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# 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

**Ahmed El-Khouly,[a] Sema Öztürk Yildirim,[b,c] Ray J. Butcher,[b]* Rahime Şimsek[a] and Cihat Şafak[a]**

[a]Hacettepe University, Faculty of Pharmacy, Dept. of Pharmaceutical Chemistry, 06100 Sihhiye-Ankara, Turkey, [b]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA, and [c]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey
Correspondence e-mail: rbutcher99@yahoo.com

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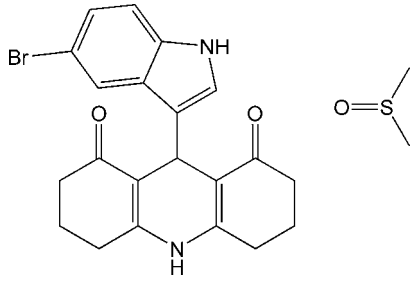
Key indicators: single-crystal X-ray study; $T$ = 123 K; mean $\sigma$(C–C) = 0.004 Å; disorder in solvent or counterion; $R$ factor = 0.052; $wR$ factor = 0.137; data-to-parameter ratio = 15.3.

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

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

### Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2011), using a multi-faceted crystal model (Clark & Reid, 1995)]
$T_{min}$ = 0.272, $T_{max}$ = 0.721
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
$wR(F^2)$ = 0.137
$S$ = 1.06
4444 reflections
290 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.96 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.35 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C16—H16$A$···O1 | 0.95 | 2.50 | 3.252 (3) | 136 |
| N1—H1$A$···O2$^{i}$ | 0.88 | 2.03 | 2.901 (3) | 173 |
| N2—H2$C$···O100$^{ii}$ | 0.88 | 2.10 | 2.920 (3) | 154 |
| N2—H2$C$···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*.

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

## References

Agilent (2011). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Berkan, Ö., Saraç, B., Şimşek, R., Yildirim, Ş., Sarioglu, Y. & Şafak, C. (2002). *Eur. J. Med. Chem.* **37**, 519–523.
Biwersi, J., Tulk, B. & Verkman, A. S. (1994). *Anal. Biochem.* **219**, 139-43.
Clark, R. C. & Reid, J. S. (1995). *Acta Cryst.* A**51**, 887–897.
Denny, W. (2002). *Curr. Med. Chem.* **9**, 1655–1665.
Guetzoyan, L., Ramiandrasoa, F., Dorizon, H., Desprez, C., Bridoux, A., Rogier, C., Pradines, B. & Perree-Fauvet, M. (2007). *Bioorg. Med. Chem.* **15**, 3278–3289.
Luan, X., Gao, C., Zhang, N., Chen, Y., Sun, Q., Tan, C., Liu, H., Jin, Y. & Jiang, Y. (2011). *Bioorg. Med. Chem.* **19**, 3312–3319.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Şimşek, R., Özkan, M., Kısmetli, E., Uma, S. & Şafak, C. (2004). *Il Farmaco*, **59**, 939–943.
Wainwright, M. J. (2001). *J. Antimicrob. Chemother.* **47**, 1–13.

# supporting information

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## 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

**Ahmed El-Khouly, Sema Öztürk Yildirim, Ray J. Butcher, Rahime Şimsek and Cihat Şafak**

### S1. 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).

### S2. 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.

### S3. 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.

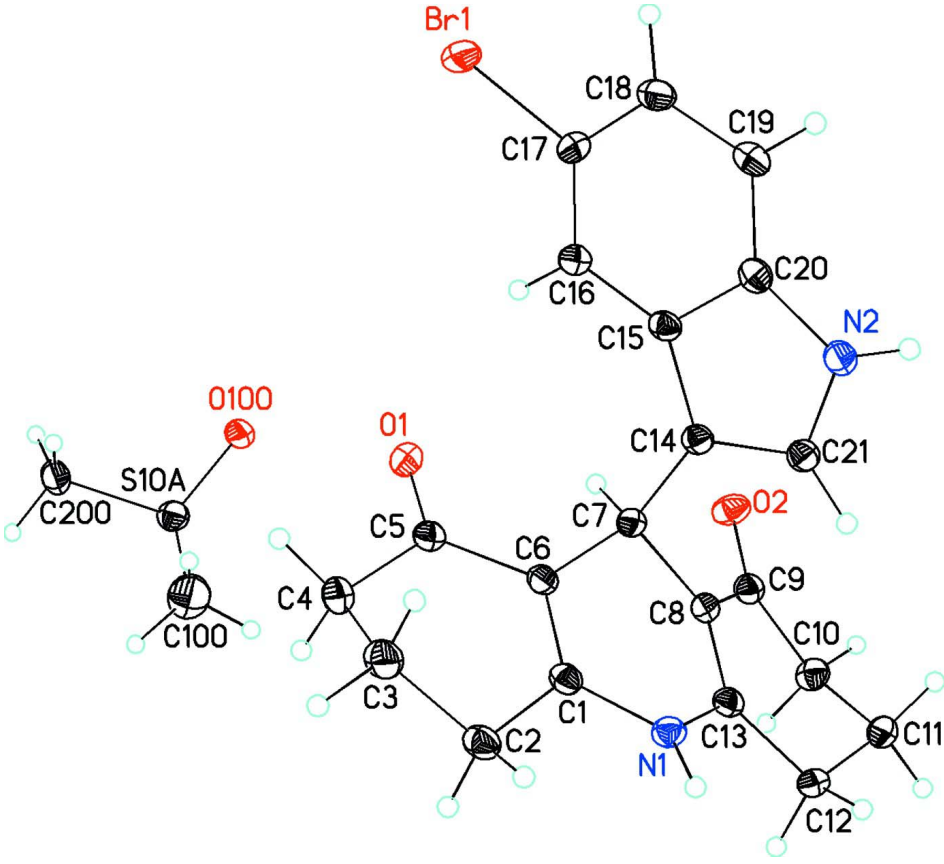


**Figure 1**

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

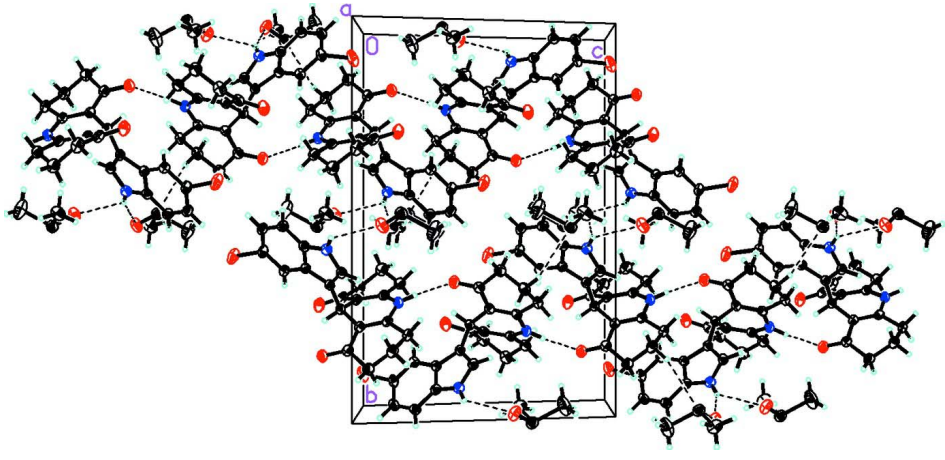


**Figure 2**

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*

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

*Data collection*

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

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.137$
$S = 1.06$
4444 reflections
290 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 3.3586P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.96$ 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 | | |

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|---|---|---|---|
| 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*···O2$^{i}$ | 0.88 | 2.03 | 2.901 (3) | 173 |
| N2—H2*C*···O100$^{ii}$ | 0.88 | 2.10 | 2.920 (3) | 154 |
| N2—H2*C*···O100$^{iii}$ | 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.