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2-[1-(4-Bromophenyl)-3-hydroxy-3-(4-methoxyphenyl)propyl]cyclohexanol

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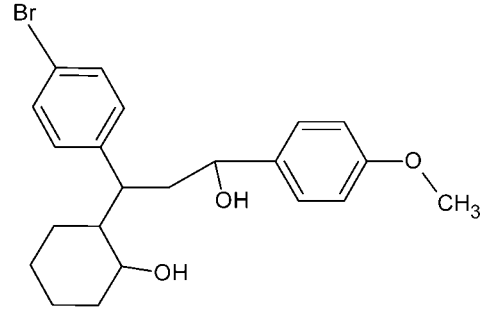
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.008 Å; R factor = 0.068; wR factor = 0.198; data-to-parameter ratio = 17.9.

In the title compound, $C_{22}H_{27}BrO_3$, the cyclohexane ring adopts a chair conformation. The dihedral angle between the benzene rings is 41.9 (4)°. In the crystal, molecules are linked by O—H···O and C—H···O hydrogen bonds, forming a three-dimensional network. In addition, π–π stacking interactions [centroid–centroid distance = 3.953 (6) Å] between the benzene rings of the methoxybenzene groups occur.

Related literature

For the biological properties of 1,5-diols, see: Flamme & Roush (2005); Hansen *et al.* (2003); Huang *et al.* (2009); Oger *et al.* (2010). For details of the synthesis, see: Ceylan & Gezegen (2008); Gezegen *et al.* (2010). For ring conformation analysis, see: Cremer & Pople (1975). For standard bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{22}H_{27}BrO_3$
M_r = 419.34
Monoclinic, $C2/c$
a = 23.2993 (14) Å
b = 10.9282 (5) Å
c = 22.3632 (11) Å
β = 133.032 (3)°
V = 4162.2 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.99 mm^{-1}
T = 296 K
0.60 × 0.34 × 0.28 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration [*X-RED32* (Stoe & Cie, 2002) and *XABS2* (Parkin *et al.*, 1995)]
T_{min} = 0.448, T_{max} = 0.572
4301 measured reflections
4301 independent reflections
2327 reflections with $I > 2\sigma(I)$
R_{int} = 0.000

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.198
S = 1.03
4301 reflections
240 parameters
149 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2^{i}	0.82	2.13	2.854 (4)	147
O2—H2A···O2^{ii}	0.78 (3)	2.46 (2)	2.871 (6)	115 (2)
C5—H5···O3^{iii}	0.93	2.36	3.287 (10)	171

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5331).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Ceylan, M. & Gezegen, H. (2008). *Turk. J. Chem.* **32**, 55–61.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Flamme, E. M. & Roush, W. R. (2005). *Beilstein J. Org. Chem.* **1**, 7. doi:10.1186/1860-5397-1-7.
Gezegen, H., Dingil, A. & Ceylan, M. (2010). *J. Heterocycl. Chem.* **47**, 1017–1024.
Hansen, T. M., Florence, G. J., Lugo-Mas, P., Chen, J., Abrams, J. N. & Forsyth, C. J. (2003). *Tetrahedron Lett.* **44**, 57–59.
Huang, K., Ortiz-Marciales, M., Jesus, M. D. & Stepanenko, V. (2009). *J. Heterocycl. Chem.* **46**, 1252–1258.
Oger, C., Marton, Z., Brinkmann, Y., Bultel-Ponce, V., Durand, T., Graber, M. & Galano, J.-M. (2010). *J. Org. Chem.* **75**, 1892–1897.

Parkin, S., Moezzi, B. & Hope, H. (1995). *J. Appl. Cryst.* **28**, 53–56.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.

supporting information

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2-[1-(4-Bromophenyl)-3-hydroxy-3-(4-methoxyphenyl)propyl]cyclohexanol

Ísmail Çelik, Mehmet Akkurt, Hayreddin Gezegen, Muhammed M. Üremiş and Narcis Duteanu

S1. Comment

1,5-Diols are useful and important compounds as intermediates in synthetic organic reactions (Huang *et al.*, 2009; Oger *et al.*, 2010). They are used in the synthesis of bioactive molecules and natural products (Flamme & Roush, 2005; Hansen *et al.*, 2003). In this paper we report synthesis of 2-(1-(4-bromophenyl)-3-hydroxy-3-(4-methoxyphenyl)propyl)cyclohexanol in moderate yield together with its crystal structure.

The cyclohexyl ring of the title compound (I, Fig. 1) adopts a chair conformation with the puckering parameters (Cremer & Pople, 1975) of Q_T = 0.564 (6) Å, θ = 2.7 (6) ° and φ = 12 (13)°. The two benzene rings (C1–C6 and C16–C21) make a dihedral angle of 41.9 (4)° with each other. The C6–C7–C8–C13, C6–C7–C14–C15, C7–C14–C15–O2 and C7–C14–C15–C16 torsion angles are 177.9 (4), 59.2 (6), 64.0 (5) and -171.5 (4)°, respectively. All bond lengths of (I) are within normal ranges (Allen *et al.*, 1987), as are the bond angles.

In the crystal, intermolecular O—H···O and C—H···O hydrogen bonds connect molecules, forming a three-dimensional network (Table 1, Fig. 2). Furthermore, π-π stacking interactions [*Cg*3···*Cg*3(2 - *x*, *y*, 1/2 - *z*) = 3.953 (6) Å, where *Cg*3 is a centroid of the C16–C21 benzene ring], between the benzene rings of the methoxybenzene groups, stabilize the crystal packing.

S2. Experimental

2-(1-(4-Bromophenyl)-3-hydroxy-3-(4-methoxyphenyl)propyl)cyclohexanol was synthesized from 2-(1-(4-bromophenyl)-3-(4-methoxyphenyl)-3-oxopropyl)cyclohexanone (1,5-diketone) (Ceylan & Gezegen, 2008; Gezegen *et al.*, 2010). To a solution of 2-(1-(4-bromophenyl)-3-(4-methoxyphenyl)-3-oxopropyl)cyclohexanone (1 mmol) in THF-MeOH (12 ml 5:1) was added $NaBH_4$ (3 mmol) and stirred for 16 h at room temperature. After completion of the reaction, the mixture was transferred to a separatory funnel, dilute HCl (10% 10 ml) was added and extracted with diethyl ether (3x20 ml). The organic layer was dried over Na_2SO_4 and evaporated. The crude product was crystallized from EtO_2-hexane (3:1) to give pure 2-(1-(4-bromophenyl)-3-hydroxy-3-(4-methoxyphenyl)propyl)cyclohexanol in 75% yield; m.p.: 470–472 K.

S3. Refinement

C-bound H atoms and the hydroxyl H atom H1A were positioned geometrically and refined using a riding model with O—H = 0.82 Å, C—H = 0.93 Å (aromatic), 0.96 Å (methyl), 0.97 Å (methylene) and 0.98 Å (methine), and with U_{iso}(H) = 1.5U_{eq}(C_{methyl}, $O_{hydroxyl}$), and U_{iso}(H) = 1.2U_{eq}($C_{aromatic,\ methylene,\ methine}$). The other hydroxyl H atom H2A was found in difference Fourier maps and the O2—H2A bond length were restrained to 0.83 (2) Å with U_{iso}(H) = 1.5U_{eq}(O). Twelve poorly fitted reflections (2 2 1), (0 2 2), (-25 3 19), (-9 3 7), (-10 2 20), (-13 7 5), (-6 2 6), (2 0 0), (-6 2 10), (10 6 2), (-9 11 3) and (-15 5 6) were omitted from the refinement owing to bad disagreement between Fo and Fc.

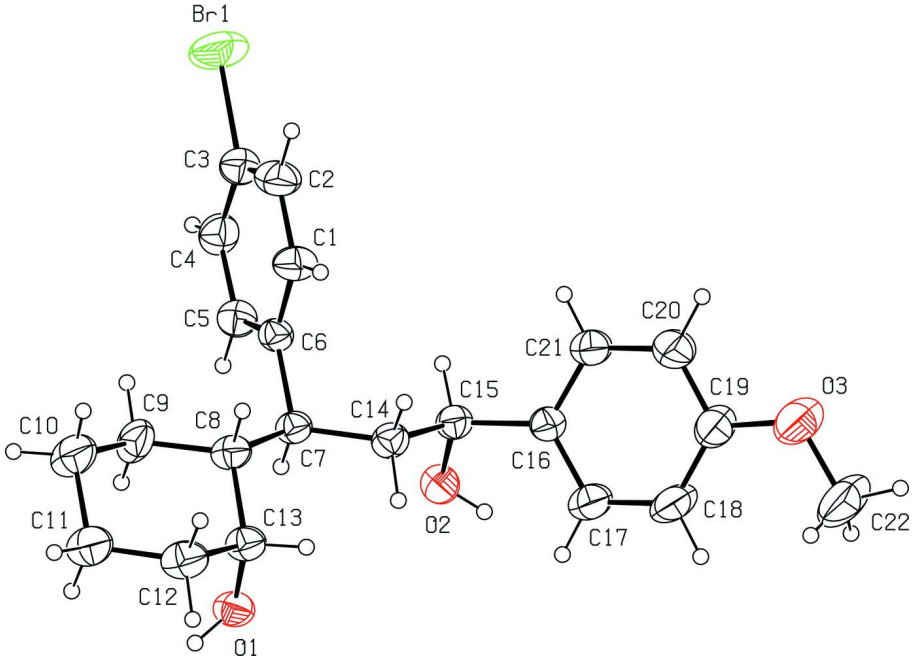

Figure 1

The title molecule with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

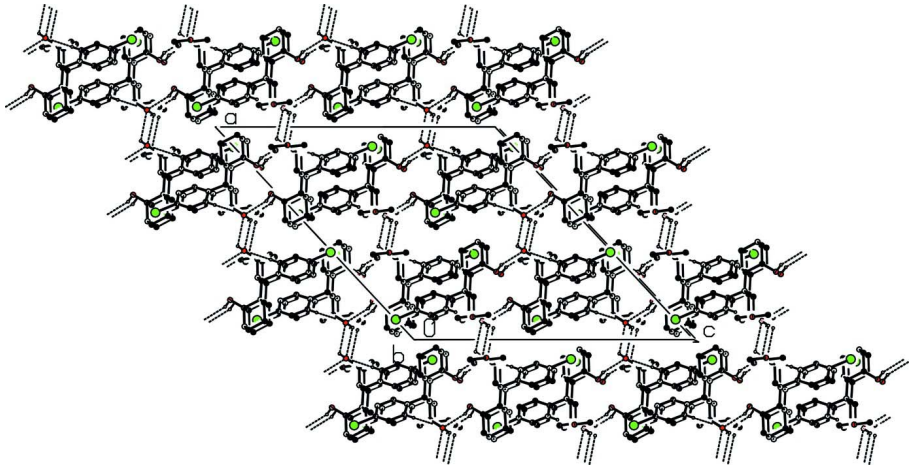

Figure 2

The packing and hydrogen bonding of the title compound viewed down the *b* axis. H atoms not involved in hydrogen bonding are omitted for clarity. Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_{22}H_{27}BrO_3$
$M_r = 419.34$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 23.2993\ (14)$ Å
$b = 10.9282\ (5)$ Å
$c = 22.3632\ (11)$ Å
$\beta = 133.032\ (3)°$
$V = 4162.2\ (4)$ Å^3
$Z = 8$

$F(000) = 1744$
$D_x = 1.338$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 538 reflections
$\theta = 1.8–28.0°$

$\mu = 1.99$ mm^{-1}
$T = 296$ K
Prism, colourless
0.60 × 0.34 × 0.28 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}
ω scans
Absorption correction: integration [*X-RED32* (Stoe & Cie, 2002) and *XABS2* (Parkin *et al.*, 1995)]

$T_{min} = 0.448$, $T_{max} = 0.572$
4301 measured reflections
4301 independent reflections
2327 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 26.5°$, $\theta_{min} = 2.0°$
$h = -29 \rightarrow 21$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.198$
$S = 1.03$
4301 reflections
240 parameters
149 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0945P)^2 + 0.8672P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.78$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.90669 (5)	0.12894 (8)	0.49681 (4)	0.1178 (4)
O1	0.68319 (18)	0.2009 (3)	−0.02218 (19)	0.0645 (10)
O2	0.91572 (17)	0.3614 (3)	0.19089 (19)	0.0594 (10)
O3	0.9041 (4)	0.9462 (3)	0.2060 (3)	0.130 (3)
C1	0.7850 (3)	0.2963 (4)	0.2729 (3)	0.0658 (16)
C2	0.8135 (3)	0.2666 (5)	0.3495 (3)	0.0762 (19)
C3	0.8642 (3)	0.1688 (5)	0.3903 (3)	0.0678 (16)
C4	0.8854 (3)	0.1024 (4)	0.3576 (3)	0.0664 (16)
C5	0.8566 (3)	0.1340 (4)	0.2812 (3)	0.0579 (16)

C6	0.8059 (2)	0.2320 (4)	0.2374 (2)	0.0479 (11)
C7	0.7740 (2)	0.2639 (4)	0.1536 (2)	0.0494 (12)
C8	0.6854 (2)	0.2325 (4)	0.0871 (2)	0.0548 (14)
C9	0.6696 (3)	0.0990 (5)	0.0910 (3)	0.0761 (16)
C10	0.5823 (3)	0.0706 (6)	0.0323 (3)	0.090 (2)
C11	0.5419 (3)	0.1067 (5)	−0.0550 (3)	0.0787 (18)
C12	0.5591 (3)	0.2377 (5)	−0.0597 (3)	0.0705 (16)
C13	0.6465 (2)	0.2654 (4)	0.0002 (2)	0.0565 (14)
C14	0.7910 (2)	0.3971 (4)	0.1491 (2)	0.0542 (12)
C15	0.8779 (2)	0.4304 (4)	0.2098 (2)	0.0541 (12)
C16	0.8874 (3)	0.5678 (4)	0.2089 (3)	0.0542 (12)
C17	0.8778 (4)	0.6232 (5)	0.1470 (4)	0.092 (3)
C18	0.8820 (5)	0.7492 (5)	0.1436 (4)	0.108 (3)
C19	0.8971 (4)	0.8204 (4)	0.2031 (4)	0.087 (3)
C20	0.9063 (4)	0.7675 (4)	0.2643 (4)	0.088 (2)
C21	0.9017 (3)	0.6423 (4)	0.2674 (3)	0.0690 (19)
C22	0.8947 (6)	1.0051 (5)	0.1428 (6)	0.146 (5)
H1	0.75060	0.36190	0.24470	0.0790*
H1A	0.65010	0.16080	−0.06370	0.0970*
H2	0.79880	0.31140	0.37260	0.0910*
H2A	0.9441 (7)	0.398 (3)	0.1907 (8)	0.0890*
H4	0.91900	0.03590	0.38580	0.0800*
H5	0.87170	0.08820	0.25880	0.0700*
H7	0.80120	0.21210	0.14380	0.0600*
H8	0.65930	0.28140	0.09940	0.0660*
H9A	0.69390	0.07980	0.14630	0.0910*
H9B	0.69360	0.04740	0.07780	0.0910*
H10A	0.55890	0.11540	0.04870	0.1080*
H10B	0.57480	−0.01610	0.03430	0.1080*
H11A	0.55980	0.05310	−0.07400	0.0950*
H11B	0.48570	0.09600	−0.09090	0.0950*
H12A	0.53430	0.29160	−0.04850	0.0850*
H12B	0.53620	0.25450	−0.11480	0.0850*
H13	0.65320	0.35340	−0.00170	0.0670*
H14A	0.76880	0.41340	0.09420	0.0650*
H14B	0.76450	0.44980	0.15880	0.0650*
H15	0.90090	0.40710	0.26470	0.0650*
H17	0.86820	0.57490	0.10680	0.1100*
H18	0.87460	0.78490	0.10100	0.1280*
H20	0.91570	0.81630	0.30430	0.1050*
H21	0.90830	0.60750	0.30980	0.0830*
H22A	0.93450	0.97650	0.14400	0.2190*
H22B	0.89960	1.09200	0.15120	0.2190*
H22C	0.84380	0.98630	0.09060	0.2190*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1390 (7)	0.1527 (7)	0.0648 (4)	−0.0010 (5)	0.0707 (5)	0.0173 (4)
O1	0.0527 (17)	0.092 (2)	0.0556 (17)	−0.0169 (16)	0.0396 (15)	−0.0152 (16)
O2	0.0511 (17)	0.0657 (18)	0.0653 (18)	−0.0043 (14)	0.0412 (15)	−0.0047 (14)
O3	0.260 (6)	0.060 (2)	0.194 (5)	0.006 (3)	0.204 (5)	0.002 (3)
C1	0.075 (3)	0.075 (3)	0.060 (2)	0.008 (2)	0.051 (3)	0.002 (2)
C2	0.095 (4)	0.084 (3)	0.071 (3)	0.001 (3)	0.065 (3)	−0.008 (3)
C3	0.071 (3)	0.076 (3)	0.053 (2)	−0.011 (2)	0.041 (2)	−0.003 (2)
C4	0.062 (3)	0.066 (3)	0.057 (2)	0.003 (2)	0.035 (2)	0.010 (2)
C5	0.055 (3)	0.063 (3)	0.056 (2)	0.002 (2)	0.038 (2)	0.000 (2)
C6	0.042 (2)	0.056 (2)	0.0412 (19)	−0.0040 (17)	0.0266 (18)	−0.0032 (17)
C7	0.046 (2)	0.060 (2)	0.045 (2)	−0.0022 (18)	0.0321 (19)	−0.0002 (18)
C8	0.040 (2)	0.078 (3)	0.046 (2)	−0.003 (2)	0.0292 (18)	0.000 (2)
C9	0.061 (3)	0.091 (3)	0.056 (2)	−0.026 (3)	0.032 (2)	0.006 (2)
C10	0.061 (3)	0.139 (5)	0.057 (3)	−0.037 (3)	0.035 (3)	−0.002 (3)
C11	0.048 (3)	0.125 (4)	0.055 (2)	−0.022 (3)	0.032 (2)	−0.005 (3)
C12	0.046 (2)	0.106 (4)	0.050 (2)	0.001 (2)	0.029 (2)	0.005 (2)
C13	0.045 (2)	0.075 (3)	0.046 (2)	−0.0012 (19)	0.0297 (19)	0.0007 (19)
C14	0.053 (2)	0.058 (2)	0.048 (2)	0.0022 (19)	0.033 (2)	0.0056 (18)
C15	0.053 (2)	0.062 (2)	0.047 (2)	−0.005 (2)	0.034 (2)	−0.0005 (19)
C16	0.057 (2)	0.059 (2)	0.053 (2)	−0.006 (2)	0.040 (2)	−0.0039 (19)
C17	0.159 (6)	0.068 (3)	0.101 (4)	−0.007 (3)	0.109 (4)	−0.002 (3)
C18	0.209 (7)	0.065 (3)	0.135 (5)	0.006 (4)	0.151 (6)	0.013 (3)
C19	0.147 (6)	0.053 (3)	0.127 (5)	0.002 (3)	0.119 (5)	−0.001 (3)
C20	0.140 (5)	0.063 (3)	0.099 (4)	−0.005 (3)	0.097 (4)	−0.011 (3)
C21	0.093 (4)	0.064 (3)	0.068 (3)	0.001 (2)	0.062 (3)	0.000 (2)
C22	0.287 (12)	0.068 (4)	0.227 (9)	0.018 (5)	0.232 (10)	0.028 (5)

Geometric parameters (Å, º)

Br1—C3	1.904 (5)	C19—C20	1.361 (11)
O1—C13	1.436 (7)	C20—C21	1.378 (6)
O2—C15	1.426 (7)	C1—H1	0.9300
O3—C19	1.381 (6)	C2—H2	0.9300
O3—C22	1.427 (13)	C4—H4	0.9300
O1—H1A	0.8200	C5—H5	0.9300
O2—H2A	0.78 (3)	C7—H7	0.9800
C1—C2	1.389 (8)	C8—H8	0.9800
C1—C6	1.374 (9)	C9—H9A	0.9700
C2—C3	1.378 (8)	C9—H9B	0.9700
C3—C4	1.341 (10)	C10—H10A	0.9700
C4—C5	1.387 (8)	C10—H10B	0.9700
C5—C6	1.387 (7)	C11—H11A	0.9700
C6—C7	1.512 (5)	C11—H11B	0.9700
C7—C8	1.550 (6)	C12—H12A	0.9700
C7—C14	1.530 (6)	C12—H12B	0.9700

C8—C9	1.522 (7)	C13—H13	0.9800
C8—C13	1.524 (5)	C14—H14A	0.9700
C9—C10	1.521 (10)	C14—H14B	0.9700
C10—C11	1.531 (7)	C15—H15	0.9800
C11—C12	1.510 (8)	C17—H17	0.9300
C12—C13	1.520 (8)	C18—H18	0.9300
C14—C15	1.524 (7)	C20—H20	0.9300
C15—C16	1.520 (6)	C21—H21	0.9300
C16—C21	1.375 (8)	C22—H22A	0.9600
C16—C17	1.382 (10)	C22—H22B	0.9600
C17—C18	1.386 (8)	C22—H22C	0.9600
C18—C19	1.364 (10)		

C19—O3—C22	118.0 (6)	C14—C7—H7	107.00
C13—O1—H1A	110.00	C7—C8—H8	107.00
C15—O2—H2A	116 (2)	C9—C8—H8	107.00
C2—C1—C6	122.2 (5)	C13—C8—H8	107.00
C1—C2—C3	118.1 (6)	C8—C9—H9A	109.00
Br1—C3—C2	119.0 (5)	C8—C9—H9B	109.00
Br1—C3—C4	119.2 (4)	C10—C9—H9A	109.00
C2—C3—C4	121.8 (5)	C10—C9—H9B	109.00
C3—C4—C5	119.2 (5)	H9A—C9—H9B	108.00
C4—C5—C6	121.7 (6)	C9—C10—H10A	110.00
C5—C6—C7	121.0 (5)	C9—C10—H10B	110.00
C1—C6—C5	117.0 (4)	C11—C10—H10A	110.00
C1—C6—C7	122.0 (4)	C11—C10—H10B	110.00
C6—C7—C14	111.8 (3)	H10A—C10—H10B	108.00
C6—C7—C8	110.2 (4)	C10—C11—H11A	109.00
C8—C7—C14	112.5 (3)	C10—C11—H11B	109.00
C7—C8—C13	114.5 (4)	C12—C11—H11A	109.00
C7—C8—C9	111.9 (4)	C12—C11—H11B	109.00
C9—C8—C13	110.0 (3)	H11A—C11—H11B	108.00
C8—C9—C10	112.3 (5)	C11—C12—H12A	109.00
C9—C10—C11	110.1 (6)	C11—C12—H12B	109.00
C10—C11—C12	111.8 (4)	C13—C12—H12A	109.00
C11—C12—C13	112.8 (5)	C13—C12—H12B	109.00
O1—C13—C12	110.7 (4)	H12A—C12—H12B	108.00
O1—C13—C8	110.6 (4)	O1—C13—H13	108.00
C8—C13—C12	110.5 (5)	C8—C13—H13	108.00
C7—C14—C15	114.5 (3)	C12—C13—H13	108.00
O2—C15—C16	113.2 (5)	C7—C14—H14A	109.00
C14—C15—C16	109.9 (4)	C7—C14—H14B	108.00
O2—C15—C14	108.9 (3)	C15—C14—H14A	109.00
C17—C16—C21	117.5 (5)	C15—C14—H14B	109.00
C15—C16—C17	121.4 (5)	H14A—C14—H14B	108.00
C15—C16—C21	121.0 (5)	O2—C15—H15	108.00
C16—C17—C18	121.4 (7)	C14—C15—H15	108.00
C17—C18—C19	119.6 (8)	C16—C15—H15	108.00

O3—C19—C20	116.3 (6)	C16—C17—H17	119.00
O3—C19—C18	123.9 (7)	C18—C17—H17	119.00
C18—C19—C20	119.8 (5)	C17—C18—H18	120.00
C19—C20—C21	120.5 (6)	C19—C18—H18	120.00
C16—C21—C20	121.1 (6)	C19—C20—H20	120.00
C2—C1—H1	119.00	C21—C20—H20	120.00
C6—C1—H1	119.00	C16—C21—H21	119.00
C1—C2—H2	121.00	C20—C21—H21	119.00
C3—C2—H2	121.00	O3—C22—H22A	109.00
C3—C4—H4	120.00	O3—C22—H22B	110.00
C5—C4—H4	120.00	O3—C22—H22C	110.00
C4—C5—H5	119.00	H22A—C22—H22B	109.00
C6—C5—H5	119.00	H22A—C22—H22C	109.00
C6—C7—H7	107.00	H22B—C22—H22C	109.00
C8—C7—H7	107.00		

C22—O3—C19—C20	−179.9 (10)	C9—C8—C13—C12	56.1 (6)
C22—O3—C19—C18	0.2 (16)	C7—C8—C13—O1	60.3 (5)
C2—C1—C6—C5	0.7 (9)	C7—C8—C13—C12	−176.9 (4)
C2—C1—C6—C7	179.3 (6)	C8—C9—C10—C11	55.8 (7)
C6—C1—C2—C3	−0.2 (10)	C9—C10—C11—C12	−53.1 (8)
C1—C2—C3—Br1	178.1 (5)	C10—C11—C12—C13	53.9 (8)
C1—C2—C3—C4	−0.7 (10)	C11—C12—C13—C8	−55.2 (6)
Br1—C3—C4—C5	−177.8 (5)	C11—C12—C13—O1	67.6 (6)
C2—C3—C4—C5	1.1 (10)	C7—C14—C15—C16	−171.5 (4)
C3—C4—C5—C6	−0.5 (10)	C7—C14—C15—O2	64.0 (5)
C4—C5—C6—C7	−179.0 (5)	O2—C15—C16—C17	44.5 (8)
C4—C5—C6—C1	−0.4 (9)	C14—C15—C16—C21	98.9 (7)
C5—C6—C7—C8	109.2 (6)	O2—C15—C16—C21	−139.1 (6)
C1—C6—C7—C8	−69.4 (6)	C14—C15—C16—C17	−77.5 (9)
C1—C6—C7—C14	56.5 (7)	C15—C16—C21—C20	−176.6 (7)
C5—C6—C7—C14	−125.0 (5)	C17—C16—C21—C20	0.0 (12)
C14—C7—C8—C9	178.6 (4)	C15—C16—C17—C18	176.3 (8)
C14—C7—C8—C13	52.5 (5)	C21—C16—C17—C18	−0.3 (13)
C6—C7—C14—C15	59.2 (6)	C16—C17—C18—C19	1.0 (16)
C8—C7—C14—C15	−176.3 (4)	C17—C18—C19—C20	−1.4 (16)
C6—C7—C8—C9	−56.0 (5)	C17—C18—C19—O3	178.5 (10)
C6—C7—C8—C13	177.9 (4)	O3—C19—C20—C21	−178.9 (9)
C13—C8—C9—C10	−57.8 (7)	C18—C19—C20—C21	1.0 (15)
C7—C8—C9—C10	173.7 (5)	C19—C20—C21—C16	−0.3 (14)
C9—C8—C13—O1	−66.8 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{i}	0.82	2.13	2.854 (4)	147

O2—H2*A*···O2^{ii}	0.78 (3)	2.46 (2)	2.871 (6)	115 (2)
C5—H5···O3^{iii}	0.93	2.36	3.287 (10)	171

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