IUCrData

ISSN 2414-3146

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

Shaaban K. Mohamed,[a,b] Necmi Dege,[c] Mehmet Akkurt,[d] Omyma A. Abd Allah[e] and Mustafa R. Albayati[f]*

[a]Chemistry and Environmental Division, Manchester Metropolitan University, Manchester M1 5GD, England, [b]Chemistry Department, Faculty of Science, Minia University, 61519 El-Minia, Egypt, [c]University of Ondokuzmayıs, Science Faculty, Department of Physics, Samsun, Turkey, [d]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, [e]Chemistry Department, Faculty of Science, Sohag University, 82524 Sohag, Egypt, and [f]Kirkuk University, College of Education, Department of Chemistry, Kirkuk, Iraq. *Correspondence e-mail: shaabankamel@yahoo.com

Received 27 March 2017
Accepted 15 April 2017

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

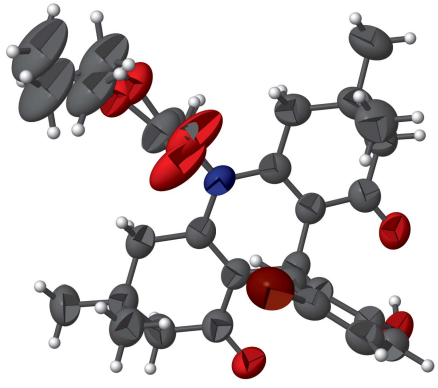
Keywords: crystal structure; acridines; acetic acid; hydrogen bonding; sofa conformation; disorder.

CCDC reference: 1530927

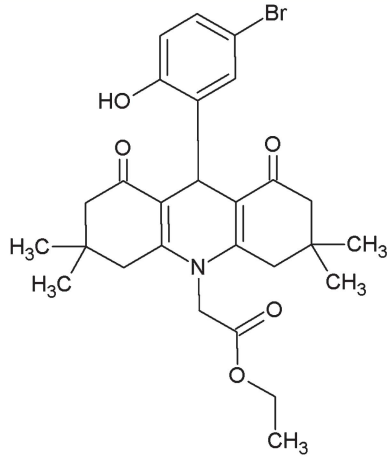
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{27}H_{32}BrNO_5$, the central 1,4-dihydropyridine ring adopts a shallow sofa conformation (with the C atom bearing the bromophenol ring as the flap), whereas the pendant cyclohexene rings both have twisted-boat conformations. The molecule features an intramolecular O—H···O hydrogen bond, which closes an $S(8)$ ring. In the crystal, molecules are linked by C—H···O interactions, forming $C(12)$ chains along the c-axis direction. The ethyl acetate grouping is disordered over two sets of sites in a 0.719 (11):0.281 (11) ratio.

3D view



Chemical scheme



Structure description

Acridine derivatives have been used as anti-malarial (Santelli-Rouvier *et al.*, 2004) anti-bacterial (Wainwright, 2001), anti-leishmanial (Delmas *et al.*, 2004) and anti-HIV (Hamy *et al.*, 1998) agents. They also have exhibited excellent results in chemotherapy of cancer (Cholody *et al.*, 1996; Rewcastle *et al.*, 1986). As part of our studies in this area, we report herein the synthesis and crystal structure of the title compound (Fig. 1).

The central 1,4-dihydropyridine ring (N1/C7/C8/C13/C16/C17) of the 1,2,3,4,5,6,7,8,9,10-decahydroacridine ring system (N1/C7–C13/C16–C21) adopts a shallow sofa conformation [the puckering parameters are Q_T = 0.229 (3) Å, θ = 66.4 (8)°, φ = 191.6 (8)°], while the cyclohexene rings (C8–C13 and C16–C21) 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 = 0.472 (4) Å, θ = 123.9 (4)°, φ = 341.3 (5)° and Q_T =

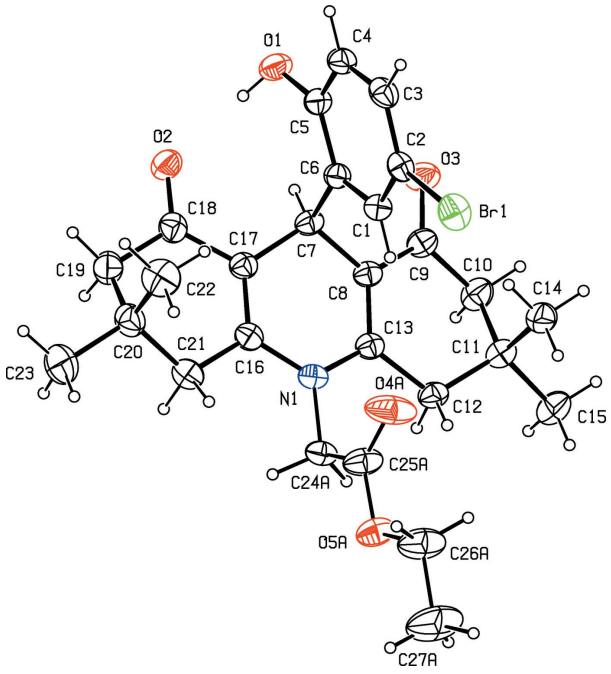


Figure 1
View of the title compound, with displacement ellipsoids for non-H atoms drawn at the 20% probability level. The minor component is not shown for clarity.

0.501 (4) Å, θ = 119.7 (5)°, φ = 46.4 (5)°, respectively]. The bond lengths and bond angles in the title compound are normal and comparable to those observed in similar structures (Akkurt *et al.*, 2015).

In the crystal, adjacent molecules are connected by C—H···O interactions, forming $C(12)$ chains along the *c*-axis direction (Table 1, Fig. 2).

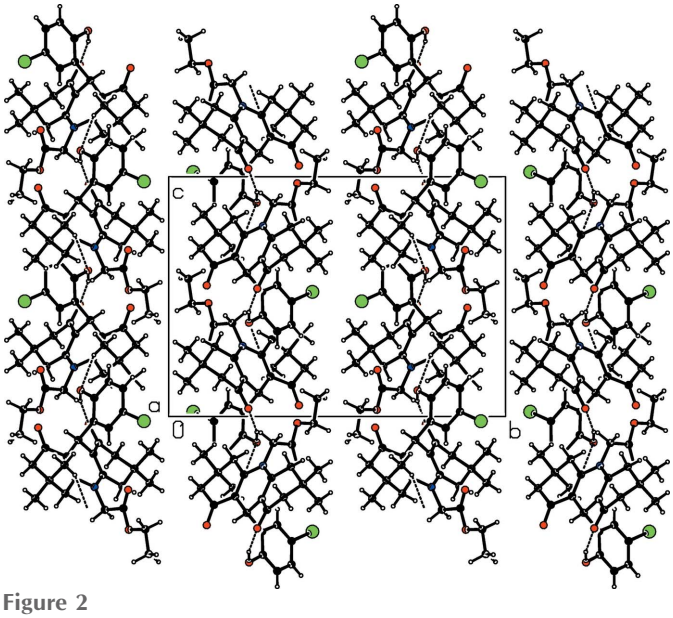


Figure 2
A view along the *a* axis of the hydrogen bonding and crystal packing of the title compound. The minor component of the disorder is not shown for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2	0.83 (4)	1.87 (4)	2.674 (3)	164 (4)
C14—H14A···O1^{i}	0.96	2.53	3.438 (4)	158

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{27}H_{32}BrNO_5$
M_r	530.44
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	10.1174 (5), 19.0567 (8), 14.0387 (7)
β (°)	105.254 (4)
V (Å^3)	2611.4 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.61
Crystal size (mm)	0.67 × 0.51 × 0.38
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2002)
T_{min}, T_{max}	0.425, 0.620
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15096, 5086, 3086
R_{int}	0.058
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.616
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.105, 0.97
No. of reflections	5086
No. of parameters	335
No. of restraints	8
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.27

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2002), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

Synthesis and crystallization

A mixture of dimedone (1.12 g, 0.008 mol), 5-bromo-2-hydroxybenzaldhyde (0.81 g; 0.004 mol), ethyl glycinate hydrochloride (0.56 g; 0.004 mol) and triethylamine (1.12 ml; 0.008 mol) in 30 ml ethanol was refluxed for 5 h. The solid product was deposited on cooling and collected by filtration under vacuum. Recrystallization of the crude product from ethanol afforded light-yellow prisms in 58% yield (m.p. 485 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The title molecule exhibits disorder of the ethyl acetate group. The atoms of the ethyl acetate group were fixed with restraints (EADP and SADI commands) and had to be split over two positions. The occupancies refined to 0.719 (11):0.281 (11).

Acknowledgements

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS 2 diffractometer (purchased under grant F.279 of the University Research Fund).

References

Akkurt, M., Jasinski, J. P., Mohamed, S. K., Allah, O. A. A., Tamam, A. H. A. & Albayati, M. R. (2015). *Acta Cryst.* E**71**, o963–o964.
Cholody, W., Horowska, B., Paradziej-Lukowicz, J., Martelli, S. & Konopa, J. (1996). *J. Med. Chem.* **39**, 1028–1032.
Delmas, F., Avellaneda, A., Di Giorgio, C., Robin, M., De Clercq, E., Timon-David, P. & Galy, J. P. (2004). *Eur. J. Med. Chem.* **39**, 685–690.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Hamy, F., Brondani, V., Flörsheimer, A., Stark, W., Blommers, M. J. J. & Klimkait, T. (1998). *Biochemistry*, **37**, 5086–5095.
Rewcastle, G., Atwell, G. J., Chambers, D., Baguley, B. C. & Denny, W. A. (1986). *J. Med. Chem.* **29**, 472–477.
Santelli-Rouvier, C., Pradines, B., Berthelot, M., Parzy, D. & Barbe, J. (2004). *Eur. J. Med. Chem.* **39**, 735–744.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.
Wainwright, M. J. (2001). *J. Antimicrob. Chemother.* **47**, 1–13.

full crystallographic data

IUCrData (2017). **2**, x170573 [https://doi.org/10.1107/S2414314617005739]

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

Shaaban K. Mohamed, Necmi Dege, Mehmet Akkurt, Omyma A. Abd Allah and Mustafa R. Albayati

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

Crystal data

$C_{27}H_{32}BrNO_5$
M_r = 530.44
Monoclinic, $P2_1/n$
a = 10.1174 (5) Å
b = 19.0567 (8) Å
c = 14.0387 (7) Å
β = 105.254 (4)°
V = 2611.4 (2) Å^3
Z = 4
$F(000)$ = 1104
D_x = 1.349 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 14005 reflections
θ = 1.5–26.5°
μ = 1.61 mm^{-1}
T = 296 K
Prism, light yellow
0.67 × 0.51 × 0.38 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)
T_{min} = 0.425, T_{max} = 0.620
15096 measured reflections
5086 independent reflections
3086 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 26.0°, θ_{min} = 1.9°
h = −12→12
k = −21→23
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.105
S = 0.97
5086 reflections
335 parameters
8 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.6851 (3)	0.67758 (15)	0.4523 (2)	0.0586 (7)	
H1	0.6899	0.6767	0.3870	0.070*	
C2	0.7688 (3)	0.63487 (16)	0.5199 (2)	0.0633 (7)	
C3	0.7638 (3)	0.63521 (17)	0.6172 (2)	0.0709 (8)	
H3	0.8229	0.6070	0.6635	0.085*	
C4	0.6711 (3)	0.67753 (18)	0.6447 (2)	0.0691 (8)	
H4	0.6663	0.6774	0.7100	0.083*	
C5	0.5848 (3)	0.72029 (16)	0.5770 (2)	0.0624 (7)	
C6	0.5930 (3)	0.72224 (15)	0.47920 (19)	0.0556 (7)	
C7	0.4953 (3)	0.76780 (15)	0.40186 (19)	0.0576 (7)	
H7	0.4575	0.8044	0.4361	0.069*	
C8	0.5669 (3)	0.80239 (15)	0.33324 (19)	0.0573 (7)	
C9	0.6446 (3)	0.86548 (16)	0.3689 (2)	0.0678 (8)	
C10	0.7075 (4)	0.90358 (18)	0.2990 (3)	0.0831 (10)	
H10A	0.7826	0.9323	0.3362	0.100*	
H10B	0.6398	0.9345	0.2580	0.100*	
C11	0.7607 (3)	0.85355 (17)	0.2335 (2)	0.0711 (8)	
C12	0.6438 (3)	0.80649 (17)	0.17857 (19)	0.0651 (8)	
H12A	0.5841	0.8334	0.1258	0.078*	
H12B	0.6817	0.7682	0.1485	0.078*	
C13	0.5594 (3)	0.77610 (15)	0.24226 (19)	0.0579 (7)	
C14	0.8774 (4)	0.8090 (2)	0.2974 (2)	0.0864 (10)	
H14A	0.9124	0.7782	0.2558	0.130*	
H14B	0.8432	0.7819	0.3434	0.130*	
H14C	0.9494	0.8393	0.3329	0.130*	
C15	0.8154 (4)	0.8958 (2)	0.1583 (3)	0.0936 (11)	
H15A	0.8504	0.8641	0.1177	0.140*	
H15B	0.8874	0.9264	0.1929	0.140*	
H15C	0.7424	0.9231	0.1176	0.140*	
C16	0.3809 (3)	0.69497 (15)	0.2560 (2)	0.0597 (7)	
C17	0.3801 (3)	0.72313 (15)	0.3446 (2)	0.0599 (7)	
C18	0.2692 (3)	0.70791 (17)	0.3893 (2)	0.0681 (8)	
C19	0.1600 (4)	0.6585 (2)	0.3369 (3)	0.0856 (10)	
H19A	0.0894	0.6846	0.2902	0.103*	
H19B	0.1184	0.6369	0.3845	0.103*	
C20	0.2158 (4)	0.6014 (2)	0.2822 (3)	0.0835 (10)	
C21	0.2848 (3)	0.63714 (17)	0.2098 (2)	0.0748 (9)	
H21A	0.3352	0.6022	0.1834	0.090*	
H21B	0.2145	0.6562	0.1551	0.090*	

C22	0.3184 (5)	0.55518 (19)	0.3538 (3)	0.1046 (12)	
H22A	0.3510	0.5193	0.3178	0.157*	
H22B	0.2746	0.5339	0.3996	0.157*	
H22C	0.3941	0.5832	0.3894	0.157*	
C23	0.0952 (5)	0.5564 (3)	0.2230 (3)	0.1238 (16)	
H23A	0.0337	0.5853	0.1751	0.186*	
H23B	0.0473	0.5365	0.2671	0.186*	
H23C	0.1297	0.5194	0.1897	0.186*	
C24	0.454 (5)	0.695 (3)	0.1031 (19)	0.065 (2)	0.281 (11)
H24A	0.4673	0.7323	0.0601	0.078*	0.281 (11)
H24B	0.3628	0.6749	0.0771	0.078*	0.281 (11)
C25	0.562 (10)	0.639 (3)	0.114 (2)	0.085 (4)	0.281 (11)
C26	0.641 (2)	0.5489 (14)	0.004 (2)	0.120 (4)	0.281 (11)
H26A	0.6698	0.5160	0.0585	0.144*	0.281 (11)
H26B	0.5872	0.5235	−0.0527	0.144*	0.281 (11)
C27	0.760 (3)	0.5801 (14)	−0.019 (2)	0.174 (4)	0.281 (11)
H27A	0.8091	0.6083	0.0362	0.261*	0.281 (11)
H27B	0.7316	0.6089	−0.0763	0.261*	0.281 (11)
H27C	0.8195	0.5436	−0.0304	0.261*	0.281 (11)
O4	0.604 (2)	0.5960 (14)	0.1773 (15)	0.132 (3)	0.281 (11)
O5	0.556 (3)	0.6046 (13)	0.032 (2)	0.089 (2)	0.281 (11)
C24A	0.4786 (15)	0.6889 (11)	0.1105 (7)	0.065 (2)	0.719 (11)
H24C	0.4938	0.7264	0.0679	0.078*	0.719 (11)
H24D	0.3901	0.6683	0.0797	0.078*	0.719 (11)
C25A	0.587 (3)	0.6342 (13)	0.1170 (7)	0.085 (4)	0.719 (11)
C26A	0.7064 (12)	0.5693 (6)	0.0292 (6)	0.120 (4)	0.719 (11)
H26D	0.6916	0.5251	0.0590	0.144*	0.719 (11)
H26E	0.7930	0.5889	0.0667	0.144*	0.719 (11)
C27A	0.7075 (12)	0.5587 (6)	−0.0742 (7)	0.174 (4)	0.719 (11)
H27D	0.6313	0.5298	−0.1066	0.261*	0.719 (11)
H27E	0.7914	0.5362	−0.0767	0.261*	0.719 (11)
H27F	0.7005	0.6033	−0.1070	0.261*	0.719 (11)
O4A	0.6676 (9)	0.6200 (5)	0.1941 (5)	0.132 (3)	0.719 (11)
O5A	0.5942 (10)	0.6180 (4)	0.0276 (8)	0.089 (2)	0.719 (11)
Br1	0.89080 (4)	0.57315 (2)	0.48022 (3)	0.08792 (16)	
N1	0.4734 (2)	0.71957 (12)	0.20561 (15)	0.0603 (6)	
O1	0.4932 (2)	0.76108 (14)	0.60854 (15)	0.0786 (7)	
H1A	0.420 (4)	0.762 (2)	0.565 (3)	0.118*	
O2	0.2634 (2)	0.73668 (13)	0.46674 (17)	0.0816 (6)	
O3	0.6568 (3)	0.88686 (13)	0.45337 (17)	0.0911 (7)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0626 (18)	0.0575 (17)	0.0548 (15)	0.0010 (15)	0.0141 (14)	−0.0010 (14)
C2	0.0578 (18)	0.0571 (18)	0.0705 (19)	−0.0048 (15)	0.0087 (15)	−0.0001 (15)
C3	0.067 (2)	0.067 (2)	0.068 (2)	−0.0044 (17)	−0.0001 (16)	0.0139 (16)
C4	0.073 (2)	0.080 (2)	0.0511 (16)	−0.0107 (18)	0.0110 (16)	0.0057 (16)

C5	0.0654 (19)	0.0663 (18)	0.0564 (17)	−0.0062 (16)	0.0179 (15)	−0.0036 (15)
C6	0.0579 (17)	0.0582 (17)	0.0498 (15)	−0.0010 (14)	0.0124 (13)	0.0000 (13)
C7	0.0654 (18)	0.0585 (17)	0.0518 (15)	0.0095 (15)	0.0203 (14)	0.0009 (13)
C8	0.0649 (18)	0.0553 (17)	0.0521 (15)	0.0086 (14)	0.0158 (14)	0.0039 (13)
C9	0.077 (2)	0.0612 (19)	0.0700 (19)	0.0064 (16)	0.0273 (17)	−0.0022 (15)
C10	0.100 (3)	0.067 (2)	0.093 (2)	−0.0124 (19)	0.044 (2)	−0.0077 (18)
C11	0.079 (2)	0.0732 (19)	0.0671 (18)	0.0019 (18)	0.0300 (18)	0.0015 (16)
C12	0.073 (2)	0.0708 (19)	0.0525 (16)	0.0077 (16)	0.0182 (15)	0.0091 (14)
C13	0.0614 (17)	0.0580 (17)	0.0531 (16)	0.0110 (15)	0.0129 (13)	0.0073 (13)
C14	0.071 (2)	0.116 (3)	0.073 (2)	0.008 (2)	0.0203 (18)	0.004 (2)
C15	0.108 (3)	0.093 (3)	0.093 (2)	−0.006 (2)	0.050 (2)	0.009 (2)
C16	0.0594 (18)	0.0567 (17)	0.0592 (17)	0.0073 (14)	0.0086 (15)	0.0047 (14)
C17	0.0608 (18)	0.0624 (18)	0.0549 (16)	0.0063 (15)	0.0127 (14)	0.0052 (14)
C18	0.0604 (19)	0.070 (2)	0.071 (2)	0.0071 (16)	0.0129 (16)	0.0098 (16)
C19	0.070 (2)	0.099 (3)	0.086 (2)	−0.004 (2)	0.0196 (19)	0.010 (2)
C20	0.082 (2)	0.077 (2)	0.087 (2)	−0.018 (2)	0.015 (2)	0.0003 (19)
C21	0.077 (2)	0.069 (2)	0.0719 (19)	0.0017 (18)	0.0086 (17)	−0.0008 (16)
C22	0.125 (3)	0.075 (3)	0.110 (3)	0.000 (2)	0.025 (3)	0.018 (2)
C23	0.116 (3)	0.120 (4)	0.134 (4)	−0.051 (3)	0.030 (3)	−0.027 (3)
C24	0.073 (6)	0.069 (4)	0.047 (2)	0.011 (4)	0.007 (3)	−0.003 (3)
C25	0.114 (13)	0.084 (4)	0.061 (2)	0.035 (4)	0.028 (3)	0.003 (2)
C26	0.155 (9)	0.126 (8)	0.081 (5)	0.065 (7)	0.034 (6)	−0.008 (4)
C27	0.230 (10)	0.192 (9)	0.119 (7)	0.085 (8)	0.081 (8)	−0.032 (6)
O4	0.156 (7)	0.158 (7)	0.063 (3)	0.090 (6)	−0.003 (4)	−0.005 (3)
O5	0.117 (6)	0.088 (4)	0.0712 (17)	0.020 (4)	0.042 (3)	−0.004 (3)
C24A	0.073 (6)	0.069 (4)	0.047 (2)	0.011 (4)	0.007 (3)	−0.003 (3)
C25A	0.114 (13)	0.084 (4)	0.061 (2)	0.035 (4)	0.028 (3)	0.003 (2)
C26A	0.155 (9)	0.126 (8)	0.081 (5)	0.065 (7)	0.034 (6)	−0.008 (4)
C27A	0.230 (10)	0.192 (9)	0.119 (7)	0.085 (8)	0.081 (8)	−0.032 (6)
O4A	0.156 (7)	0.158 (7)	0.063 (3)	0.090 (6)	−0.003 (4)	−0.005 (3)
O5A	0.117 (6)	0.088 (4)	0.0712 (17)	0.020 (4)	0.042 (3)	−0.004 (3)
Br1	0.0766 (2)	0.0737 (2)	0.1095 (3)	0.0167 (2)	0.01751 (19)	−0.0036 (2)
N1	0.0684 (15)	0.0621 (15)	0.0479 (12)	0.0061 (13)	0.0113 (12)	−0.0024 (11)
O1	0.0881 (16)	0.0931 (16)	0.0612 (13)	0.0074 (15)	0.0313 (12)	−0.0061 (12)
O2	0.0757 (15)	0.1000 (17)	0.0752 (14)	0.0071 (13)	0.0309 (12)	0.0005 (13)
O3	0.121 (2)	0.0860 (16)	0.0742 (14)	−0.0224 (15)	0.0390 (14)	−0.0258 (13)

Geometric parameters (Å, º)

C1—C2	1.363 (4)	C19—C20	1.524 (5)
C1—C6	1.386 (4)	C19—H19A	0.9700
C1—H1	0.9300	C19—H19B	0.9700
C2—C3	1.380 (4)	C20—C22	1.522 (5)
C2—Br1	1.892 (3)	C20—C21	1.534 (4)
C3—C4	1.368 (4)	C20—C23	1.544 (5)
C3—H3	0.9300	C21—H21A	0.9700
C4—C5	1.376 (4)	C21—H21B	0.9700
C4—H4	0.9300	C22—H22A	0.9600

C5—O1	1.369 (4)	C22—H22B	0.9600
C5—C6	1.398 (4)	C22—H22C	0.9600
C6—C7	1.531 (4)	C23—H23A	0.9600
C7—C17	1.496 (4)	C23—H23B	0.9600
C7—C8	1.501 (4)	C23—H23C	0.9600
C7—H7	0.9800	C24—N1	1.479 (15)
C8—C13	1.355 (4)	C24—C25	1.498 (16)
C8—C9	1.451 (4)	C24—H24A	0.9700
C9—O3	1.229 (3)	C24—H24B	0.9700
C9—C10	1.491 (4)	C25—O4	1.21 (2)
C10—C11	1.518 (4)	C25—O5	1.322 (18)
C10—H10A	0.9700	C26—C27	1.458 (18)
C10—H10B	0.9700	C26—O5	1.476 (16)
C11—C12	1.522 (4)	C26—H26A	0.9700
C11—C14	1.536 (5)	C26—H26B	0.9700
C11—C15	1.541 (4)	C27—H27A	0.9600
C12—C13	1.505 (4)	C27—H27B	0.9600
C12—H12A	0.9700	C27—H27C	0.9600
C12—H12B	0.9700	C24A—N1	1.470 (7)
C13—N1	1.395 (4)	C24A—C25A	1.496 (7)
C14—H14A	0.9600	C24A—H24C	0.9700
C14—H14B	0.9600	C24A—H24D	0.9700
C14—H14C	0.9600	C25A—O4A	1.204 (14)
C15—H15A	0.9600	C25A—O5A	1.314 (9)
C15—H15B	0.9600	C26A—O5A	1.463 (7)
C15—H15C	0.9600	C26A—C27A	1.468 (10)
C16—C17	1.357 (4)	C26A—H26D	0.9700
C16—N1	1.394 (3)	C26A—H26E	0.9700
C16—C21	1.499 (4)	C27A—H27D	0.9600
C17—C18	1.452 (4)	C27A—H27E	0.9600
C18—O2	1.233 (4)	C27A—H27F	0.9600
C18—C19	1.489 (5)	O1—H1A	0.83 (4)

C2—C1—C6	121.0 (3)	C20—C19—H19B	109.2
C2—C1—H1	119.5	H19A—C19—H19B	107.9
C6—C1—H1	119.5	C22—C20—C19	111.0 (3)
C1—C2—C3	120.6 (3)	C22—C20—C21	110.2 (3)
C1—C2—Br1	120.0 (2)	C19—C20—C21	108.1 (3)
C3—C2—Br1	119.4 (2)	C22—C20—C23	109.8 (3)
C4—C3—C2	119.2 (3)	C19—C20—C23	108.9 (3)
C4—C3—H3	120.4	C21—C20—C23	108.8 (3)
C2—C3—H3	120.4	C16—C21—C20	113.3 (3)
C3—C4—C5	120.8 (3)	C16—C21—H21A	108.9
C3—C4—H4	119.6	C20—C21—H21A	108.9
C5—C4—H4	119.6	C16—C21—H21B	108.9
O1—C5—C4	118.2 (3)	C20—C21—H21B	108.9
O1—C5—C6	121.5 (3)	H21A—C21—H21B	107.7
C4—C5—C6	120.2 (3)	C20—C22—H22A	109.5

C1—C6—C5	118.0 (3)	C20—C22—H22B	109.5
C1—C6—C7	120.6 (2)	H22A—C22—H22B	109.5
C5—C6—C7	121.2 (2)	C20—C22—H22C	109.5
C17—C7—C8	110.1 (2)	H22A—C22—H22C	109.5
C17—C7—C6	109.0 (2)	H22B—C22—H22C	109.5
C8—C7—C6	112.0 (2)	C20—C23—H23A	109.5
C17—C7—H7	108.6	C20—C23—H23B	109.5
C8—C7—H7	108.6	H23A—C23—H23B	109.5
C6—C7—H7	108.6	C20—C23—H23C	109.5
C13—C8—C9	121.5 (2)	H23A—C23—H23C	109.5
C13—C8—C7	121.7 (3)	H23B—C23—H23C	109.5
C9—C8—C7	116.8 (2)	N1—C24—C25	103 (2)
O3—C9—C8	121.0 (3)	N1—C24—H24A	111.2
O3—C9—C10	121.4 (3)	C25—C24—H24A	111.2
C8—C9—C10	117.6 (3)	N1—C24—H24B	111.2
C9—C10—C11	111.9 (3)	C25—C24—H24B	111.3
C9—C10—H10A	109.2	H24A—C24—H24B	109.2
C11—C10—H10A	109.2	O4—C25—O5	103 (4)
C9—C10—H10B	109.2	O4—C25—C24	131 (6)
C11—C10—H10B	109.2	O5—C25—C24	114 (3)
H10A—C10—H10B	107.9	C27—C26—O5	110 (2)
C10—C11—C12	109.0 (3)	C27—C26—H26A	109.8
C10—C11—C14	109.5 (3)	O5—C26—H26A	109.8
C12—C11—C14	110.2 (3)	C27—C26—H26B	109.8
C10—C11—C15	109.6 (3)	O5—C26—H26B	109.8
C12—C11—C15	109.4 (3)	H26A—C26—H26B	108.2
C14—C11—C15	109.2 (3)	C26—C27—H27A	109.5
C13—C12—C11	114.3 (2)	C26—C27—H27B	109.5
C13—C12—H12A	108.7	H27A—C27—H27B	109.5
C11—C12—H12A	108.7	C26—C27—H27C	109.5
C13—C12—H12B	108.7	H27A—C27—H27C	109.5
C11—C12—H12B	108.7	H27B—C27—H27C	109.5
H12A—C12—H12B	107.6	C25—O5—C26	134 (3)
C8—C13—N1	120.5 (2)	N1—C24A—C25A	114.9 (8)
C8—C13—C12	121.2 (3)	N1—C24A—H24C	108.5
N1—C13—C12	118.3 (2)	C25A—C24A—H24C	108.5
C11—C14—H14A	109.5	N1—C24A—H24D	108.5
C11—C14—H14B	109.5	C25A—C24A—H24D	108.5
H14A—C14—H14B	109.5	H24C—C24A—H24D	107.5
C11—C14—H14C	109.5	O4A—C25A—O5A	127.5 (16)
H14A—C14—H14C	109.5	O4A—C25A—C24A	121.6 (10)
H14B—C14—H14C	109.5	O5A—C25A—C24A	109.3 (7)
C11—C15—H15A	109.5	O5A—C26A—C27A	106.3 (8)
C11—C15—H15B	109.5	O5A—C26A—H26D	110.5
H15A—C15—H15B	109.5	C27A—C26A—H26D	110.5
C11—C15—H15C	109.5	O5A—C26A—H26E	110.5
H15A—C15—H15C	109.5	C27A—C26A—H26E	110.5
H15B—C15—H15C	109.5	H26D—C26A—H26E	108.7

C17—C16—N1	119.9 (3)	C26A—C27A—H27D	109.5
C17—C16—C21	121.8 (3)	C26A—C27A—H27E	109.5
N1—C16—C21	118.3 (2)	H27D—C27A—H27E	109.5
C16—C17—C18	120.6 (3)	C26A—C27A—H27F	109.5
C16—C17—C7	121.7 (2)	H27D—C27A—H27F	109.5
C18—C17—C7	117.6 (2)	H27E—C27A—H27F	109.5
O2—C18—C17	120.9 (3)	C25A—O5A—C26A	112.0 (10)
O2—C18—C19	121.0 (3)	C16—N1—C13	120.6 (2)
C17—C18—C19	118.0 (3)	C16—N1—C24A	121.0 (9)
C18—C19—C20	112.0 (3)	C13—N1—C24A	118.3 (10)
C18—C19—H19A	109.2	C16—N1—C24	117 (2)
C20—C19—H19A	109.2	C13—N1—C24	121 (3)
C18—C19—H19B	109.2	C5—O1—H1A	109 (3)

C6—C1—C2—C3	−0.1 (4)	C6—C7—C17—C16	−96.9 (3)
C6—C1—C2—Br1	178.7 (2)	C8—C7—C17—C18	−156.6 (2)
C1—C2—C3—C4	1.8 (4)	C6—C7—C17—C18	80.2 (3)
Br1—C2—C3—C4	−177.0 (2)	C16—C17—C18—O2	−175.5 (3)
C2—C3—C4—C5	−1.0 (5)	C7—C17—C18—O2	7.3 (4)
C3—C4—C5—O1	179.4 (3)	C16—C17—C18—C19	2.3 (4)
C3—C4—C5—C6	−1.5 (5)	C7—C17—C18—C19	−174.8 (3)
C2—C1—C6—C5	−2.3 (4)	O2—C18—C19—C20	−148.7 (3)
C2—C1—C6—C7	−177.5 (3)	C17—C18—C19—C20	33.4 (4)
O1—C5—C6—C1	−177.8 (3)	C18—C19—C20—C22	63.8 (4)
C4—C5—C6—C1	3.1 (4)	C18—C19—C20—C21	−57.2 (4)
O1—C5—C6—C7	−2.7 (4)	C18—C19—C20—C23	−175.3 (3)
C4—C5—C6—C7	178.2 (3)	C17—C16—C21—C20	−15.0 (4)
C1—C6—C7—C17	77.5 (3)	N1—C16—C21—C20	164.7 (3)
C5—C6—C7—C17	−97.5 (3)	C22—C20—C21—C16	−73.3 (4)
C1—C6—C7—C8	−44.6 (4)	C19—C20—C21—C16	48.2 (4)
C5—C6—C7—C8	140.4 (3)	C23—C20—C21—C16	166.3 (3)
C17—C7—C8—C13	−22.0 (4)	N1—C24—C25—O4	39 (12)
C6—C7—C8—C13	99.3 (3)	N1—C24—C25—O5	174 (6)
C17—C7—C8—C9	157.9 (3)	O4—C25—O5—C26	−35 (11)
C6—C7—C8—C9	−80.7 (3)	C24—C25—O5—C26	177 (4)
C13—C8—C9—O3	−176.1 (3)	C27—C26—O5—C25	−82 (7)
C7—C8—C9—O3	4.0 (4)	N1—C24A—C25A—O4A	−4 (5)
C13—C8—C9—C10	4.5 (4)	N1—C24A—C25A—O5A	−171.2 (18)
C7—C8—C9—C10	−175.5 (3)	O4A—C25A—O5A—C26A	9 (4)
O3—C9—C10—C11	143.5 (3)	C24A—C25A—O5A—C26A	175 (2)
C8—C9—C10—C11	−37.1 (4)	C27A—C26A—O5A—C25A	−177 (2)
C9—C10—C11—C12	56.5 (4)	C17—C16—N1—C13	−4.2 (4)
C9—C10—C11—C14	−64.1 (4)	C21—C16—N1—C13	176.1 (3)
C9—C10—C11—C15	176.1 (3)	C17—C16—N1—C24A	178.7 (7)
C10—C11—C12—C13	−45.9 (4)	C21—C16—N1—C24A	−1.0 (8)
C14—C11—C12—C13	74.2 (3)	C17—C16—N1—C24	−170 (2)
C15—C11—C12—C13	−165.7 (3)	C21—C16—N1—C24	10 (2)
C9—C8—C13—N1	−173.6 (3)	C8—C13—N1—C16	8.4 (4)

C7—C8—C13—N1	6.3 (4)	C12—C13—N1—C16	−172.1 (2)
C9—C8—C13—C12	6.9 (4)	C8—C13—N1—C24A	−174.4 (7)
C7—C8—C13—C12	−173.2 (3)	C12—C13—N1—C24A	5.1 (7)
C11—C12—C13—C8	15.2 (4)	C8—C13—N1—C24	174 (2)
C11—C12—C13—N1	−164.3 (3)	C12—C13—N1—C24	−7 (2)
N1—C16—C17—C18	168.4 (3)	C25A—C24A—N1—C16	−97 (2)
C21—C16—C17—C18	−11.9 (4)	C25A—C24A—N1—C13	86 (3)
N1—C16—C17—C7	−14.6 (4)	C25—C24—N1—C16	−102 (5)
C21—C16—C17—C7	165.1 (3)	C25—C24—N1—C13	92 (6)
C8—C7—C17—C16	26.3 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2	0.83 (4)	1.87 (4)	2.674 (3)	164 (4)
C7—H7···O1	0.98	2.49	2.910 (3)	105
C14—H14*A*···O1^{i}	0.96	2.53	3.438 (4)	158

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