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trans-2-(2-Bromo-4-methylphenyloxy)cyclohexanol

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.122
Data-to-parameter ratio = 21.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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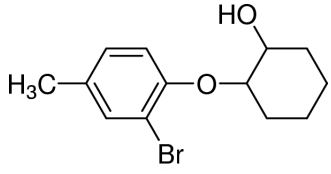
The crystal structure of the title molecule, $C_{13}H_{17}BrO_2$, contains two crystallographically independent molecules in the asymmetric unit. The cyclohexane rings of these two molecules adopt chair conformations. In the solid state, the molecules are aggregated around the cell corners to form a four-membered cooperative O—H···O—H···O—H··· hydrogen-bonded ring.

Comment

The cyclohexanol and its derivatives were proven to be an important tool in both biochemical and physiological studies of the cholinergin nerve terminal (Rogers *et al.*, 1989). Also, many of the cyclohexanol derivatives exhibit good receptor properties against the inhibition of acetylcholine storage by nerve terminal synaptic vesicles (Marshall & Parsons, 1987). The crystal structure determination of the title compound, (I), one of the above derivatives, was performed in order to elucidate its molecular conformation.



(I)

The asymmetric unit of (I) contains two crystallographically independent molecules linked by an O2*B*—H2*B*···O2*A* hydrogen bond (Table 1). No significant differences in the corresponding bond lengths and angles of these two molecules are observed and they show normal values. The cyclohexane ring in both molecules adopts the chair conformation and the hydroxyl and benzoyl groups are equatorially attached. In the solid state, the inversion-related molecules (symmetry code: $2-x, -y, 2-z$) are linked by O—H···O hydrogen bonds to form molecular aggregates with a four-membered cooperative O—H···O—H···O—H··· hydrogen-bonded ring. The crystal structure is further stabilized by weak C—H···π interactions involving the phenyl rings of molecule *A* (C_gA = centroid of C1*A*—C6*A*) and molecule *B* (C_gB = centroid of C1*B*—C6*B*).

Experimental

To a mixture of cyclohexene oxide (0.5 g, 5 mmol) and neutral alumina (3.5 g) in dry benzene (50 ml) was added dropwise 2-bromo-4-methylphenol (0.95 g, 5 mmol) at room temperature. It was further refluxed until disappearance of the starting material in TLC. Then it was filtered and the solvent was removed under vaccum. The residue

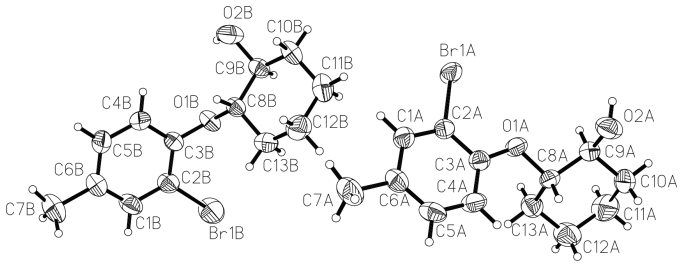


Figure 1
The structure of the asymmetric unit of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

obtained was further purified by column chromatography using silica gel to afford the title compound in good yield (1.25 g, 86%). Single crystals were grown by slow evaporation of the solvent from a solution of the compound in chloroform–methanol.

Crystal data

$C_{13}H_{17}BrO_2$	$Z = 4$
$M_r = 285.18$	$D_x = 1.462$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 9.7151$ (2) Å	Cell parameters from 4186 reflections
$b = 11.6911$ (3) Å	$\theta = 1.7$–$28.3°$
$c = 12.4332$ (3) Å	$\mu = 3.16$ mm^{-1}
$\alpha = 77.401$ (1)°	$T = 293$ (2) K
$\beta = 80.521$ (1)°	Plate, colourless
$\gamma = 70.905$ (1)°	0.24 × 0.20 × 0.10 mm
$V = 1295.65$ (5) Å^3	

Data collection

Siemens SMART CCD area-detector diffractometer	6185 independent reflections
	3937 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.030$
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 28.3°$
	$h = -9 \rightarrow 12$
$T_{min} = 0.518$, $T_{max} = 0.743$	$k = -14 \rightarrow 15$
9000 measured reflections	$l = -16 \rightarrow 15$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.122$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.00$	$\Delta\rho_{max} = 0.42$ e Å^{-3}
6185 reflections	$\Delta\rho_{min} = -0.63$ e Å^{-3}
293 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
O2B—H2B···O2A	0.82	1.97	2.712 (4)	151
O2A—H2A···O2B^{i}	0.82	1.98	2.777 (4)	164
C4A—H4A···$C_g B$	0.93	3.21	3.968 (4)	140
C12A—H12A···$C_g A^{ii}$	0.97	3.32	4.272 (5)	166

Symmetry codes: (i) $2 - x, -y, 2 - z$; (ii) $2 - x, 1 - y, 1 - z$.

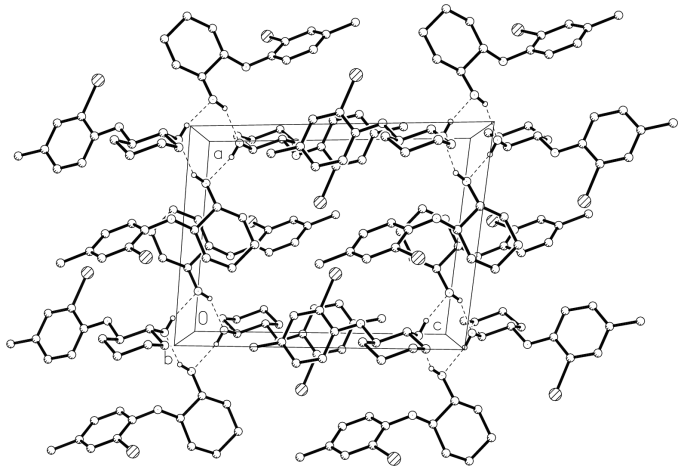


Figure 2
A plot of the molecular aggregates of (I), viewed down the *b* axis.

After checking their presence in a difference map, all the H atoms were placed at geometrically calculated positions and a riding model was used for their refinement; rotating group refinement was used for the methyl and hydroxyl groups.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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