3)	0.034 (3)	0.013 (2)	−0.0027 (18)	0.005 (2)
C6A	0.040 (3)	0.054 (3)	0.034 (2)	0.005 (3)	−0.005 (2)	0.002 (2)
C7A	0.035 (4)	0.041 (4)	0.028 (3)	0.006 (3)	0.000 (2)	−0.007 (4)
C4B	0.047 (4)	0.035 (4)	0.036 (3)	0.009 (3)	0.002 (2)	0.004 (2)
C5B	0.044 (3)	0.052 (3)	0.034 (3)	0.013 (2)	−0.0027 (18)	0.005 (2)
C6B	0.040 (3)	0.054 (3)	0.034 (2)	0.005 (3)	−0.005 (2)	0.002 (2)
C7B	0.035 (4)	0.041 (4)	0.028 (3)	0.006 (3)	0.000 (2)	−0.007 (4)
C8	0.0310 (13)	0.0346 (16)	0.0274 (13)	0.0042 (11)	0.0025 (10)	0.0014 (11)
C9	0.0313 (13)	0.0247 (14)	0.0270 (13)	−0.0019 (10)	0.0035 (10)	−0.0015 (10)
C10A	0.021 (4)	0.025 (2)	0.0314 (16)	0.006 (3)	−0.008 (2)	0.0031 (15)
C11A	0.046 (4)	0.034 (3)	0.048 (2)	−0.008 (3)	0.000 (3)	−0.0066 (18)
C12A	0.057 (5)	0.040 (3)	0.050 (3)	−0.005 (3)	−0.010 (3)	−0.014 (2)
C13A	0.066 (5)	0.051 (4)	0.035 (2)	0.001 (4)	0.001 (3)	−0.016 (2)
C14A	0.065 (5)	0.047 (3)	0.041 (3)	−0.014 (4)	0.016 (3)	−0.011 (2)
C15A	0.036 (5)	0.033 (2)	0.036 (2)	−0.006 (3)	0.001 (3)	−0.0046 (18)
C10B	0.021 (4)	0.025 (2)	0.0314 (16)	0.006 (3)	−0.008 (2)	0.0031 (15)
C11B	0.046 (4)	0.034 (3)	0.048 (2)	−0.008 (3)	0.000 (3)	−0.0066 (18)
C12B	0.057 (5)	0.040 (3)	0.050 (3)	−0.005 (3)	−0.010 (3)	−0.014 (2)
C13B	0.066 (5)	0.051 (4)	0.035 (2)	0.001 (4)	0.001 (3)	−0.016 (2)
C14B	0.065 (5)	0.047 (3)	0.041 (3)	−0.014 (4)	0.016 (3)	−0.011 (2)
C15B	0.036 (5)	0.033 (2)	0.036 (2)	−0.006 (3)	0.001 (3)	−0.0046 (18)
C16	0.0319 (12)	0.0288 (15)	0.0279 (11)	−0.0029 (11)	0.0007 (9)	−0.0002 (11)
C17	0.0279 (12)	0.0257 (13)	0.0295 (12)	−0.0045 (10)	0.0009 (10)	0.0043 (10)
C18	0.0368 (13)	0.0284 (16)	0.0308 (13)	0.0034 (11)	0.0061 (10)	−0.0007 (10)
C19	0.0421 (14)	0.0324 (14)	0.0270 (11)	0.0005 (14)	0.0011 (10)	−0.0064 (13)
C20	0.0313 (13)	0.0319 (15)	0.0296 (12)	−0.0043 (11)	−0.0004 (10)	0.0039 (11)
C21	0.0332 (13)	0.0268 (13)	0.0330 (14)	−0.0008 (11)	0.0002 (10)	0.0016 (11)
C22	0.0375 (14)	0.0282 (14)	0.0292 (12)	−0.0039 (11)	−0.0001 (10)	−0.0031 (11)

Geometric parameters (Å, º)

S1—C8	1.730 (3)	C7B—H7BA	0.9902
S1—C9	1.758 (3)	C7B—H7BB	0.9900
O1A—C1	1.213 (18)	C7B—C8	1.55 (4)
O1B—C1	1.27 (3)	C10A—C11A	1.3900
O2—N2	1.214 (4)	C10A—C15A	1.3900
O3—N2	1.221 (4)	C11A—H11A	0.9500
N1—C9	1.384 (3)	C11A—C12A	1.3900
N1—C16	1.282 (4)	C12A—H12A	0.9500

N2—C20	1.470 (3)	C12A—C13A	1.3900
C1—C2	1.487 (4)	C13A—H13A	0.9500
C1—C10A	1.499 (9)	C13A—C14A	1.3900
C1—C10B	1.493 (13)	C14A—H14A	0.9500
C2—C3	1.437 (4)	C14A—C15A	1.3900
C2—C9	1.378 (4)	C15A—H15A	0.9500
C3—C4A	1.51 (3)	C10B—C11B	1.3900
C3—C4B	1.50 (4)	C10B—C15B	1.3900
C3—C8	1.359 (4)	C11B—H11B	0.9500
C4A—H4AA	0.9898	C11B—C12B	1.3900
C4A—H4AB	0.9899	C12B—H12B	0.9500
C4A—C5A	1.537 (14)	C12B—C13B	1.3900
C5A—H5AA	0.9902	C13B—H13B	0.9500
C5A—H5AB	0.9899	C13B—C14B	1.3900
C5A—C6A	1.523 (9)	C14B—H14B	0.9500
C6A—H6AA	0.9900	C14B—C15B	1.3900
C6A—H6AB	0.9900	C15B—H15B	0.9500
C6A—C7A	1.550 (13)	C16—H16	0.9500
C7A—H7AA	0.9900	C16—C17	1.462 (3)
C7A—H7AB	0.9899	C17—C18	1.393 (4)
C7A—C8	1.48 (2)	C17—C22	1.396 (4)
C4B—H4BA	0.9900	C18—H18	0.9500
C4B—H4BB	0.9901	C18—C19	1.382 (4)
C4B—C5B	1.524 (17)	C19—H19	0.9500
C5B—H5BA	0.9900	C19—C20	1.389 (4)
C5B—H5BB	0.9899	C20—C21	1.377 (4)
C5B—C6B	1.511 (12)	C21—H21	0.9500
C6B—H6BA	0.9900	C21—C22	1.390 (4)
C6B—H6BB	0.9900	C22—H22	0.9500
C6B—C7B	1.553 (17)		
C8—S1—C9	91.40 (14)	C8—C7B—H7BB	113.4
C16—N1—C9	119.2 (2)	C3—C8—S1	112.3 (2)
O2—N2—O3	123.5 (3)	C3—C8—C7A	123.4 (5)
O2—N2—C20	118.4 (3)	C3—C8—C7B	130.5 (6)
O3—N2—C20	118.1 (2)	C7A—C8—S1	124.3 (5)
O1A—C1—C2	118.8 (9)	C7B—C8—S1	117.1 (6)
O1A—C1—C10A	117.4 (10)	N1—C9—S1	123.3 (2)
O1B—C1—C2	117.5 (13)	C2—C9—S1	110.8 (2)
O1B—C1—C10B	118.0 (15)	C2—C9—N1	125.8 (2)
C2—C1—C10A	121.2 (5)	C11A—C10A—C1	116.5 (7)
C2—C1—C10B	122.5 (7)	C11A—C10A—C15A	120.0
C3—C2—C1	122.2 (2)	C15A—C10A—C1	123.5 (7)
C9—C2—C1	125.0 (2)	C10A—C11A—H11A	120.0
C9—C2—C3	112.6 (2)	C12A—C11A—C10A	120.0
C2—C3—C4A	122.6 (5)	C12A—C11A—H11A	120.0
C2—C3—C4B	130.1 (8)	C11A—C12A—H12A	120.0
C8—C3—C2	112.9 (2)	C11A—C12A—C13A	120.0

C8—C3—C4A	124.5 (5)	C13A—C12A—H12A	120.0
C8—C3—C4B	117.0 (8)	C12A—C13A—H13A	120.0
C3—C4A—H4AA	110.8	C14A—C13A—C12A	120.0
C3—C4A—H4AB	108.5	C14A—C13A—H13A	120.0
C3—C4A—C5A	109.3 (14)	C13A—C14A—H14A	120.0
H4AA—C4A—H4AB	108.6	C15A—C14A—C13A	120.0
C5A—C4A—H4AA	110.8	C15A—C14A—H14A	120.0
C5A—C4A—H4AB	108.7	C10A—C15A—H15A	120.0
C4A—C5A—H5AA	110.6	C14A—C15A—C10A	120.0
C4A—C5A—H5AB	107.8	C14A—C15A—H15A	120.0
H5AA—C5A—H5AB	107.9	C11B—C10B—C1	119.7 (11)
C6A—C5A—C4A	111.7 (11)	C11B—C10B—C15B	120.0
C6A—C5A—H5AA	109.7	C15B—C10B—C1	119.6 (10)
C6A—C5A—H5AB	109.0	C10B—C11B—H11B	120.0
C5A—C6A—H6AA	109.7	C12B—C11B—C10B	120.0
C5A—C6A—H6AB	109.7	C12B—C11B—H11B	120.0
C5A—C6A—C7A	109.1 (9)	C11B—C12B—H12B	120.0
H6AA—C6A—H6AB	108.3	C13B—C12B—C11B	120.0
C7A—C6A—H6AA	111.8	C13B—C12B—H12B	120.0
C7A—C6A—H6AB	108.2	C12B—C13B—H13B	120.0
C6A—C7A—H7AA	107.9	C14B—C13B—C12B	120.0
C6A—C7A—H7AB	110.4	C14B—C13B—H13B	120.0
H7AA—C7A—H7AB	107.5	C13B—C14B—H14B	120.0
C8—C7A—C6A	110.0 (13)	C13B—C14B—C15B	120.0
C8—C7A—H7AA	109.1	C15B—C14B—H14B	120.0
C8—C7A—H7AB	111.9	C10B—C15B—H15B	120.0
C3—C4B—H4BA	106.4	C14B—C15B—C10B	120.0
C3—C4B—H4BB	111.5	C14B—C15B—H15B	120.0
C3—C4B—C5B	113 (2)	N1—C16—H16	119.0
H4BA—C4B—H4BB	107.7	N1—C16—C17	122.0 (3)
C5B—C4B—H4BA	106.4	C17—C16—H16	119.0
C5B—C4B—H4BB	111.0	C18—C17—C16	121.7 (2)
C4B—C5B—H5BA	107.1	C18—C17—C22	119.5 (2)
C4B—C5B—H5BB	110.3	C22—C17—C16	118.8 (2)
H5BA—C5B—H5BB	108.2	C17—C18—H18	119.7
C6B—C5B—C4B	112.0 (16)	C19—C18—C17	120.6 (3)
C6B—C5B—H5BA	110.4	C19—C18—H18	119.7
C6B—C5B—H5BB	108.8	C18—C19—H19	120.9
C5B—C6B—H6BA	108.9	C18—C19—C20	118.3 (2)
C5B—C6B—H6BB	109.6	C20—C19—H19	120.9
C5B—C6B—C7B	111.6 (15)	C19—C20—N2	118.6 (2)
H6BA—C6B—H6BB	107.7	C21—C20—N2	118.6 (3)
C7B—C6B—H6BA	106.1	C21—C20—C19	122.8 (2)
C7B—C6B—H6BB	112.8	C20—C21—H21	121.0
C6B—C7B—H7BA	107.1	C20—C21—C22	118.1 (3)
C6B—C7B—H7BB	113.8	C22—C21—H21	121.0
H7BA—C7B—H7BB	108.2	C17—C22—H22	119.6
C8—C7B—C6B	106.3 (17)	C21—C22—C17	120.7 (3)

C8—C7B—H7BA	107.7	C21—C22—H22	119.6
O1A—C1—C2—C3	40.0 (6)	C4B—C3—C8—S1	−178.4 (18)
O1A—C1—C2—C9	−133.7 (5)	C4B—C3—C8—C7A	4 (2)
O1A—C1—C10A—C11A	25.5 (8)	C4B—C3—C8—C7B	−3 (2)
O1A—C1—C10A—C15A	−155.8 (7)	C4B—C5B—C6B—C7B	63 (2)
O1A—C1—C10B—C11B	14.0 (11)	C5B—C6B—C7B—C8	−43 (2)
O1A—C1—C10B—C15B	−157.1 (8)	C6B—C7B—C8—S1	−169.6 (11)
O1B—C1—C2—C3	60.5 (10)	C6B—C7B—C8—C3	16 (3)
O1B—C1—C2—C9	−113.2 (10)	C6B—C7B—C8—C7A	−25 (10)
O1B—C1—C10A—C11A	5.2 (11)	C8—S1—C9—N1	174.9 (2)
O1B—C1—C10A—C15A	−176.1 (10)	C8—S1—C9—C2	−0.8 (2)
O1B—C1—C10B—C11B	−6.6 (13)	C8—C3—C4A—C5A	−16 (2)
O1B—C1—C10B—C15B	−177.7 (11)	C8—C3—C4B—C5B	19 (3)
O2—N2—C20—C19	1.1 (4)	C9—S1—C8—C3	−1.1 (2)
O2—N2—C20—C21	−179.3 (3)	C9—S1—C8—C7A	176.7 (11)
O3—N2—C20—C19	179.9 (3)	C9—S1—C8—C7B	−176.8 (14)
O3—N2—C20—C21	−0.5 (4)	C9—N1—C16—C17	−179.2 (2)
N1—C16—C17—C18	−12.3 (4)	C9—C2—C3—C4A	175.7 (13)
N1—C16—C17—C22	166.3 (3)	C9—C2—C3—C4B	178 (2)
N2—C20—C21—C22	−179.6 (2)	C9—C2—C3—C8	−3.2 (3)
C1—C2—C3—C4A	1.3 (14)	C10A—C1—C2—C3	−121.2 (4)
C1—C2—C3—C4B	4 (2)	C10A—C1—C2—C9	65.1 (5)
C1—C2—C3—C8	−177.6 (3)	C10A—C1—C10B—C11B	102 (5)
C1—C2—C9—S1	176.5 (2)	C10A—C1—C10B—C15B	−69 (5)
C1—C2—C9—N1	1.0 (4)	C10A—C11A—C12A—C13A	0.0
C1—C10A—C11A—C12A	178.7 (6)	C11A—C10A—C15A—C14A	0.0
C1—C10A—C15A—C14A	−178.7 (7)	C11A—C12A—C13A—C14A	0.0
C1—C10B—C11B—C12B	−171.1 (11)	C12A—C13A—C14A—C15A	0.0
C1—C10B—C15B—C14B	171.1 (11)	C13A—C14A—C15A—C10A	0.0
C2—C1—C10A—C11A	−173.1 (3)	C15A—C10A—C11A—C12A	0.0
C2—C1—C10A—C15A	5.6 (7)	C10B—C1—C2—C3	−135.7 (7)
C2—C1—C10B—C11B	−170.3 (5)	C10B—C1—C2—C9	50.6 (7)
C2—C1—C10B—C15B	18.6 (11)	C10B—C1—C10A—C11A	−73 (5)
C2—C3—C4A—C5A	164.9 (8)	C10B—C1—C10A—C15A	105 (5)
C2—C3—C4B—C5B	−162.1 (11)	C10B—C11B—C12B—C13B	0.0
C2—C3—C8—S1	2.6 (3)	C11B—C10B—C15B—C14B	0.0
C2—C3—C8—C7A	−175.2 (11)	C11B—C12B—C13B—C14B	0.0
C2—C3—C8—C7B	177.6 (17)	C12B—C13B—C14B—C15B	0.0
C3—C2—C9—S1	2.3 (3)	C13B—C14B—C15B—C10B	0.0
C3—C2—C9—N1	−173.3 (2)	C15B—C10B—C11B—C12B	0.0
C3—C4A—C5A—C6A	45.5 (19)	C16—N1—C9—S1	−14.8 (4)
C3—C4B—C5B—C6B	−49 (3)	C16—N1—C9—C2	160.3 (3)
C4A—C3—C4B—C5B	−148 (21)	C16—C17—C18—C19	178.7 (2)
C4A—C3—C8—S1	−176.3 (13)	C16—C17—C22—C21	−178.1 (2)
C4A—C3—C8—C7A	5.9 (17)	C17—C18—C19—C20	−0.8 (4)
C4A—C3—C8—C7B	−1 (2)	C18—C17—C22—C21	0.5 (4)
C4A—C5A—C6A—C7A	−65.1 (16)	C18—C19—C20—N2	−179.7 (2)

C5A—C6A—C7A—C8	51.0 (15)	C18—C19—C20—C21	0.8 (4)
C6A—C7A—C8—S1	159.4 (6)	C19—C20—C21—C22	−0.1 (4)
C6A—C7A—C8—C3	−23.0 (18)	C20—C21—C22—C17	−0.6 (4)
C6A—C7A—C8—C7B	120 (13)	C22—C17—C18—C19	0.2 (4)
C4B—C3—C4A—C5A	−2 (18)		

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C4*A*—H4*AA*⋯O1*B*[i]	0.99	2.38	3.15 (4)	135
C4*B*—H4*BA*⋯O1*A*[i]	0.99	2.50	3.45 (4)	162
C4*B*—H4*BA*⋯O1*B*[i]	0.99	2.26	3.18 (4)	154
C7*B*—H7*BB*⋯O2[ii]	0.99	2.46	3.44 (3)	169
C13*B*—H13*B*⋯O3[iii]	0.95	2.55	3.371 (13)	145
C21—H21⋯O1*A*[iv]	0.95	2.40	3.127 (18)	133
C21—H21⋯O1*B*[iv]	0.95	2.44	3.13 (3)	129

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