organic compounds

9-(5-Bromo-1H-indol-3-yl)-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione dimethyl sulfoxide monosolvate

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In the title compound, $C_{21}H_{19}BrN_2O_2 \cdot C_2H_6OS$, the indole ring system is essentially planar, with a maximum deviation of 0.050 (3) Å for the non-bridgehead C atom adjacent to the N atom. The two cyclohex-2-enone rings adopt half-chair conformations. An intramolecular C—H···O hydrogen bond occurs. The solvent molecule exhibits minor disorder of the S atom [site occupancies = 0.8153 (16) and 0.1847 (18)]. In the crystal, molecules are linked by N—H···O hydrogen bonds, forming layers parallel to the bc plane.

Related literature

For biological properties of acridines, including antibacterial, anti-parasitic, and antitumor activity, see: Biwersi et al. (1994); Wainwright (2001); Guetzoyan et al. (2007); Denny (2002); Luan et al. (2011). For recent studies showing that some acridine analogs having aryl and heteroaryl substituents at the ten position on the ring exert potassium-channel-modulating activiy, see: Şimşek et al. (2004), Berkan et al. (2002). For a description of the Cambridge Structural Database, see: Allen, (2002).

Experimental

Crystal data

$C_{21}H_{19}BrN_2O_2 \cdot C_2H_6OS$	V = 2169.72 (16) Å^3
M_r = 489.42	Z = 4
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation
a = 9.1544 (4) Å	μ = 3.71 mm^{-1}
b = 18.9619 (8) Å	T = 123 K
c = 12.9790 (5) Å	$0.51 \times 0.23 \times 0.12$ mm
β = 105.623 (4)°	

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer	model (Clark & Reid, 1995)]
	T_{min} = 0.272, T_{max} = 0.721
Absorption correction: analytical [CrysAlis PRO (Agilent, 2011), using a multi-faceted crystal	14006 measured reflections
	4444 independent reflections
	4183 reflections with $I > 2\sigma(I)$
	R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052	6 restraints
$wR(F^2)$ = 0.137	H-atom parameters constrained
S = 1.06	$\Delta\rho_{max}$ = 1.96 e Å^{-3}
4444 reflections	$\Delta\rho_{min}$ = −1.35 e Å^{-3}
290 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16A···O1	0.95	2.50	3.252 (3)	136
N1—H1A···O2^i	0.88	2.03	2.901 (3)	173
N2—H2C···O100^{ii}	0.88	2.10	2.920 (3)	154
N2—H2C···O100^{iii}	0.88	2.52	3.038 (3)	118

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

Data collection: CrysAlis PRO (Agilent, 2011); cell refinement: CrysAlis PRO; data reduction: CrysAlis PRO; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5266).

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supplementary materials

9-(5-Bromo-1*H*-indol-3-yl)-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione dimethyl sulfoxide monosolvate

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Comment

Acridines display abroad range of biological properties including antibacterial, anti-parasitic, and antitumor activities (Biwersi *et al.*, 1994; Wainwright, 2001;Guetzoyan *et al.*, 2007; Denny, 2002; Luan *et al.*, 2011). Furthermore, the indole moiety also has a wide range of biological activities which may enhance the acridine ring properties. Recent studies show that some acridine analogs having aryl and heteroaryl substituents on their ten position on the ring exert potassium channel modulating activities (Şimşek *et al.*, 2004; Berkan *et al.*, 2002).

   The title acridine compound contains (Fig. 1), 5-bromo-3-methyl-1*H*-indole connected to the 3,4,6,7,9,10-hexa-hydro-2H,5*H*-acridine-1,8-dione system and the disordered dimethyl sulfoxide solvent molecule. The Br—C bond distance [1.910 (3) Å] is in the normal range (Allen, 2002). The 1-H indole ring system is essentially planar with a maximum deviation of -0.050 (3) Å for atom C21. The 1*H*-indol ring system forms a dihedral angle of 22.40 (12) ° with the 1,4-dihydro-pyridine ring (N1/C1/C6—C8/C13). The two cyclohex-2-enone rings (C1—C6 and C8—C13) adopt half chair conformations with C3 atom 0.345 (3) Å and C11 atom 0.335 (3) Å out of the mean-plane formed by the remaining ring atoms. The solvent molecule exhibits minor disorder of its S atom [site occupancies = 0.8153 (16) and 0.1847 (18)]. In the crystal, molecules are linked by C—H···O and N—H···O hydrogen bonds (Tab. 1 & Fig. 2).

Experimental

A mixture of 5-bromoindole-3-carbaldehyde (1.0 mmol), 1,3-cyclohexanedione (2.0 mmol), ammonium acetate (5.0 mmol) was dissolved in 5 ml of methanol and refluxed until the reaction was completed (monitored by TLC). The forming precipitate was filtered off and crystallized from ethanol. Crystals were grown by slow evaporation of an di-methyl sulfoxide/methanol mixed solution.

Refinement

All disordered components were subjected to rigid bond and similarity restraints and all major and minor disordered components were refined anisotropically. Hydrogen atoms were positioned geometrically [C—H = 0.95–1.00 Å; N—H = 0.88 Å] and refined using a riding model, with $U_{iso}(H) = 1.2\ U_{eq}(C, N)$ or $1.5\ U_{eq}(C_{methyl})$. A rotating-group model was applied for methyl groups.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).
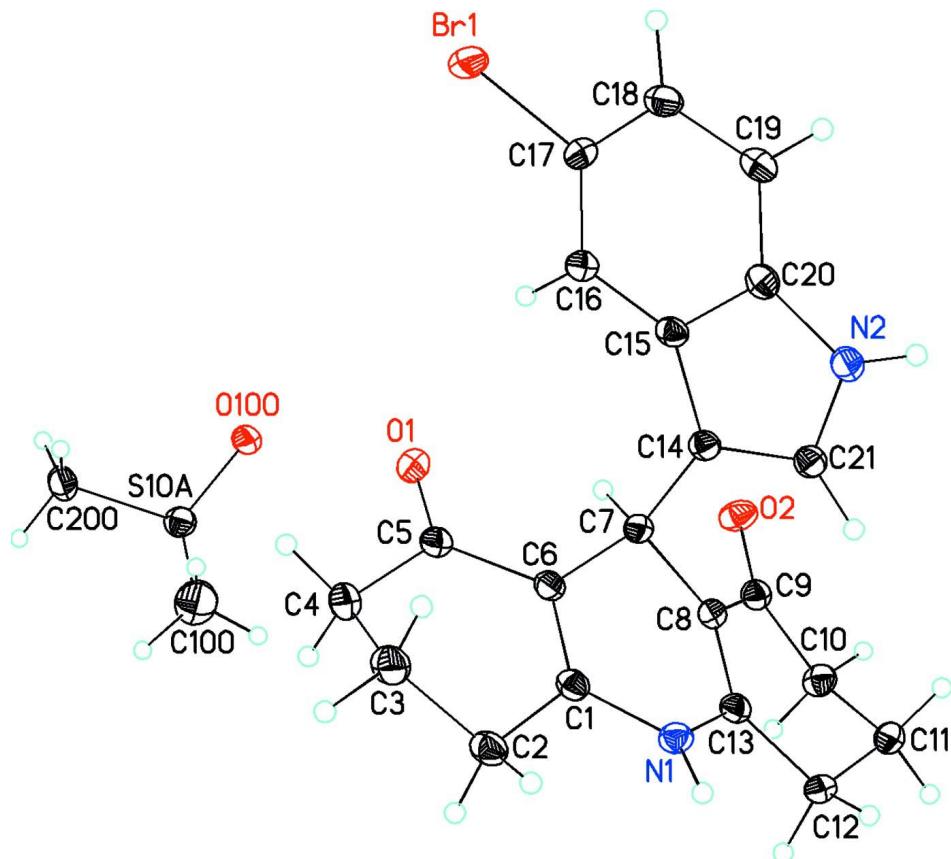
Figure 1

The molecular structure of (I) with 30% probability ellipsoids for non-H atoms. Only major disordered component for the solvent molecule is shown.
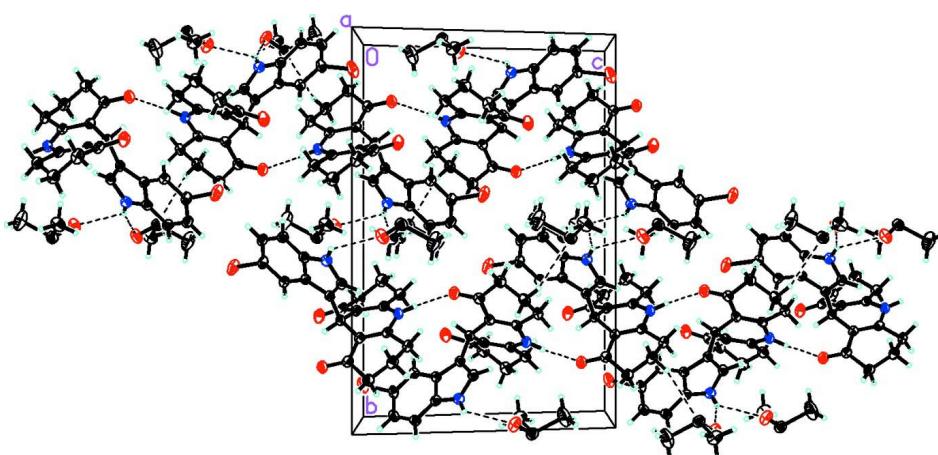
Figure 2

The packing of (I), viewed down *a* axis, showing molecules are linked into plane parallel to *bc* plane. Only major disordered component for the solvent molecule is shown and hydrogen bonds are shown as dashed lines.

9-(5-Bromo-1*H*-indol-3-yl)-1,2,3,4,5,6,7,8,9,10- decahydroacridine-1,8-dione dimethyl sulfoxide monosolvate

Crystal data

$C_{21}H_{19}BrN_2O_2 \cdot C_2H_6OS$	$F(000) = 1008$
$M_r = 489.42$	$D_x = 1.498$ Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Hall symbol: -P 2ybc	Cell parameters from 7435 reflections
$a = 9.1544$ (4) Å	$\theta = 3.5$–$75.5°$
$b = 18.9619$ (8) Å	$\mu = 3.71$ mm^{-1}
$c = 12.9790$ (5) Å	$T = 123$ K
$\beta = 105.623$ (4)°	Prism, light-yellow
$V = 2169.72$ (16) Å^3	$0.51 \times 0.23 \times 0.12$ mm
$Z = 4$	

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer	$T_{min} = 0.272$, $T_{max} = 0.721$
Radiation source: Enhance (Cu) X-ray Source	14006 measured reflections
Graphite monochromator	4444 independent reflections
Detector resolution: 10.5081 pixels mm^{-1}	4183 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.041$
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2011), using a multi-faceted crystal model (Clark & Reid, 1995)]	$\theta_{max} = 75.7°$, $\theta_{min} = 4.2°$
	$h = -10 \rightarrow 11$
	$k = -20 \rightarrow 23$
	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.052$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.137$	
$S = 1.06$	H-atom parameters constrained
4444 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 3.3586P]$ where $P = (F_o^2 + 2F_c^2)/3$
290 parameters	
6 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 1.96$ e Å^{-3}
	$\Delta\rho_{min} = -1.35$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S10A	−0.13796 (9)	0.48345 (4)	0.17928 (7)	0.03482 (18)	0.8153 (16)
O100	−0.0432 (3)	0.51319 (16)	0.1107 (2)	0.0382 (7)	0.8153 (16)
C100	−0.0885 (11)	0.5268 (4)	0.3027 (4)	0.0648 (14)	0.8153 (16)
H10C	0.0119	0.5110	0.3443	0.097*	0.8153 (16)

H10D	−0.1634	0.5160	0.3419	0.097*	0.8153 (16)
H10E	−0.0863	0.5778	0.2910	0.097*	0.8153 (16)
C200	−0.3247 (4)	0.5167 (3)	0.1308 (6)	0.0412 (9)	0.8153 (16)
H20A	−0.3729	0.4955	0.0611	0.062*	0.8153 (16)
H20B	−0.3210	0.5680	0.1231	0.062*	0.8153 (16)
H20C	−0.3835	0.5050	0.1814	0.062*	0.8153 (16)
S10B	−0.1368 (4)	0.54694 (19)	0.1798 (3)	0.03482 (18)	0.1847 (16)
O101	−0.0449 (18)	0.4988 (9)	0.1276 (13)	0.0382 (7)	0.1847 (16)
C101	−0.096 (5)	0.517 (2)	0.3127 (12)	0.0648 (14)	0.1847 (16)
H10F	0.0071	0.4981	0.3344	0.097*	0.1847 (16)
H10G	−0.1685	0.4803	0.3183	0.097*	0.1847 (16)
H10H	−0.1043	0.5567	0.3596	0.097*	0.1847 (16)
C201	−0.3306 (14)	0.5260 (18)	0.132 (3)	0.0412 (9)	0.1847 (16)
H20D	−0.3710	0.5458	0.0604	0.062*	0.1847 (16)
H20E	−0.3858	0.5458	0.1805	0.062*	0.1847 (16)
H20F	−0.3430	0.4746	0.1291	0.062*	0.1847 (16)
Br1	0.25418 (3)	0.905937 (19)	0.00832 (2)	0.04525 (11)	
O1	0.2313 (2)	0.77721 (11)	0.33651 (15)	0.0352 (4)	
O2	0.7181 (2)	0.65871 (10)	0.37707 (14)	0.0340 (4)	
N1	0.6163 (2)	0.78876 (11)	0.65949 (16)	0.0260 (4)	
H1A	0.6514	0.8073	0.7235	0.031*	
N2	0.7825 (2)	0.92633 (11)	0.39600 (17)	0.0269 (4)	
H2C	0.8614	0.9546	0.4157	0.032*	
C1	0.4715 (3)	0.80553 (12)	0.59876 (19)	0.0243 (5)	
C2	0.3651 (3)	0.83425 (14)	0.6587 (2)	0.0296 (5)	
H2A	0.4191	0.8691	0.7123	0.036*	
H2B	0.3301	0.7954	0.6972	0.036*	
C3	0.2284 (3)	0.86936 (15)	0.5820 (2)	0.0365 (6)	
H3A	0.1516	0.8807	0.6205	0.044*	
H3B	0.2604	0.9140	0.5551	0.044*	
C4	0.1589 (3)	0.82086 (16)	0.4883 (2)	0.0364 (6)	
H4A	0.1152	0.7791	0.5149	0.044*	
H4B	0.0753	0.8460	0.4372	0.044*	
C5	0.2730 (3)	0.79654 (13)	0.4305 (2)	0.0275 (5)	
C6	0.4318 (3)	0.79381 (12)	0.49155 (19)	0.0239 (5)	
C7	0.5513 (2)	0.77218 (12)	0.43652 (18)	0.0225 (4)	
H7A	0.5026	0.7423	0.3730	0.027*	
C8	0.6706 (3)	0.72888 (12)	0.51402 (18)	0.0235 (5)	
C9	0.7466 (3)	0.67249 (13)	0.4734 (2)	0.0265 (5)	
C10	0.8583 (3)	0.62845 (14)	0.5553 (2)	0.0305 (5)	
H10A	0.9300	0.6052	0.5210	0.037*	
H10B	0.8032	0.5912	0.5830	0.037*	
C11	0.9464 (3)	0.67432 (14)	0.6478 (2)	0.0303 (5)	
H11A	1.0056	0.7101	0.6207	0.036*	
H11B	1.0183	0.6446	0.7008	0.036*	
C12	0.8380 (3)	0.71116 (13)	0.70158 (19)	0.0270 (5)	
H12A	0.8002	0.6766	0.7455	0.032*	
H12B	0.8934	0.7486	0.7498	0.032*	
C13	0.7059 (3)	0.74323 (12)	0.62042 (19)	0.0241 (5)	

C14	0.6227 (3)	0.83598 (12)	0.39910 (18)	0.0230 (4)
C15	0.5613 (3)	0.87732 (12)	0.30416 (18)	0.0235 (4)
C16	0.4347 (3)	0.86999 (13)	0.21609 (19)	0.0261 (5)
H16A	0.3642	0.8327	0.2122	0.031*
C17	0.4162 (3)	0.91920 (14)	0.1349 (2)	0.0296 (5)
C18	0.5129 (3)	0.97717 (14)	0.1398 (2)	0.0300 (5)
H18A	0.4911	1.0114	0.0843	0.036*
C19	0.6399 (3)	0.98455 (13)	0.2253 (2)	0.0276 (5)
H19A	0.7078	1.0229	0.2291	0.033*
C20	0.6650 (3)	0.93375 (13)	0.30590 (19)	0.0253 (5)
C21	0.7575 (3)	0.86762 (13)	0.45088 (19)	0.0253 (5)
H21A	0.8242	0.8512	0.5157	0.030*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S10A	0.0375 (4)	0.0344 (4)	0.0344 (4)	0.0084 (3)	0.0128 (3)	0.0046 (3)
O100	0.0318 (9)	0.0508 (17)	0.0359 (14)	0.0114 (11)	0.0159 (9)	0.0126 (11)
C100	0.058 (2)	0.093 (4)	0.0423 (19)	0.008 (2)	0.0118 (19)	−0.023 (2)
C200	0.0305 (13)	0.045 (2)	0.0518 (17)	0.0033 (13)	0.0173 (13)	0.0110 (16)
S10B	0.0375 (4)	0.0344 (4)	0.0344 (4)	0.0084 (3)	0.0128 (3)	0.0046 (3)
O101	0.0318 (9)	0.0508 (17)	0.0359 (14)	0.0114 (11)	0.0159 (9)	0.0126 (11)
C101	0.058 (2)	0.093 (4)	0.0423 (19)	0.008 (2)	0.0118 (19)	−0.023 (2)
C201	0.0305 (13)	0.045 (2)	0.0518 (17)	0.0033 (13)	0.0173 (13)	0.0110 (16)
Br1	0.03879 (18)	0.0606 (2)	0.02995 (16)	−0.00943 (13)	−0.00175 (13)	0.01575 (12)
O1	0.0266 (8)	0.0499 (11)	0.0276 (9)	0.0020 (8)	0.0049 (7)	0.0038 (8)
O2	0.0406 (10)	0.0372 (10)	0.0242 (8)	0.0118 (8)	0.0085 (7)	−0.0004 (7)
N1	0.0283 (9)	0.0290 (10)	0.0209 (9)	0.0015 (8)	0.0071 (8)	−0.0011 (7)
N2	0.0255 (9)	0.0284 (10)	0.0278 (10)	−0.0025 (8)	0.0089 (8)	−0.0006 (8)
C1	0.0260 (10)	0.0227 (10)	0.0262 (11)	0.0004 (8)	0.0102 (9)	0.0033 (8)
C2	0.0329 (12)	0.0307 (12)	0.0293 (12)	0.0062 (10)	0.0153 (10)	0.0024 (9)
C3	0.0352 (13)	0.0416 (14)	0.0368 (13)	0.0128 (11)	0.0170 (11)	0.0044 (11)
C4	0.0254 (11)	0.0499 (15)	0.0364 (14)	0.0071 (11)	0.0126 (10)	0.0076 (12)
C5	0.0260 (11)	0.0303 (12)	0.0273 (11)	0.0022 (9)	0.0090 (9)	0.0074 (9)
C6	0.0233 (10)	0.0252 (11)	0.0250 (11)	0.0019 (8)	0.0096 (9)	0.0042 (8)
C7	0.0220 (10)	0.0248 (10)	0.0209 (10)	0.0014 (8)	0.0062 (8)	0.0009 (8)
C8	0.0214 (10)	0.0255 (11)	0.0239 (11)	0.0001 (8)	0.0065 (8)	0.0028 (9)
C9	0.0257 (11)	0.0279 (11)	0.0266 (11)	0.0018 (9)	0.0082 (9)	0.0018 (9)
C10	0.0297 (11)	0.0318 (12)	0.0298 (12)	0.0095 (10)	0.0077 (10)	0.0017 (10)
C11	0.0241 (11)	0.0369 (13)	0.0291 (12)	0.0049 (10)	0.0059 (9)	0.0056 (10)
C12	0.0267 (11)	0.0321 (12)	0.0213 (10)	0.0016 (9)	0.0048 (9)	0.0042 (9)
C13	0.0245 (10)	0.0243 (10)	0.0246 (11)	0.0000 (9)	0.0083 (9)	0.0039 (8)
C14	0.0242 (10)	0.0252 (11)	0.0210 (10)	0.0034 (8)	0.0086 (8)	−0.0006 (8)
C15	0.0259 (10)	0.0239 (10)	0.0233 (10)	0.0037 (9)	0.0115 (9)	0.0014 (8)
C16	0.0268 (11)	0.0272 (11)	0.0246 (11)	0.0003 (9)	0.0077 (9)	0.0028 (9)
C17	0.0256 (11)	0.0388 (13)	0.0224 (11)	0.0013 (10)	0.0032 (9)	0.0050 (10)
C18	0.0348 (12)	0.0311 (12)	0.0267 (11)	0.0038 (10)	0.0129 (10)	0.0065 (9)
C19	0.0314 (11)	0.0251 (11)	0.0305 (12)	−0.0002 (9)	0.0154 (10)	0.0025 (9)
C20	0.0259 (10)	0.0254 (11)	0.0272 (11)	0.0015 (9)	0.0113 (9)	−0.0022 (9)
C21	0.0248 (10)	0.0288 (11)	0.0235 (10)	0.0022 (9)	0.0086 (9)	0.0000 (9)

Geometric parameters (Å, º)

S10A—O100	1.510 (3)	C3—H3B	0.9900
S10A—C100	1.748 (6)	C4—C5	1.512 (4)
S10A—C200	1.771 (4)	C4—H4A	0.9900
C100—H10C	0.9800	C4—H4B	0.9900
C100—H10D	0.9800	C5—C6	1.457 (3)
C100—H10E	0.9800	C6—C7	1.515 (3)
C200—H20A	0.9800	C7—C8	1.512 (3)
C200—H20B	0.9800	C7—C14	1.515 (3)
C200—H20C	0.9800	C7—H7A	1.0000
S10B—O101	1.519 (11)	C8—C13	1.358 (3)
S10B—C101	1.757 (11)	C8—C9	1.450 (3)
S10B—C201	1.760 (10)	C9—C10	1.513 (3)
C101—H10F	0.9800	C10—C11	1.525 (4)
C101—H10G	0.9800	C10—H10A	0.9900
C101—H10H	0.9800	C10—H10B	0.9900
C201—H20D	0.9800	C11—C12	1.527 (3)
C201—H20E	0.9800	C11—H11A	0.9900
C201—H20F	0.9800	C11—H11B	0.9900
Br1—C17	1.910 (3)	C12—C13	1.502 (3)
O1—C5	1.232 (3)	C12—H12A	0.9900
O2—C9	1.235 (3)	C12—H12B	0.9900
N1—C13	1.378 (3)	C14—C21	1.374 (3)
N1—C1	1.385 (3)	C14—C15	1.441 (3)
N1—H1A	0.8800	C15—C16	1.398 (3)
N2—C20	1.366 (3)	C15—C20	1.426 (3)
N2—C21	1.374 (3)	C16—C17	1.383 (3)
N2—H2C	0.8800	C16—H16A	0.9500
C1—C6	1.358 (3)	C17—C18	1.402 (4)
C1—C2	1.503 (3)	C18—C19	1.382 (4)
C2—C3	1.526 (4)	C18—H18A	0.9500
C2—H2A	0.9900	C19—C20	1.394 (3)
C2—H2B	0.9900	C19—H19A	0.9500
C3—C4	1.520 (4)	C21—H21A	0.9500
C3—H3A	0.9900		
O100—S10A—C100	108.4 (3)	C8—C7—H7A	108.8
O100—S10A—C200	108.1 (3)	C6—C7—H7A	108.8
C100—S10A—C200	98.3 (4)	C14—C7—H7A	108.8
O101—S10B—C101	104.4 (16)	C13—C8—C9	120.7 (2)
O101—S10B—C201	109.5 (14)	C13—C8—C7	119.9 (2)
C101—S10B—C201	101 (2)	C9—C8—C7	119.3 (2)
S10B—C101—H10F	109.5	O2—C9—C8	122.2 (2)
S10B—C101—H10G	109.5	O2—C9—C10	120.9 (2)
H10F—C101—H10G	109.5	C8—C9—C10	116.8 (2)
S10B—C101—H10H	109.5	C9—C10—C11	110.6 (2)
H10F—C101—H10H	109.5	C9—C10—H10A	109.5
H10G—C101—H10H	109.5	C11—C10—H10A	109.5
S10B—C201—H20D	109.5	C9—C10—H10B	109.5

S10B—C201—H20E	109.5	C11—C10—H10B	109.5
H20D—C201—H20E	109.5	H10A—C10—H10B	108.1
S10B—C201—H20F	109.5	C10—C11—C12	110.5 (2)
H20D—C201—H20F	109.5	C10—C11—H11A	109.6
H20E—C201—H20F	109.5	C12—C11—H11A	109.6
C13—N1—C1	120.5 (2)	C10—C11—H11B	109.6
C13—N1—H1A	119.7	C12—C11—H11B	109.6
C1—N1—H1A	119.7	H11A—C11—H11B	108.1
C20—N2—C21	108.9 (2)	C13—C12—C11	111.3 (2)
C20—N2—H2C	125.5	C13—C12—H12A	109.4
C21—N2—H2C	125.5	C11—C12—H12A	109.4
C6—C1—N1	119.7 (2)	C13—C12—H12B	109.4
C6—C1—C2	123.9 (2)	C11—C12—H12B	109.4
N1—C1—C2	116.3 (2)	H12A—C12—H12B	108.0
C1—C2—C3	110.6 (2)	C8—C13—N1	119.8 (2)
C1—C2—H2A	109.5	C8—C13—C12	123.9 (2)
C3—C2—H2A	109.5	N1—C13—C12	116.2 (2)
C1—C2—H2B	109.5	C21—C14—C15	105.8 (2)
C3—C2—H2B	109.5	C21—C14—C7	126.8 (2)
H2A—C2—H2B	108.1	C15—C14—C7	127.3 (2)
C4—C3—C2	110.5 (2)	C16—C15—C20	119.3 (2)
C4—C3—H3A	109.5	C16—C15—C14	133.7 (2)
C2—C3—H3A	109.5	C20—C15—C14	106.9 (2)
C4—C3—H3B	109.5	C17—C16—C15	117.4 (2)
C2—C3—H3B	109.5	C17—C16—H16A	121.3
H3A—C3—H3B	108.1	C15—C16—H16A	121.3
C5—C4—C3	112.7 (2)	C16—C17—C18	123.2 (2)
C5—C4—H4A	109.1	C16—C17—Br1	118.29 (19)
C3—C4—H4A	109.1	C18—C17—Br1	118.48 (19)
C5—C4—H4B	109.1	C19—C18—C17	120.0 (2)
C3—C4—H4B	109.1	C19—C18—H18A	120.0
H4A—C4—H4B	107.8	C17—C18—H18A	120.0
O1—C5—C6	121.7 (2)	C18—C19—C20	117.8 (2)
O1—C5—C4	120.7 (2)	C18—C19—H19A	121.1
C6—C5—C4	117.5 (2)	C20—C19—H19A	121.1
C1—C6—C5	120.2 (2)	N2—C20—C19	130.2 (2)
C1—C6—C7	120.0 (2)	N2—C20—C15	107.7 (2)
C5—C6—C7	119.7 (2)	C19—C20—C15	122.1 (2)
C8—C7—C6	108.48 (19)	N2—C21—C14	110.7 (2)
C8—C7—C14	110.51 (19)	N2—C21—H21A	124.6
C6—C7—C14	111.30 (19)	C14—C21—H21A	124.6
C13—N1—C1—C6	−18.3 (3)	C7—C8—C13—N1	10.9 (3)
C13—N1—C1—C2	159.9 (2)	C9—C8—C13—C12	9.5 (4)
C6—C1—C2—C3	−17.9 (3)	C7—C8—C13—C12	−170.2 (2)
N1—C1—C2—C3	164.0 (2)	C1—N1—C13—C8	17.0 (3)
C1—C2—C3—C4	49.7 (3)	C1—N1—C13—C12	−161.9 (2)
C2—C3—C4—C5	−54.8 (3)	C11—C12—C13—C8	12.8 (3)
C3—C4—C5—O1	−156.3 (2)	C11—C12—C13—N1	−168.3 (2)

C3—C4—C5—C6	26.5 (3)	C8—C7—C14—C21	−22.8 (3)
N1—C1—C6—C5	166.7 (2)	C6—C7—C14—C21	97.8 (3)
C2—C1—C6—C5	−11.4 (4)	C8—C7—C14—C15	159.4 (2)
N1—C1—C6—C7	−8.5 (3)	C6—C7—C14—C15	−80.0 (3)
C2—C1—C6—C7	173.4 (2)	C21—C14—C15—C16	174.8 (3)
O1—C5—C6—C1	−170.3 (2)	C7—C14—C15—C16	−7.1 (4)
C4—C5—C6—C1	6.9 (3)	C21—C14—C15—C20	−1.1 (3)
O1—C5—C6—C7	4.9 (4)	C7—C14—C15—C20	177.1 (2)
C4—C5—C6—C7	−177.9 (2)	C20—C15—C16—C17	−0.7 (3)
C1—C6—C7—C8	32.0 (3)	C14—C15—C16—C17	−176.1 (2)
C5—C6—C7—C8	−143.2 (2)	C15—C16—C17—C18	−3.5 (4)
C1—C6—C7—C14	−89.8 (3)	C15—C16—C17—Br1	174.75 (18)
C5—C6—C7—C14	95.0 (2)	C16—C17—C18—C19	4.6 (4)
C6—C7—C8—C13	−33.3 (3)	Br1—C17—C18—C19	−173.56 (19)
C14—C7—C8—C13	89.0 (3)	C17—C18—C19—C20	−1.4 (4)
C6—C7—C8—C9	147.0 (2)	C21—N2—C20—C19	−179.6 (2)
C14—C7—C8—C9	−90.7 (3)	C21—N2—C20—C15	0.1 (3)
C13—C8—C9—O2	−179.2 (2)	C18—C19—C20—N2	177.0 (2)
C7—C8—C9—O2	0.5 (4)	C18—C19—C20—C15	−2.7 (4)
C13—C8—C9—C10	3.6 (3)	C16—C15—C20—N2	−176.0 (2)
C7—C8—C9—C10	−176.7 (2)	C14—C15—C20—N2	0.6 (3)
O2—C9—C10—C11	145.2 (2)	C16—C15—C20—C19	3.8 (3)
C8—C9—C10—C11	−37.5 (3)	C14—C15—C20—C19	−179.6 (2)
C9—C10—C11—C12	58.8 (3)	C20—N2—C21—C14	−0.9 (3)
C10—C11—C12—C13	−46.2 (3)	C15—C14—C21—N2	1.2 (3)
C9—C8—C13—N1	−169.4 (2)	C7—C14—C21—N2	−177.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···O1	1.00	2.49	2.869 (3)	102
C16—H16*A*···O1	0.95	2.50	3.252 (3)	136
N1—H1*A*···O2ⁱ	0.88	2.03	2.901 (3)	173
N2—H2*C*···O100ⁱⁱ	0.88	2.10	2.920 (3)	154
N2—H2*C*···O100ⁱⁱⁱ	0.88	2.52	3.038 (3)	118

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