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ISSN 2414-3146

7-Acetyl-8-(4-chlorophenyl)-3-ethylsulfanyl-6-hydroxy-1,6-dimethyl-5,6,7,8-tetrahydroisoquinoline-4-carbonitrile

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Received 8 March 2017
Accepted 10 March 2017

Edited by J. Simpson, University of Otago, New Zealand

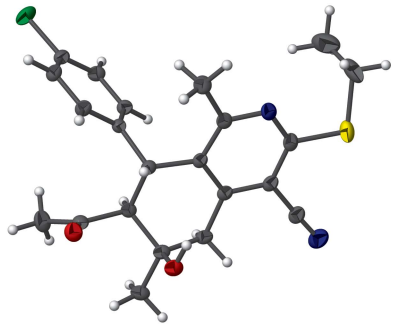
Keywords: crystal structure; isoquinolines; hydrogen bonds; inversion dimers.

CCDC reference: 1537099

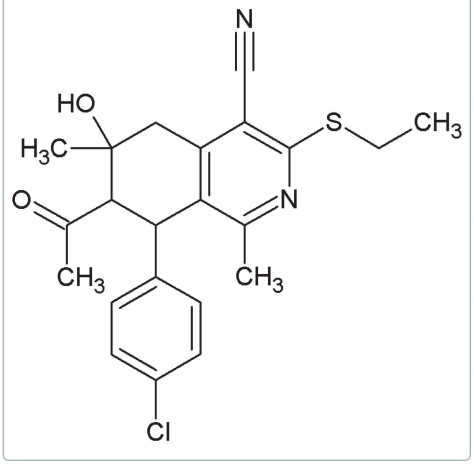
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{22}H_{23}ClN_2O_2S$, the chlorophenyl ring is inclined to the pyridine ring of the isoquinoline ring system by 79.78 (4)°. The cyclohexane ring adopts a flattened boat conformation. In the crystal, dimers form through complementary sets of inversion-related O—H···O and C—H···O hydrogen bonds. These are connected into zigzag chains along the *c*-axis direction by pairwise C—H···N interactions that also form inversion dimers.

3D view



Chemical scheme



Structure description

It is well known that partially hydrogenated isoquinoline derivatives exhibit antifungal activity by inhibition of the enzymes in sterol biosynthesis (Krauss *et al.*, 2014; Zhu *et al.*, 2006). The influence of substitution of the aromatic rings of tetrahydroisoquinolines (THIQ) on their anti-fungal activities has also been reported (Bojarski *et al.*, 2002). In the light of such findings and as part of our studies in this area, we report herein the synthesis and crystal structure of the title compound.

In the title molecule (Fig. 1), the dihedral angle between the 4-chlorophenyl ring and the pyridine ring of the tetrahydroisoquinoline ring system is 79.78 (4)°. A puckering analysis of the C1–C6 ring yielded the parameters: Q = 0.521 (2) Å, θ = 52.8 (2)° and φ = 37.5 (2)° and the substituted cyclohexane ring can best be described as adopting a flattened boat conformation. In the crystal, O1 acts as a bifurcated acceptor, forming C5—H5···O1^{i} and C14—H14···O1^{i} inversion dimers, Table 1, that enclose $R_2^1(6)$ rings. Classical O1—H1···O2^{i} hydrogen bonds strongly reinforce these dimers and generate

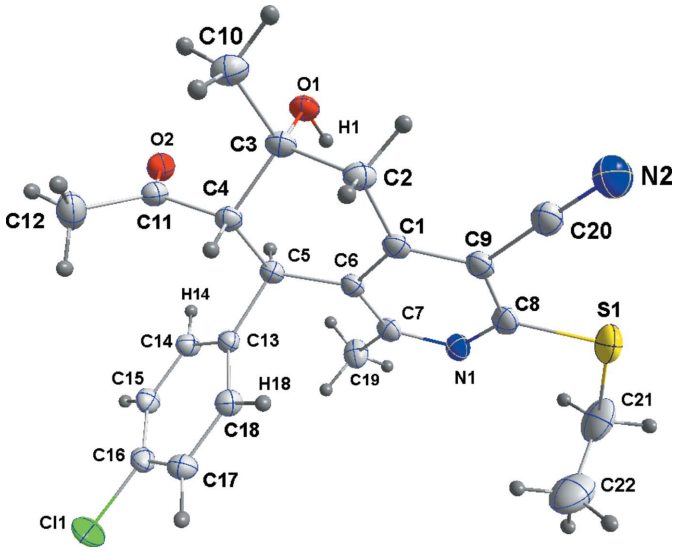


Figure 1
The title molecule with the labeling scheme and 50% probability ellipsoids.

$R_2^2(12)$ rings. These pairs of molecules are connected into zigzag chains along the *c* axis by inversion-related C18—H18···N2^{ii} interactions, Figs. 2 and 3. These chains stack to form layers parallel to (110).

Synthesis and crystallization

A mixture of 7-acetyl-8-(4-chlorophenyl)-1,6-dimethyl-6-hydroxy-3-thioxo-2,3,5,6,7,8-hexahydroisoquinoline-4-carbonitrile (10 mmol), ethyl iodide (10 mmol) and sodium acetate trihydrate (11 mmol) in ethanol (30 ml) was heated under reflux for 1 h. The precipitate that formed after cooling was

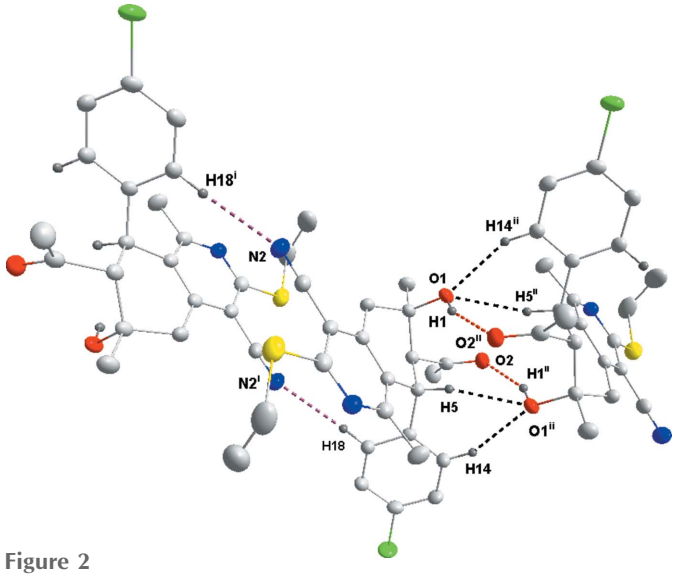


Figure 2
Detail of the intermolecular interactions (O—H···O, C—H···O and C—H···N hydrogen bonds are shown, respectively as red, black and purple dashed lines). [Symmetry codes: (i) $1-x, 2-y, -z$; (ii) $1-x, 2-y, 1-z$].

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.87 (2)	2.06 (2)	2.8655 (15)	155 (2)
C5—H5···O1^{i}	0.98 (2)	2.49 (2)	3.4276 (16)	160 (1)
C14—H14···O1^{i}	0.97 (2)	2.46 (2)	3.3523 (17)	154 (2)
C18—H18···N2^{ii}	0.98 (2)	2.56 (2)	3.538 (2)	174 (2)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{23}ClN_2O_2S$
M_r	414.93
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, *b*, *c* (Å)	9.4083 (3), 9.5140 (3), 12.7168 (4)
α, β, γ (°)	86.863 (1), 79.175 (1), 71.222 (1)
V (Å^3)	1058.50 (6)
Z	2
Radiation type	Cu *K*α
μ (mm^{-1})	2.68
Crystal size (mm)	0.27 × 0.09 × 0.07
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.71, 0.84
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8327, 3971, 3653
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.035, 0.096, 1.03
No. of reflections	3971
No. of parameters	345
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.38

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL 2014/7* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

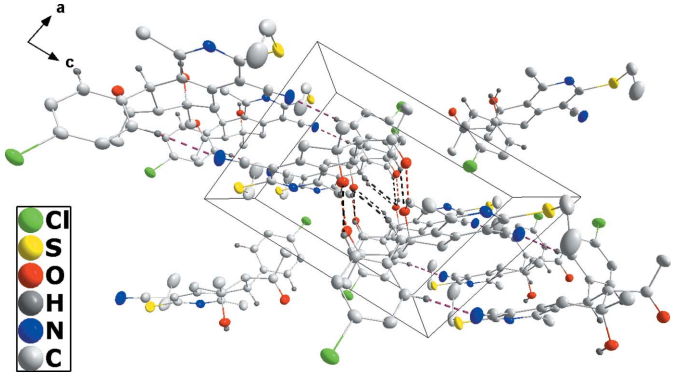


Figure 3
Packing viewed along the *b* axis (color code for hydrogen bonds is given in Fig. 2).

collected and recrystallized from ethanol in the form of colorless needles. Yield: 80%, m.p.: 450 K. IR: 3420 (OH), 2225 (CN), 1710 (CO) cm^{-1}. ^1H NMR ($CDCl_3$): δ 6.8–7.3 (*dd*, 4H, Ar—H), 4.2–4.4 (*d*, 1H, CH at C-7), 2.8–3.2 (*m*, 5H: SCH_2, CH at C-8 and CH_2 of cyclohexanone ring), 1H, CH at C-7), 2.0 (*s*, 3H, $COCH_3$), 1.8 (*s*, 3H, CH_3 at C-1), 1.2–1.5 (*m*, 6H, CH_3 at C-6 and CH_3 of the ethylsulfanyl group).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The support of NSF–MRI Grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

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full crystallographic data

IUCrData (2017). **2**, x170390 [https://doi.org/10.1107/S241431461700390X]

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Crystal data

$C_{22}H_{23}ClN_2O_2S$
M_r = 414.93
Triclinic, $P\overline{1}$
a = 9.4083 (3) Å
b = 9.5140 (3) Å
c = 12.7168 (4) Å
α = 86.863 (1)°
β = 79.175 (1)°
γ = 71.222 (1)°
V = 1058.50 (6) Å^3
Z = 2
$F(000)$ = 436
D_x = 1.302 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 7163 reflections
θ = 3.5–72.5°
μ = 2.68 mm^{-1}
T = 150 K
Needle, colourless
0.27 × 0.09 × 0.07 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.71, T_{max} = 0.84
8327 measured reflections
3971 independent reflections
3653 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 72.5°, θ_{min} = 3.5°
h = −11→11
k = −10→11
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.03
3971 reflections
345 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.319P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 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.

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}
Cl1	1.09551 (4)	0.23534 (4)	0.31423 (3)	0.03778 (13)
S1	0.14426 (4)	0.89211 (5)	0.06323 (3)	0.03559 (13)
O1	0.48196 (11)	1.16451 (11)	0.38291 (8)	0.0247 (2)
H1	0.425 (3)	1.108 (3)	0.4007 (18)	0.052 (6)*
O2	0.72775 (12)	0.97289 (12)	0.49818 (8)	0.0281 (2)
N1	0.30224 (13)	0.76948 (14)	0.21841 (10)	0.0254 (3)
N2	0.27102 (19)	1.20457 (16)	0.00856 (12)	0.0403 (3)
C1	0.45908 (15)	0.97740 (14)	0.20289 (10)	0.0203 (3)
C2	0.53508 (17)	1.09605 (15)	0.19497 (11)	0.0234 (3)
H2A	0.615 (2)	1.0794 (19)	0.1321 (14)	0.026 (4)*
H2B	0.458 (2)	1.196 (2)	0.1858 (14)	0.028 (4)*
C3	0.60142 (15)	1.10524 (15)	0.29429 (11)	0.0216 (3)
C4	0.70289 (15)	0.94682 (15)	0.31644 (11)	0.0204 (3)
H4	0.780 (2)	0.9114 (18)	0.2514 (14)	0.024 (4)*
C5	0.61087 (15)	0.83658 (14)	0.34326 (10)	0.0194 (3)
H5	0.5601 (19)	0.8533 (18)	0.4182 (13)	0.021 (4)*
C6	0.49137 (15)	0.85821 (14)	0.27322 (10)	0.0195 (3)
C7	0.40342 (15)	0.76146 (15)	0.28225 (11)	0.0224 (3)
C8	0.27810 (15)	0.87785 (16)	0.14645 (11)	0.0246 (3)
C9	0.34990 (15)	0.98684 (15)	0.13912 (11)	0.0227 (3)
C10	0.69246 (18)	1.21369 (17)	0.27516 (13)	0.0289 (3)
H10A	0.725 (2)	1.230 (2)	0.3415 (15)	0.032 (5)*
H10B	0.783 (2)	1.180 (2)	0.2178 (16)	0.038 (5)*
H10C	0.628 (2)	1.311 (2)	0.2543 (16)	0.038 (5)*
C11	0.79209 (16)	0.94474 (15)	0.40579 (11)	0.0227 (3)
C12	0.96168 (18)	0.9034 (2)	0.37436 (15)	0.0350 (4)
H12A	1.006 (3)	0.806 (3)	0.3426 (19)	0.054 (6)*
H12B	1.006 (3)	0.907 (3)	0.4372 (19)	0.055 (6)*
H12C	0.988 (3)	0.967 (2)	0.3204 (19)	0.048 (6)*
C13	0.72613 (14)	0.68024 (14)	0.33571 (11)	0.0191 (3)
C14	0.76798 (16)	0.60575 (15)	0.42788 (11)	0.0235 (3)
H14	0.715 (2)	0.649 (2)	0.4969 (16)	0.036 (5)*
C15	0.88127 (16)	0.46802 (16)	0.42125 (12)	0.0249 (3)
H15	0.911 (2)	0.416 (2)	0.4836 (15)	0.032 (5)*
C16	0.95203 (15)	0.40606 (15)	0.32158 (12)	0.0245 (3)

C17	0.91100 (16)	0.47659 (16)	0.22861 (12)	0.0248 (3)
H17	0.958 (2)	0.430 (2)	0.1584 (17)	0.043 (5)*
C18	0.79745 (16)	0.61425 (15)	0.23609 (11)	0.0224 (3)
H18	0.770 (2)	0.667 (2)	0.1709 (15)	0.030 (4)*
C19	0.41091 (18)	0.64464 (18)	0.36753 (14)	0.0309 (3)
H19A	0.414 (3)	0.679 (2)	0.4372 (19)	0.050 (6)*
H19B	0.506 (3)	0.560 (3)	0.3502 (18)	0.051 (6)*
H19C	0.326 (3)	0.612 (2)	0.3722 (17)	0.049 (6)*
C20	0.30823 (17)	1.10873 (16)	0.06672 (11)	0.0274 (3)
C21	0.1198 (2)	0.7110 (3)	0.07781 (16)	0.0475 (5)
H21A	0.029 (3)	0.723 (3)	0.048 (2)	0.065 (7)*
H21B	0.094 (3)	0.694 (3)	0.158 (2)	0.054 (6)*
C22	0.2547 (4)	0.5899 (3)	0.0220 (2)	0.0628 (6)
H22A	0.236 (3)	0.493 (3)	0.034 (2)	0.082 (9)*
H22B	0.284 (3)	0.607 (3)	−0.058 (2)	0.076 (8)*
H22C	0.350 (3)	0.578 (3)	0.051 (2)	0.068 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0316 (2)	0.02441 (19)	0.0462 (2)	0.00513 (14)	−0.00492 (17)	0.00079 (16)
S1	0.0288 (2)	0.0465 (2)	0.0356 (2)	−0.01295 (17)	−0.01517 (17)	0.00370 (18)
O1	0.0250 (5)	0.0217 (5)	0.0239 (5)	−0.0043 (4)	0.0005 (4)	−0.0053 (4)
O2	0.0294 (5)	0.0314 (5)	0.0234 (5)	−0.0092 (4)	−0.0051 (4)	−0.0018 (4)
N1	0.0202 (6)	0.0273 (6)	0.0290 (6)	−0.0079 (5)	−0.0046 (5)	0.0008 (5)
N2	0.0543 (9)	0.0340 (7)	0.0313 (7)	−0.0082 (7)	−0.0166 (7)	0.0053 (6)
C1	0.0203 (6)	0.0184 (6)	0.0187 (6)	−0.0027 (5)	−0.0007 (5)	−0.0024 (5)
C2	0.0300 (7)	0.0186 (6)	0.0213 (6)	−0.0075 (6)	−0.0044 (6)	0.0020 (5)
C3	0.0236 (6)	0.0185 (6)	0.0206 (6)	−0.0058 (5)	−0.0004 (5)	−0.0012 (5)
C4	0.0202 (6)	0.0196 (6)	0.0202 (6)	−0.0060 (5)	−0.0012 (5)	−0.0014 (5)
C5	0.0196 (6)	0.0176 (6)	0.0198 (6)	−0.0046 (5)	−0.0027 (5)	−0.0002 (5)
C6	0.0181 (6)	0.0181 (6)	0.0195 (6)	−0.0027 (5)	−0.0019 (5)	−0.0013 (5)
C7	0.0185 (6)	0.0223 (6)	0.0243 (6)	−0.0049 (5)	−0.0016 (5)	−0.0004 (5)
C8	0.0182 (6)	0.0283 (7)	0.0246 (7)	−0.0037 (5)	−0.0030 (5)	−0.0022 (6)
C9	0.0226 (6)	0.0206 (6)	0.0207 (6)	−0.0017 (5)	−0.0029 (5)	−0.0008 (5)
C10	0.0333 (8)	0.0234 (7)	0.0328 (8)	−0.0134 (6)	−0.0046 (7)	0.0007 (6)
C11	0.0236 (7)	0.0191 (6)	0.0257 (7)	−0.0072 (5)	−0.0042 (6)	−0.0006 (5)
C12	0.0235 (7)	0.0447 (10)	0.0369 (9)	−0.0100 (7)	−0.0053 (7)	−0.0047 (8)
C13	0.0178 (6)	0.0181 (6)	0.0219 (6)	−0.0060 (5)	−0.0040 (5)	0.0003 (5)
C14	0.0258 (7)	0.0218 (6)	0.0215 (7)	−0.0053 (5)	−0.0047 (6)	0.0001 (5)
C15	0.0257 (7)	0.0230 (7)	0.0254 (7)	−0.0056 (5)	−0.0082 (6)	0.0043 (6)
C16	0.0206 (6)	0.0190 (6)	0.0329 (7)	−0.0050 (5)	−0.0044 (6)	0.0003 (6)
C17	0.0257 (7)	0.0224 (7)	0.0241 (7)	−0.0065 (5)	0.0001 (6)	−0.0024 (6)
C18	0.0247 (7)	0.0213 (6)	0.0210 (6)	−0.0071 (5)	−0.0039 (5)	0.0004 (5)
C19	0.0289 (8)	0.0312 (8)	0.0369 (8)	−0.0154 (7)	−0.0088 (7)	0.0104 (7)
C20	0.0306 (7)	0.0264 (7)	0.0228 (7)	−0.0041 (6)	−0.0076 (6)	−0.0004 (6)
C21	0.0533 (11)	0.0673 (13)	0.0416 (10)	−0.0419 (10)	−0.0179 (9)	0.0072 (9)
C22	0.100 (2)	0.0483 (12)	0.0535 (13)	−0.0358 (13)	−0.0245 (13)	−0.0011 (10)

Geometric parameters (Å, º)

Cl1—C16	1.7398 (14)	C10—H10A	0.987 (19)
S1—C8	1.7603 (14)	C10—H10B	0.99 (2)
S1—C21	1.805 (2)	C10—H10C	0.98 (2)
O1—C3	1.4273 (16)	C11—C12	1.495 (2)
O1—H1	0.87 (2)	C12—H12A	0.96 (2)
O2—C11	1.2171 (17)	C12—H12B	0.97 (2)
N1—C8	1.3327 (19)	C12—H12C	0.94 (2)
N1—C7	1.3445 (19)	C13—C14	1.3906 (19)
N2—C20	1.148 (2)	C13—C18	1.3950 (19)
C1—C6	1.3955 (19)	C14—C15	1.393 (2)
C1—C9	1.402 (2)	C14—H14	0.96 (2)
C1—C2	1.5086 (18)	C15—C16	1.382 (2)
C2—C3	1.5283 (19)	C15—H15	0.950 (19)
C2—H2A	0.968 (18)	C16—C17	1.384 (2)
C2—H2B	1.014 (18)	C17—C18	1.393 (2)
C3—C10	1.5234 (19)	C17—H17	0.98 (2)
C3—C4	1.5461 (18)	C18—H18	0.979 (19)
C4—C11	1.5300 (19)	C19—H19A	0.97 (2)
C4—C5	1.5493 (17)	C19—H19B	0.99 (2)
C4—H4	0.987 (18)	C19—H19C	0.94 (2)
C5—C6	1.5187 (18)	C21—C22	1.501 (4)
C5—C13	1.5283 (17)	C21—H21A	0.97 (3)
C5—H5	0.979 (17)	C21—H21B	1.01 (2)
C6—C7	1.4101 (19)	C22—H22A	1.00 (3)
C7—C19	1.504 (2)	C22—H22B	1.02 (3)
C8—C9	1.399 (2)	C22—H22C	1.00 (3)
C9—C20	1.440 (2)		
C8—S1—C21	101.82 (8)	H10B—C10—H10C	107.6 (16)
C3—O1—H1	111.8 (15)	O2—C11—C12	121.86 (13)
C8—N1—C7	118.86 (12)	O2—C11—C4	121.37 (12)
C6—C1—C9	118.03 (12)	C12—C11—C4	116.76 (12)
C6—C1—C2	122.39 (12)	C11—C12—H12A	111.8 (14)
C9—C1—C2	119.57 (12)	C11—C12—H12B	109.4 (14)
C1—C2—C3	112.98 (11)	H12A—C12—H12B	109.4 (19)
C1—C2—H2A	110.1 (10)	C11—C12—H12C	110.3 (14)
C3—C2—H2A	109.7 (10)	H12A—C12—H12C	104.4 (19)
C1—C2—H2B	109.4 (10)	H12B—C12—H12C	111.5 (19)
C3—C2—H2B	107.6 (10)	C14—C13—C18	119.17 (12)
H2A—C2—H2B	106.8 (14)	C14—C13—C5	120.31 (12)
O1—C3—C10	105.58 (11)	C18—C13—C5	120.41 (12)
O1—C3—C2	110.47 (11)	C13—C14—C15	120.67 (13)
C10—C3—C2	109.94 (11)	C13—C14—H14	119.4 (11)
O1—C3—C4	112.05 (11)	C15—C14—H14	119.9 (11)
C10—C3—C4	111.59 (12)	C16—C15—C14	119.08 (13)
C2—C3—C4	107.25 (11)	C16—C15—H15	119.4 (11)

C11—C4—C3	112.15 (11)	C14—C15—H15	121.5 (11)
C11—C4—C5	109.54 (11)	C15—C16—C17	121.49 (13)
C3—C4—C5	112.56 (11)	C15—C16—Cl1	118.67 (11)
C11—C4—H4	105.9 (10)	C17—C16—Cl1	119.85 (11)
C3—C4—H4	107.8 (10)	C16—C17—C18	118.99 (13)
C5—C4—H4	108.6 (10)	C16—C17—H17	121.1 (12)
C6—C5—C13	113.33 (10)	C18—C17—H17	119.9 (12)
C6—C5—C4	112.58 (11)	C17—C18—C13	120.58 (13)
C13—C5—C4	107.19 (10)	C17—C18—H18	119.9 (11)
C6—C5—H5	109.1 (10)	C13—C18—H18	119.5 (10)
C13—C5—H5	107.0 (9)	C7—C19—H19A	113.7 (13)
C4—C5—H5	107.3 (9)	C7—C19—H19B	110.8 (13)
C1—C6—C7	117.95 (12)	H19A—C19—H19B	104.6 (18)
C1—C6—C5	121.75 (12)	C7—C19—H19C	108.6 (13)
C7—C6—C5	120.20 (12)	H19A—C19—H19C	109.5 (19)
N1—C7—C6	123.02 (13)	H19B—C19—H19C	109.6 (18)
N1—C7—C19	114.57 (12)	N2—C20—C9	177.77 (17)
C6—C7—C19	122.37 (13)	C22—C21—S1	113.37 (15)
N1—C8—C9	121.71 (13)	C22—C21—H21A	111.5 (15)
N1—C8—S1	119.68 (11)	S1—C21—H21A	103.7 (15)
C9—C8—S1	118.50 (11)	C22—C21—H21B	113.4 (13)
C8—C9—C1	119.97 (13)	S1—C21—H21B	106.3 (13)
C8—C9—C20	119.01 (13)	H21A—C21—H21B	108 (2)
C1—C9—C20	121.01 (13)	C21—C22—H22A	109.8 (17)
C3—C10—H10A	110.9 (11)	C21—C22—H22B	114.1 (16)
C3—C10—H10B	112.5 (11)	H22A—C22—H22B	111 (2)
H10A—C10—H10B	109.2 (16)	C21—C22—H22C	112.5 (15)
C3—C10—H10C	110.1 (12)	H22A—C22—H22C	105 (2)
H10A—C10—H10C	106.2 (16)	H22B—C22—H22C	104 (2)

C6—C1—C2—C3	−20.84 (18)	C7—N1—C8—S1	−179.14 (10)
C9—C1—C2—C3	158.07 (12)	C21—S1—C8—N1	−16.72 (14)
C1—C2—C3—O1	−70.69 (14)	C21—S1—C8—C9	166.99 (12)
C1—C2—C3—C10	173.18 (12)	N1—C8—C9—C1	4.9 (2)
C1—C2—C3—C4	51.68 (15)	S1—C8—C9—C1	−178.87 (10)
O1—C3—C4—C11	−65.87 (14)	N1—C8—C9—C20	−174.18 (13)
C10—C3—C4—C11	52.30 (15)	S1—C8—C9—C20	2.02 (17)
C2—C3—C4—C11	172.75 (11)	C6—C1—C9—C8	−0.62 (19)
O1—C3—C4—C5	58.20 (14)	C2—C1—C9—C8	−179.58 (12)
C10—C3—C4—C5	176.36 (11)	C6—C1—C9—C20	178.46 (12)
C2—C3—C4—C5	−63.19 (14)	C2—C1—C9—C20	−0.49 (19)
C11—C4—C5—C6	166.36 (10)	C3—C4—C11—O2	67.98 (16)
C3—C4—C5—C6	40.86 (15)	C5—C4—C11—O2	−57.75 (16)
C11—C4—C5—C13	−68.35 (13)	C3—C4—C11—C12	−113.30 (14)
C3—C4—C5—C13	166.15 (11)	C5—C4—C11—C12	120.97 (14)
C9—C1—C6—C7	−5.11 (18)	C6—C5—C13—C14	−135.46 (13)
C2—C1—C6—C7	173.82 (12)	C4—C5—C13—C14	99.70 (14)
C9—C1—C6—C5	178.36 (12)	C6—C5—C13—C18	48.46 (16)

C2—C1—C6—C5	−2.72 (19)	C4—C5—C13—C18	−76.37 (15)
C13—C5—C6—C1	−129.28 (13)	C18—C13—C14—C15	1.0 (2)
C4—C5—C6—C1	−7.41 (17)	C5—C13—C14—C15	−175.14 (12)
C13—C5—C6—C7	54.26 (16)	C13—C14—C15—C16	0.1 (2)
C4—C5—C6—C7	176.13 (11)	C14—C15—C16—C17	−1.1 (2)
C8—N1—C7—C6	−3.2 (2)	C14—C15—C16—Cl1	178.98 (11)
C8—N1—C7—C19	174.33 (13)	C15—C16—C17—C18	1.1 (2)
C1—C6—C7—N1	7.4 (2)	Cl1—C16—C17—C18	−179.05 (10)
C5—C6—C7—N1	−176.06 (12)	C16—C17—C18—C13	0.0 (2)
C1—C6—C7—C19	−170.04 (13)	C14—C13—C18—C17	−1.0 (2)
C5—C6—C7—C19	6.6 (2)	C5—C13—C18—C17	175.07 (12)
C7—N1—C8—C9	−3.0 (2)	C8—S1—C21—C22	−72.41 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.87 (2)	2.06 (2)	2.8655 (15)	155 (2)
C5—H5···O1^{i}	0.98 (2)	2.49 (2)	3.4276 (16)	160 (1)
C14—H14···O1^{i}	0.97 (2)	2.46 (2)	3.3523 (17)	154 (2)
C18—H18···N2^{ii}	0.98 (2)	2.56 (2)	3.538 (2)	174 (2)

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