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# Crystal structure of ethyl 2-[9-(5-bromo-2-hydroxyphenyl)-1,8-dioxo-1,2,3,4,5,-6,7,8,9,10-decahydroacridin-10-yl]-acetate

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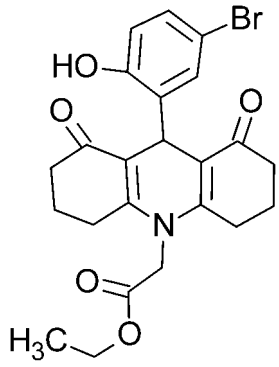
In the title compound, $C_{23}H_{24}BrNO_5$, the central 1,4-dihydropyridine ring of the 1,2,3,4,5,6,7,8,9,10-decahydroacridine ring system adopts a half-chair conformation. The two cyclohexene rings fused to the central ring both have a twisted-boat conformation. The mean planes of the bromohydroxyphenyl ring and the major and minor components of the disordered ethyl aminoacetate moiety make dihedral angles of 78.99 (12), 85.9 (2) and 88.3 (9)°, respectively, with the 1,4-dihydropyridine ring. The terminal ethyl group of the ethyl aminoacetate moiety is disordered over two sets of sites with refined occupancies of 0.768 (17) and 0.232 (17). The molecular conformation is stabilized by an intramolecular O—H···O hydrogen bond, forming an $S(8)$ ring motif. In the crystal, C—H···O hydrogen bonds connect the molecules into layers parallel to (001), enclosing $R_1^2(7)$ ring motifs.

**Keywords:** crystal structure; acridines; hydroacridinones; hydrogen bonding.

**CCDC reference**: 1437968

## 1. Related literature

For biological activities of hydroquinolines, see: Moghadam *et al.* (2011); Miri *et al.* (2007). For accridinones, see: Okoro *et al.* (2012). For dihydropyridines, see: Aydin *et al.* (2006); Rose (1990, 1991); Rose & Draeger (1992).




## 2. Experimental

### 2.1. Crystal data

$C_{23}H_{24}BrNO_5$
$M_r$ = 474.33
Orthorhombic, *Pbca*
$a$ = 8.8287 (3) Å
$b$ = 14.2531 (5) Å
$c$ = 33.1222 (11) Å
$V$ = 4168.0 (2) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 2.01 mm$^{-1}$
$T$ = 293 K
0.32 × 0.11 × 0.08 mm

### 2.2. Data collection

Agilent Xcalibur Eos Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
$T_{min}$ = 0.579, $T_{max}$ = 1.000
51745 measured reflections
5281 independent reflections
3379 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.060

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.153
$S$ = 1.02
5281 reflections
281 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.59 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H5···O1 | 0.83 (3) | 1.91 (3) | 2.709 (4) | 163 (6) |
| C10—H10$A$···O4$^{i}$ | 0.97 | 2.41 | 3.228 (4) | 142 |
| C14—H14$B$···O4$^{i}$ | 0.97 | 2.42 | 3.267 (4) | 146 |

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

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *PARST* (Nardelli, 1995).

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5239).

## References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies Ltd, Yarnton, England.
Aydin, F., Safak, C., Simşek, R., Erol, K., Ülgen, M. & Linden, A. (2006). *Pharmazie*, **61**, 655–659.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Miri, R., Javidnia, K., Mirkhani, H., Hemmateenejad, B., Sepeher, Z., Zalpour, M., Behzad, T., Khoshneviszadeh, M., Edraki, N. & Mehdipour, A. R. (2007). *Chem. Biol. Drug Des.* **70**, 329–336.
Moghadam, M. J. F., Amini, M., Asadieskandar, A. & Shaffiee, A. (2011). *J. Sci. Islamic Republic Iran*, 22, 21–25.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Okoro, C. O., Ogunwale, M. A. & Siddiquee, T. (2012). *Appl. Sci.* **2**, 368–374.
Rose, U. (1990). *J. Heterocycl. Chem.* **27**, 237–242.
Rose, U. (1991). *Arzneim. Forsch. Drug. Res.* **41**, 199–203.
Rose, U. & Draeger, M. (1992). *J. Med. Chem.* **35**, 2238–2243.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

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## Crystal structure of ethyl 2-[9-(5-bromo-2-hydroxyphenyl)-1,8-dioxo-1,2,3,4,5,6,7,8,9,10-decahydroacridin-10-yl]acetate

**Shaaban K. Mohamed, Mehmet Akkurt, Jerry P. Jasinski, Antar A. Abdelhamid, Asmaa H. Tamam and Mustafa R. Albayati**

### S1. Comment

Hydroquinolines (Moghadam *et al.*, 2011; Miri *et al.*, 2007), acridinediones (Okoro *et al.*, 2012) and other anellated dihydropyridines (Aydin *et al.*, 2006; Rose, 1990) were developed that exhibit selective cardial agonist activity while calcium antagonistic effects were observed on smooth musculature (Rose & Draeger, 1992; Rose, 1991). In this context, we report here the synthesis and crystal structure of the title compound, $C_{23}H_{24}BrNO_5$, (I).

In the structure of (I) (Fig. 1), the central 1,4-dihydropyridine ring (N1/C5–C9) of the 1,2,3,4,5,6,7,8,9,10-decahydroacridine ring system (N1/C1–C13) adopts a half-chair conformation [the puckering parameters are $Q_T$ = 0.235 (3) Å, $\theta$ = 102.0 (7) °, $\varphi$ = 6.0 (7) °]. The two cyclohexene rings (C1–C6 and C8–C13) of the 1,2,3,4,5,6,7,8,9,10-decahydroacridine ring system have a twisted-boat conformation [the puckering parameters are $Q_T$ = 0.428 (4) Å, $\theta$ = 51.0 (5) °, $\varphi$ = 109.2 (6) °, and $Q_T$ = 0.468 (4) Å, $\theta$ = 60.6 (4) °, $\varphi$ = 187.1 (5) °, respectively].

The mean planes of the bromo-hydroxyphenyl ring (C18–C23) and the major and minor components of the disordered ethyl aminoacetate moiety make dihedral angles of 78.99 (12), 85.9 (2) and 88.3 (9)°, respectively, with the 1,4-dihydropyridine ring (N1/C5–C9).

All bond lengths and bond angles in the title molecule are within the normal ranges and comparable with each other and with those obtained earlier for similar compounds.

The molecular conformation is stabilized by an intramolecular O—H···O hydrogen bond, which generates an *S*(8) ring motif (Fig. 1, Table 1).

In the crystal, molecules are linked by intermolecular C—H···O hydrogen bonds into layers parallel to (001), enclosing $R^2_1(7)$ ring motifs (Table 1, Fig. 2).

### S2. Experimental

A mixture of 1 mmol (112 mg) of cyclohexane-1,3-dione, 1 mmol (201 mg) of 4-bromo-2-hydroxybenzaldehyde and 1 mmol (103 mg) ethyl 2-aminoacetate in 30 ml ethanol was refluxed at 351 K. The reaction was monitored by TLC until completion. The solid product was deposited on cooling and collected by filtration under vacuum. Recystallization of the crude product from ethanol afforded crystals sufficient for X-ray diffraction. M.p. 515 K.

### S3. Refinement

The hydroxyl hydrogen atom was found from a difference Fourier map and its O—H bond length was restrained using a *DFIX* restraint of 0.82 (2) Å, with its displacement parameter set equal to 1.5$U_{eq}$(O). The other H atoms were placed in calculated positions with C—H = 0.93 - 0.98 Å, and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C). The terminal ethyl group (C16, C17) of the ethyl aminoacetate moiety is disordered over two sets of sites with an occupancy ratio of

0.768 (17):0.232 (17).

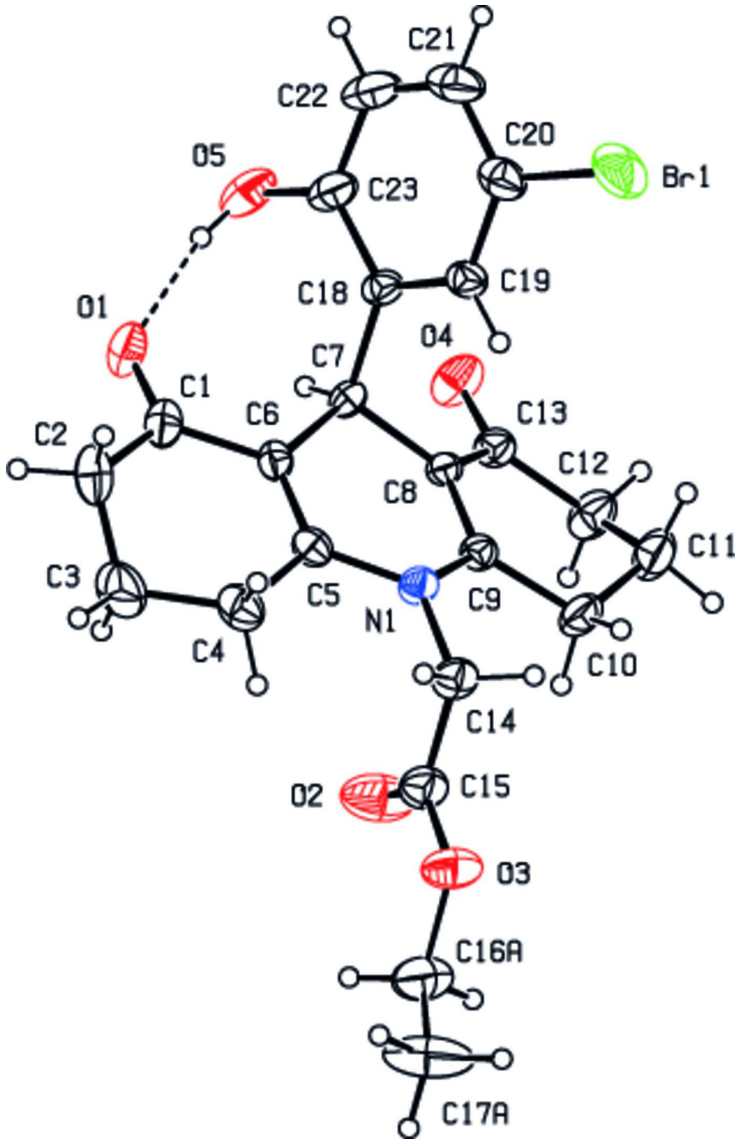


**Figure 1**

View of the title molecule with displacement ellipsoids drawn at the 30% probability level. Only the major component (C16A, C17A) of disorder is shown.

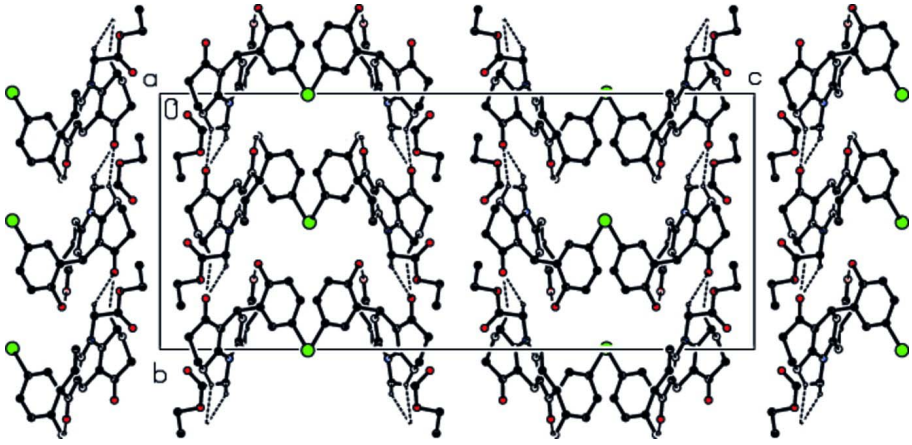


**Figure 2**

The packing of molecules in the the title compound viewed down [100]. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in hydrogen bonding and the minor component of disorder have been removed for clarity.

**Ethyl 2-[9-(5-bromo-2-hydroxyphenyl)-1,8-dioxo-1,2,3,4,5,6,7,8,9,10-decahydroacridin-10-yl]acetate**

*Crystal data*

$C_{23}H_{24}BrNO_5$
$M_r$ = 474.33
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a$ = 8.8287 (3) Å
$b$ = 14.2531 (5) Å
$c$ = 33.1222 (11) Å
$V$ = 4168.0 (2) Å$^3$
$Z$ = 8
$F(000)$ = 1952
$D_x$ = 1.512 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 4787 reflections
$\theta$ = 3.7–24.1°
$\mu$ = 2.01 mm$^{-1}$
$T$ = 293 K
Needle, colourless
0.32 × 0.11 × 0.08 mm

*Data collection*

Agilent Xcalibur Eos Gemini diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
$T_{min}$ = 0.579, $T_{max}$ = 1.000
51745 measured reflections
5281 independent reflections
3379 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.060
$\theta_{max}$ = 28.5°, $\theta_{min}$ = 2.9°
$h$ = −11→11
$k$ = −19→19
$l$ = −44→44

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.153
$S$ = 1.02
5281 reflections
281 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 5.0593P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.53 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.59 e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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) |
|---|---|---|---|---|---|
| Br1 | 0.93921 (6) | 0.00364 (4) | 0.25138 (2) | 0.0856 (2) | |
| O1 | 0.3868 (3) | −0.27352 (16) | 0.34249 (8) | 0.0639 (9) | |
| O2 | 0.3448 (4) | 0.0831 (2) | 0.45391 (9) | 0.0919 (13) | |
| O3 | 0.3033 (3) | 0.22761 (18) | 0.43222 (9) | 0.0716 (10) | |
| O4 | 0.8428 (3) | −0.19850 (15) | 0.42308 (8) | 0.0626 (9) | |
| O5 | 0.6762 (3) | −0.33400 (18) | 0.33476 (10) | 0.0738 (10) | |
| N1 | 0.4997 (3) | 0.03570 (15) | 0.38366 (7) | 0.0386 (7) | |
| C1 | 0.3472 (4) | −0.1908 (2) | 0.34564 (10) | 0.0487 (11) | |
| C2 | 0.1926 (4) | −0.1605 (3) | 0.33252 (13) | 0.0640 (11) | |
| C3 | 0.1358 (4) | −0.0829 (3) | 0.35687 (14) | 0.0781 (15) | |
| C4 | 0.2432 (4) | −0.0007 (2) | 0.35809 (11) | 0.0545 (11) | |
| C5 | 0.4030 (3) | −0.0314 (2) | 0.36740 (9) | 0.0382 (8) | |
| C6 | 0.4505 (3) | −0.12057 (19) | 0.36125 (8) | 0.0360 (8) | |
| C7 | 0.6109 (3) | −0.15066 (18) | 0.36981 (8) | 0.0347 (8) | |
| C8 | 0.6843 (3) | −0.08124 (18) | 0.39812 (8) | 0.0319 (8) | |
| C9 | 0.6337 (3) | 0.00739 (18) | 0.40212 (8) | 0.0350 (8) | |
| C10 | 0.7157 (4) | 0.0782 (2) | 0.42760 (11) | 0.0526 (11) | |
| C11 | 0.8757 (4) | 0.0494 (2) | 0.43624 (13) | 0.0643 (14) | |
| C12 | 0.8835 (4) | −0.0486 (2) | 0.45150 (11) | 0.0590 (12) | |
| C13 | 0.8066 (3) | −0.1162 (2) | 0.42346 (9) | 0.0410 (9) | |
| C14 | 0.4438 (4) | 0.1312 (2) | 0.38959 (10) | 0.0473 (10) | |
| C15 | 0.3597 (4) | 0.1431 (2) | 0.42924 (12) | 0.0546 (11) | |
| C16A | 0.2173 (11) | 0.2445 (7) | 0.4704 (2) | 0.076 (3) | 0.768 (17) |
| C16B | 0.261 (4) | 0.281 (2) | 0.4685 (9) | 0.076 (3) | 0.232 (17) |
| C17A | 0.1256 (11) | 0.3290 (10) | 0.4650 (2) | 0.109 (4) | 0.768 (17) |
| C17B | 0.108 (5) | 0.271 (3) | 0.4674 (9) | 0.109 (4) | 0.232 (17) |
| C18 | 0.7040 (3) | −0.1655 (2) | 0.33158 (9) | 0.0389 (8) | |
| C19 | 0.7647 (3) | −0.0892 (2) | 0.31119 (8) | 0.0408 (9) | |
| C20 | 0.8555 (4) | −0.1024 (3) | 0.27790 (10) | 0.0544 (10) | |
| C21 | 0.8894 (4) | −0.1919 (4) | 0.26418 (11) | 0.0693 (14) | |
| C22 | 0.8278 (5) | −0.2668 (3) | 0.28343 (12) | 0.0690 (16) | |
| C23 | 0.7340 (4) | −0.2559 (2) | 0.31685 (11) | 0.0539 (11) | |
| H2A | 0.19630 | −0.14140 | 0.30440 | 0.0770* | |
| H2B | 0.12350 | −0.21310 | 0.33470 | 0.0770* | |
| H3A | 0.11830 | −0.10490 | 0.38420 | 0.0940* | |

| | | | | | |
|---|---|---|---|---|---|
| H3B | 0.03940 | −0.06220 | 0.34600 | 0.0940* | |
| H4A | 0.20970 | 0.04340 | 0.37850 | 0.0650* | |
| H4B | 0.24150 | 0.03120 | 0.33220 | 0.0650* | |
| H5 | 0.584 (3) | −0.327 (4) | 0.3388 (18) | 0.1280* | |
| H7 | 0.60600 | −0.21100 | 0.38390 | 0.0420* | |
| H10A | 0.71620 | 0.13820 | 0.41370 | 0.0630* | |
| H10B | 0.66200 | 0.08620 | 0.45290 | 0.0630* | |
| H11A | 0.93530 | 0.05460 | 0.41180 | 0.0770* | |
| H11B | 0.91880 | 0.09150 | 0.45620 | 0.0770* | |
| H12A | 0.83570 | −0.05180 | 0.47780 | 0.0710* | |
| H12B | 0.98880 | −0.06660 | 0.45470 | 0.0710* | |
| H14A | 0.37650 | 0.14730 | 0.36750 | 0.0570* | |
| H14B | 0.52870 | 0.17430 | 0.38890 | 0.0570* | |
| H16B | 0.15260 | 0.19120 | 0.47630 | 0.0910* | 0.768 (17) |
| H16C | 0.28690 | 0.25290 | 0.49280 | 0.0910* | 0.768 (17) |
| H16D | 0.30410 | 0.25330 | 0.49270 | 0.0910* | 0.232 (17) |
| H16E | 0.29200 | 0.34600 | 0.46650 | 0.0910* | 0.232 (17) |
| H17A | 0.06790 | 0.34060 | 0.48900 | 0.1640* | 0.768 (17) |
| H17B | 0.05800 | 0.32030 | 0.44260 | 0.1640* | 0.768 (17) |
| H17C | 0.19080 | 0.38150 | 0.45980 | 0.1640* | 0.768 (17) |
| H17D | 0.06370 | 0.30440 | 0.48980 | 0.1640* | 0.232 (17) |
| H17E | 0.08130 | 0.20620 | 0.46900 | 0.1640* | 0.232 (17) |
| H17F | 0.06960 | 0.29720 | 0.44260 | 0.1640* | 0.232 (17) |
| H19 | 0.74390 | −0.02870 | 0.32010 | 0.0490* | |
| H21 | 0.95330 | −0.20040 | 0.24220 | 0.0830* | |
| H22 | 0.84880 | −0.32690 | 0.27400 | 0.0830* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.0760 (3) | 0.1199 (4) | 0.0608 (3) | −0.0183 (3) | 0.0258 (2) | 0.0017 (2) |
| O1 | 0.0663 (16) | 0.0464 (14) | 0.0790 (18) | −0.0189 (12) | −0.0077 (13) | −0.0061 (12) |
| O2 | 0.128 (3) | 0.0738 (19) | 0.0739 (19) | 0.0340 (19) | 0.0361 (19) | 0.0174 (16) |
| O3 | 0.0829 (19) | 0.0565 (15) | 0.0754 (18) | 0.0255 (14) | 0.0114 (14) | −0.0102 (13) |
| O4 | 0.0684 (16) | 0.0387 (12) | 0.0806 (17) | 0.0162 (11) | −0.0263 (13) | −0.0053 (11) |
| O5 | 0.099 (2) | 0.0375 (13) | 0.085 (2) | 0.0067 (15) | −0.0074 (18) | −0.0181 (13) |
| N1 | 0.0388 (12) | 0.0304 (11) | 0.0466 (14) | 0.0060 (10) | −0.0021 (11) | −0.0012 (10) |
| C1 | 0.0490 (18) | 0.0478 (19) | 0.0494 (18) | −0.0132 (15) | −0.0033 (15) | 0.0018 (14) |
| C2 | 0.0460 (19) | 0.072 (2) | 0.074 (2) | −0.0196 (18) | −0.0144 (18) | 0.009 (2) |
| C3 | 0.0402 (19) | 0.110 (3) | 0.084 (3) | 0.001 (2) | −0.011 (2) | −0.010 (3) |
| C4 | 0.0366 (15) | 0.065 (2) | 0.062 (2) | 0.0090 (15) | −0.0047 (16) | 0.0018 (17) |
| C5 | 0.0347 (14) | 0.0435 (15) | 0.0364 (14) | 0.0027 (12) | 0.0012 (12) | 0.0044 (12) |
| C6 | 0.0340 (14) | 0.0395 (15) | 0.0346 (14) | −0.0068 (12) | 0.0003 (11) | 0.0021 (12) |
| C7 | 0.0421 (15) | 0.0253 (12) | 0.0366 (14) | 0.0000 (11) | −0.0024 (12) | −0.0011 (11) |
| C8 | 0.0349 (13) | 0.0286 (13) | 0.0321 (13) | 0.0009 (11) | 0.0028 (11) | −0.0026 (10) |
| C9 | 0.0359 (13) | 0.0326 (14) | 0.0364 (13) | 0.0009 (11) | 0.0022 (11) | 0.0002 (11) |
| C10 | 0.056 (2) | 0.0357 (16) | 0.066 (2) | 0.0024 (14) | −0.0072 (17) | −0.0151 (15) |
| C11 | 0.058 (2) | 0.052 (2) | 0.083 (3) | −0.0059 (17) | −0.016 (2) | −0.0189 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C12 | 0.060 (2) | 0.055 (2) | 0.062 (2) | 0.0062 (17) | −0.0251 (18) | −0.0121 (17) |
| C13 | 0.0406 (15) | 0.0386 (16) | 0.0437 (16) | 0.0049 (13) | −0.0027 (13) | −0.0018 (13) |
| C14 | 0.0518 (18) | 0.0330 (15) | 0.0571 (19) | 0.0108 (14) | −0.0006 (15) | 0.0009 (14) |
| C15 | 0.0530 (19) | 0.0468 (19) | 0.064 (2) | 0.0106 (16) | 0.0004 (17) | −0.0051 (17) |
| C16A | 0.098 (6) | 0.060 (5) | 0.070 (3) | 0.013 (4) | 0.020 (3) | −0.007 (4) |
| C16B | 0.098 (6) | 0.060 (5) | 0.070 (3) | 0.013 (4) | 0.020 (3) | −0.007 (4) |
| C17A | 0.133 (6) | 0.134 (9) | 0.061 (3) | 0.085 (7) | 0.011 (3) | −0.005 (5) |
| C17B | 0.133 (6) | 0.134 (9) | 0.061 (3) | 0.085 (7) | 0.011 (3) | −0.005 (5) |
| C18 | 0.0371 (15) | 0.0423 (15) | 0.0372 (14) | 0.0068 (12) | −0.0072 (12) | −0.0110 (12) |
| C19 | 0.0329 (14) | 0.0515 (17) | 0.0379 (15) | 0.0052 (13) | −0.0017 (12) | −0.0068 (13) |
| C20 | 0.0393 (16) | 0.083 (2) | 0.0410 (17) | 0.0051 (17) | −0.0019 (14) | −0.0070 (17) |
| C21 | 0.050 (2) | 0.111 (3) | 0.0470 (19) | 0.020 (2) | −0.0008 (17) | −0.028 (2) |
| C22 | 0.073 (3) | 0.070 (3) | 0.064 (2) | 0.025 (2) | −0.009 (2) | −0.033 (2) |
| C23 | 0.057 (2) | 0.0488 (19) | 0.056 (2) | 0.0120 (17) | −0.0086 (17) | −0.0192 (16) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C20 | 1.898 (4) | C19—C20 | 1.376 (4) |
| O1—C1 | 1.234 (4) | C20—C21 | 1.387 (7) |
| O2—C15 | 1.190 (5) | C21—C22 | 1.357 (7) |
| O3—C16A | 1.495 (8) | C22—C23 | 1.391 (5) |
| O3—C15 | 1.307 (4) | C2—H2B | 0.9700 |
| O3—C16B | 1.47 (3) | C2—H2A | 0.9700 |
| O4—C13 | 1.216 (4) | C3—H3B | 0.9700 |
| O5—C23 | 1.361 (4) | C3—H3A | 0.9700 |
| N1—C5 | 1.391 (4) | C4—H4B | 0.9700 |
| N1—C9 | 1.392 (4) | C4—H4A | 0.9700 |
| N1—C14 | 1.461 (4) | C7—H7 | 0.9800 |
| O5—H5 | 0.83 (3) | C10—H10A | 0.9700 |
| C1—C2 | 1.496 (5) | C10—H10B | 0.9700 |
| C1—C6 | 1.450 (4) | C11—H11A | 0.9700 |
| C2—C3 | 1.458 (6) | C11—H11B | 0.9700 |
| C3—C4 | 1.508 (5) | C12—H12A | 0.9700 |
| C4—C5 | 1.509 (4) | C12—H12B | 0.9700 |
| C5—C6 | 1.354 (4) | C14—H14A | 0.9700 |
| C6—C7 | 1.507 (4) | C14—H14B | 0.9700 |
| C7—C8 | 1.509 (4) | C16A—H16C | 0.9700 |
| C7—C18 | 1.524 (4) | C16A—H16B | 0.9700 |
| C8—C13 | 1.456 (4) | C16B—H16E | 0.9700 |
| C8—C9 | 1.347 (4) | C16B—H16D | 0.9700 |
| C9—C10 | 1.502 (4) | C17A—H17C | 0.9600 |
| C10—C11 | 1.499 (5) | C17A—H17A | 0.9600 |
| C11—C12 | 1.487 (4) | C17A—H17B | 0.9600 |
| C12—C13 | 1.501 (4) | C17B—H17D | 0.9600 |
| C14—C15 | 1.518 (5) | C17B—H17F | 0.9600 |
| C16A—C17A | 1.462 (16) | C17B—H17E | 0.9500 |
| C16B—C17B | 1.36 (6) | C19—H19 | 0.9300 |
| C18—C23 | 1.403 (4) | C21—H21 | 0.9300 |

| | | | |
|---|---|---|---|
| C18—C19 | 1.388 (4) | C22—H22 | 0.9300 |
| | | | |
| C15—O3—C16A | 113.9 (5) | C2—C3—H3A | 109.00 |
| C15—O3—C16B | 129.4 (12) | C2—C3—H3B | 109.00 |
| C5—N1—C9 | 119.5 (2) | C3—C4—H4A | 109.00 |
| C5—N1—C14 | 119.0 (3) | C5—C4—H4B | 109.00 |
| C9—N1—C14 | 119.9 (2) | C3—C4—H4B | 109.00 |
| C23—O5—H5 | 110 (4) | H4A—C4—H4B | 108.00 |
| O1—C1—C6 | 120.8 (3) | C5—C4—H4A | 109.00 |
| C2—C1—C6 | 118.6 (3) | C6—C7—H7 | 107.00 |
| O1—C1—C2 | 120.6 (3) | C8—C7—H7 | 107.00 |
| C1—C2—C3 | 111.9 (3) | C18—C7—H7 | 107.00 |
| C2—C3—C4 | 112.8 (3) | C9—C10—H10A | 109.00 |
| C3—C4—C5 | 111.6 (3) | C9—C10—H10B | 109.00 |
| C4—C5—C6 | 122.1 (3) | C11—C10—H10A | 109.00 |
| N1—C5—C6 | 120.9 (2) | H10A—C10—H10B | 108.00 |
| N1—C5—C4 | 117.0 (2) | C11—C10—H10B | 109.00 |
| C1—C6—C5 | 120.5 (3) | C10—C11—H11B | 109.00 |
| C5—C6—C7 | 122.0 (2) | C10—C11—H11A | 109.00 |
| C1—C6—C7 | 117.5 (2) | H11A—C11—H11B | 108.00 |
| C6—C7—C8 | 109.5 (2) | C12—C11—H11A | 109.00 |
| C6—C7—C18 | 113.0 (2) | C12—C11—H11B | 109.00 |
| C8—C7—C18 | 112.1 (2) | C13—C12—H12A | 109.00 |
| C9—C8—C13 | 120.7 (2) | C13—C12—H12B | 109.00 |
| C7—C8—C13 | 116.9 (2) | H12A—C12—H12B | 108.00 |
| C7—C8—C9 | 122.3 (2) | C11—C12—H12A | 109.00 |
| C8—C9—C10 | 121.7 (3) | C11—C12—H12B | 109.00 |
| N1—C9—C8 | 120.7 (2) | N1—C14—H14A | 109.00 |
| N1—C9—C10 | 117.5 (2) | C15—C14—H14A | 109.00 |
| C9—C10—C11 | 112.2 (3) | N1—C14—H14B | 109.00 |
| C10—C11—C12 | 111.5 (3) | H14A—C14—H14B | 108.00 |
| C11—C12—C13 | 111.8 (3) | C15—C14—H14B | 109.00 |
| O4—C13—C12 | 120.5 (3) | H16B—C16A—H16C | 108.00 |
| O4—C13—C8 | 121.3 (3) | O3—C16A—H16B | 110.00 |
| C8—C13—C12 | 118.2 (2) | C17A—C16A—H16C | 110.00 |
| N1—C14—C15 | 112.7 (2) | C17A—C16A—H16B | 110.00 |
| O3—C15—C14 | 110.8 (3) | O3—C16A—H16C | 110.00 |
| O2—C15—O3 | 124.6 (4) | C17B—C16B—H16D | 112.00 |
| O2—C15—C14 | 124.6 (3) | C17B—C16B—H16E | 112.00 |
| O3—C16A—C17A | 108.1 (6) | O3—C16B—H16E | 112.00 |
| O3—C16B—C17B | 100 (2) | O3—C16B—H16D | 111.00 |
| C7—C18—C23 | 121.2 (3) | H16D—C16B—H16E | 110.00 |
| C7—C18—C19 | 120.3 (2) | H17A—C17A—H17C | 109.00 |
| C19—C18—C23 | 118.5 (3) | H17B—C17A—H17C | 110.00 |
| C18—C19—C20 | 120.5 (3) | C16A—C17A—H17B | 109.00 |
| Br1—C20—C19 | 119.3 (3) | C16A—C17A—H17C | 109.00 |
| C19—C20—C21 | 120.9 (4) | C16A—C17A—H17A | 110.00 |
| Br1—C20—C21 | 119.8 (3) | H17A—C17A—H17B | 109.00 |

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|---|---|---|---|
| C20—C21—C22 | 118.9 (3) | C16B—C17B—H17D | 109.00 |
| C21—C22—C23 | 121.6 (4) | C16B—C17B—H17E | 110.00 |
| C18—C23—C22 | 119.5 (3) | H17D—C17B—H17F | 109.00 |
| O5—C23—C18 | 121.9 (3) | H17E—C17B—H17F | 110.00 |
| O5—C23—C22 | 118.6 (3) | C16B—C17B—H17F | 109.00 |
| C3—C2—H2A | 109.00 | H17D—C17B—H17E | 110.00 |
| C3—C2—H2B | 109.00 | C20—C19—H19 | 120.00 |
| H2A—C2—H2B | 108.00 | C18—C19—H19 | 120.00 |
| C1—C2—H2B | 109.00 | C22—C21—H21 | 121.00 |
| C1—C2—H2A | 109.00 | C20—C21—H21 | 121.00 |
| C4—C3—H3B | 109.00 | C21—C22—H22 | 119.00 |
| C4—C3—H3A | 109.00 | C23—C22—H22 | 119.00 |
| H3A—C3—H3B | 108.00 | | |

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|---|---|---|---|
| C16A—O3—C15—O2 | 0.7 (6) | C18—C7—C8—C9 | 104.1 (3) |
| C16A—O3—C15—C14 | 179.5 (4) | C6—C7—C18—C23 | −102.6 (3) |
| C15—O3—C16A—C17A | −163.9 (6) | C6—C7—C18—C19 | 79.3 (3) |
| C14—N1—C9—C8 | 177.7 (3) | C6—C7—C8—C9 | −22.1 (3) |
| C9—N1—C14—C15 | −81.1 (3) | C18—C7—C8—C13 | −80.4 (3) |
| C14—N1—C5—C4 | −1.8 (4) | C7—C8—C13—C12 | 179.1 (3) |
| C5—N1—C14—C15 | 84.6 (3) | C9—C8—C13—O4 | 172.3 (3) |
| C14—N1—C5—C6 | 179.7 (3) | C7—C8—C9—N1 | 7.6 (4) |
| C9—N1—C5—C4 | 164.0 (3) | C7—C8—C9—C10 | −174.6 (3) |
| C5—N1—C9—C8 | 12.1 (4) | C7—C8—C13—O4 | −3.3 (4) |
| C5—N1—C9—C10 | −165.8 (3) | C13—C8—C9—N1 | −167.8 (2) |
| C14—N1—C9—C10 | −0.2 (4) | C9—C8—C13—C12 | −5.2 (4) |
| C9—N1—C5—C6 | −14.5 (4) | C13—C8—C9—C10 | 10.0 (4) |
| C2—C1—C6—C7 | −173.2 (3) | C8—C9—C10—C11 | 17.9 (4) |
| C6—C1—C2—C3 | −33.1 (5) | N1—C9—C10—C11 | −164.2 (3) |
| O1—C1—C6—C7 | 5.4 (4) | C9—C10—C11—C12 | −49.8 (4) |
| O1—C1—C6—C5 | −175.2 (3) | C10—C11—C12—C13 | 54.3 (4) |
| O1—C1—C2—C3 | 148.3 (4) | C11—C12—C13—C8 | −27.2 (4) |
| C2—C1—C6—C5 | 6.2 (5) | C11—C12—C13—O4 | 155.2 (3) |
| C1—C2—C3—C4 | 54.1 (5) | N1—C14—C15—O3 | −175.9 (3) |
| C2—C3—C4—C5 | −48.2 (5) | N1—C14—C15—O2 | 2.8 (5) |
| C3—C4—C5—N1 | −157.2 (3) | C7—C18—C19—C20 | 176.5 (3) |
| C3—C4—C5—C6 | 21.3 (4) | C23—C18—C19—C20 | −1.7 (4) |
| C4—C5—C6—C1 | −0.6 (4) | C7—C18—C23—O5 | 3.0 (5) |
| C4—C5—C6—C7 | 178.8 (3) | C7—C18—C23—C22 | −175.7 (3) |
| N1—C5—C6—C1 | 177.8 (3) | C19—C18—C23—O5 | −178.8 (3) |
| N1—C5—C6—C7 | −2.8 (4) | C19—C18—C23—C22 | 2.5 (5) |
| C5—C6—C7—C8 | 19.6 (4) | C18—C19—C20—Br1 | −178.9 (2) |
| C1—C6—C7—C8 | −160.9 (2) | C18—C19—C20—C21 | −0.5 (5) |
| C5—C6—C7—C18 | −106.0 (3) | Br1—C20—C21—C22 | −179.7 (3) |
| C1—C6—C7—C18 | 73.4 (3) | C19—C20—C21—C22 | 1.9 (5) |
| C8—C7—C18—C19 | −45.0 (3) | C20—C21—C22—C23 | −1.1 (6) |
| C8—C7—C18—C23 | 133.2 (3) | C21—C22—C23—O5 | −179.9 (4) |
| C6—C7—C8—C13 | 153.5 (2) | C21—C22—C23—C18 | −1.1 (6) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O5—H5···O1 | 0.83 (3) | 1.91 (3) | 2.709 (4) | 163 (6) |
| C7—H7···O5 | 0.98 | 2.47 | 2.917 (4) | 107 |
| C10—H10*A*···O4$^{i}$ | 0.97 | 2.41 | 3.228 (4) | 142 |
| C14—H14*B*···O4$^{i}$ | 0.97 | 2.42 | 3.267 (4) | 146 |

Symmetry code: (i) −*x*+3/2, *y*+1/2, *z*.