

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, C₂₃H₂₄BrNO₅, 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 R₁²(7) ring motifs.

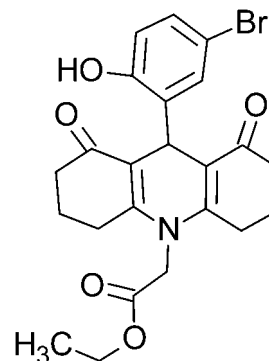
Keywords: crystal structure; acridines; hydroacridinones; hydrogen bonding.

CCDC reference: 1437968

1. Related literature

For biological activities of hydroquinolines, see: Moghadam *et al.* (2011); Miri *et al.* (2007). For acridinones, see: Okoro *et al.*

(2012). For dihydropyridines, see: Aydin *et al.* (2006); Rose (1990, 1991); Rose & Draeger (1992).



2. Experimental

2.1. Crystal data

C₂₃H₂₄BrNO₅
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) Å³
Z = 8
Mo Kα radiation
μ = 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 > 2σ(I)
R_{int} = 0.060

2.3. Refinement

R[F² > 2σ(F²)] = 0.059
wR(F²) = 0.153
S = 1.02
5281 reflections
281 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
Δρ_{max} = 0.53 e Å⁻³
Δρ_{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—H10A...O4 ⁱ	0.97	2.41	3.228 (4)	142
C14—H14B...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).

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supporting information

Acta Cryst. (2015). E71, o989–o990 [doi:10.1107/S2056989015022240]

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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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 cardiac 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, C₂₃H₂₄BrNO₅, (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*₂¹(7) ring motifs (Table 1, Fig. 2).

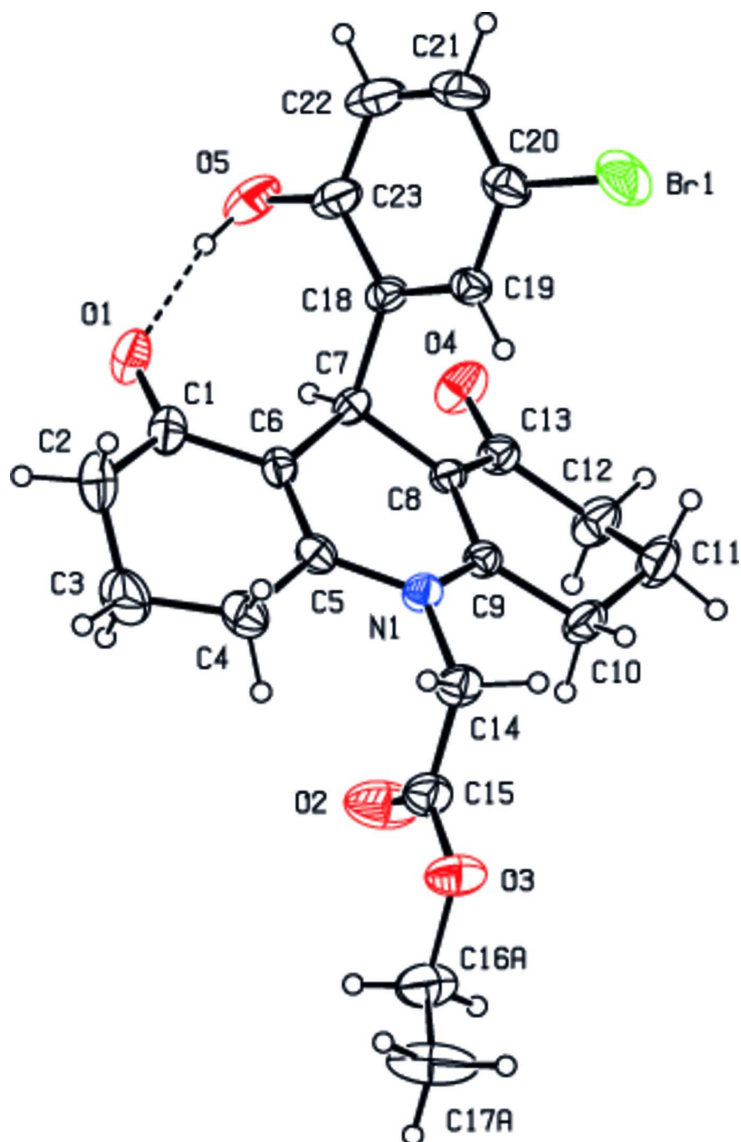
S2. Experimental

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. Recrystallization 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.

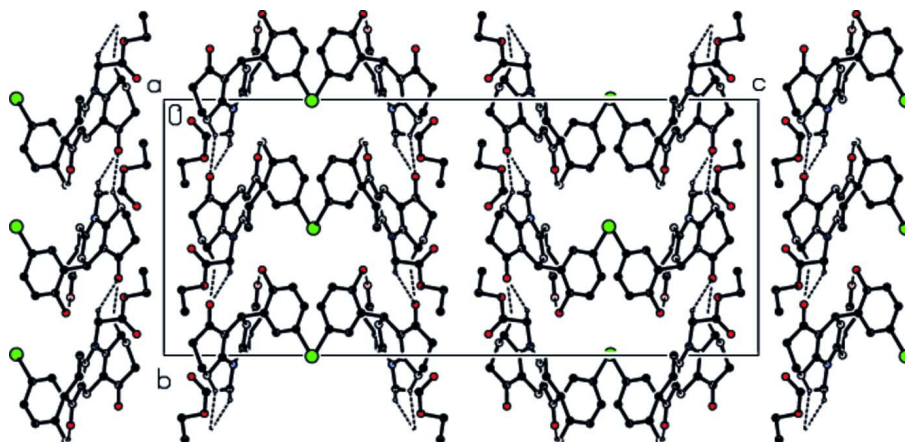


Figure 2

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) Å³

$Z = 8$

$F(000) = 1952$

$D_x = 1.512$ Mg m⁻³

Mo *K*α 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

$0.32 \times 0.11 \times 0.08$ mm

Data collection

Agilent Xcalibur Eos Gemini
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.0416 pixels mm⁻¹

ω 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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.060$

$\theta_{\max} = 28.5^\circ$, $\theta_{\min} = 2.9^\circ$

$h = -11 \rightarrow 11$

$k = -19 \rightarrow 19$

$l = -44 \rightarrow 44$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	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 (\AA^2)

	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, °)

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

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		
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, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
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 <i>A</i> ···O4 ⁱ	0.97	2.41	3.228 (4)	142
C14—H14 <i>B</i> ···O4 ⁱ	0.97	2.42	3.267 (4)	146

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