

Data collection

Enraf–Nonius CAD-4
diffractometer $\theta_{\max} = 25^\circ$
 θ – 2θ scans $h = -24 \rightarrow 24$
Absorption correction: $k = 0 \rightarrow 11$
none $l = 0 \rightarrow 24$
7059 measured reflections 3 standard reflections
6913 independent reflections [(500), (004) and (131)]
4559 observed reflections frequency: 120 min
[$I > 3\sigma(I)$] intensity variation:
 $R_{\text{int}} = 0.015$ insignificant

Refinement

Refinement on F $\Delta\rho_{\max} = 0.49 \text{ e } \text{\AA}^{-3}$
 $R = 0.054$ $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$
 $wR = 0.059$ Extinction correction: none
 $S = 1.71$ Atomic scattering factors
4559 reflections from *International Tables*
406 parameters for *X-ray Crystallography*
 $w = 1/[\sigma^2(F) + (0.04F)^2]$ (1974, Vol. IV)
 $(\Delta/\sigma)_{\max} < 0.01$

The unit-cell dimensions and systematic absences [($0k0$) $k = 2n + 1$, ($h0l$) $l = 2n + 1$] uniquely determined the space group as $P2_1/c$. Corrections to the intensity measurements and contributions to the e.s.d.'s were calculated from polynomial fits to the observed behavior of the standards (McClandlish, Stout & Andrews, 1975). The structure was solved by direct methods using *MULTAN11/82* (Main *et al.*, 1982). The initial E map contained 44 non-H-atom peaks corresponding to two molecules per asymmetric unit. Least-squares refinement and subsequent Fourier synthesis resulted in one additional atom identified as the O atom of a water molecule. Least-squares refinement of the occupancy of this site led to a value of 0.25 (1), in agreement with the experimental density. All H atoms except those of the water molecules were placed in calculated positions with C–H 0.95 Å and temperature factors defined as $1.3 \times B$ of the bonded atom. Water H atoms were located on a difference Fourier map. Non-H atoms were refined with anisotropic displacement parameters. H-atom coordinates and temperature factors were held fixed. Comparison of F_o and F_c for the strongest reflections gave no indication of secondary extinction. Corrections were made for anomalous-scattering contributions. The program system used was *SDP* (Frenz, 1978).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: GR1007). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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***trans*-2-Bromo-5,6-methylenedioxy-1-indanol**

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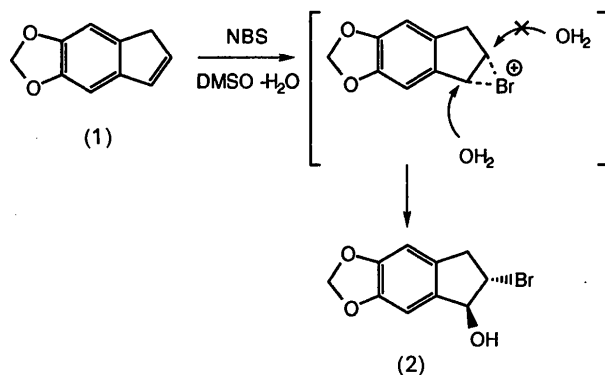
Abstract

The title compound, C₁₀H₉BrO₃, is a key intermediate in the synthesis of some mechanism-based inhibitors of dopamine β -hydroxylase. The X-ray analysis of the title compound reveals *trans* stereochemistry for the bromo and hydroxyl substituents.

Comment

trans-2-Bromo-5,6-methylenedioxy-1-indanol, (2), which is a key intermediate (Eydoux & Réglie, 1993) in the synthesis of some mechanism-based inhibitors of dopamine β -hydroxylase (Fitzpatrick & Villafranca, 1987), was quantitatively obtained by hydroxybromination of 5,6-methylenedioxyindene, (1), with *N*-bromosuccinimide (NBS) in aqueous dimethyl sulfoxide (DMSO). The hydroxybromination of olefins is known to proceed by *anti* addition of water molecules to a cyclic Br⁺ ion (Dalton, Henrickson & Jones, 1966; Hassner, 1971; Mitchell,

Lai & Williams, 1979; Buchi & Wüest, 1979). However, in order to determine the regiochemistry and confirm the stereochemistry we determined the structure of compound (2) by X-ray analysis.



The bond distances and bond angles are similar to those found in other compounds (Allen *et al.*, 1987). Compound (2) is the result of an effectively *anti* addition of water to the benzylic position of the cyclic bromonium ion. A view of (2) (Fig. 1) shows the relative boat conformation: the atoms O(2), O(1), C(8) and C(10) are coplanar with the phenyl ring; atoms C(9) and C(1) are 0.62 (1) and 0.10 (1) Å, respectively, out of the phenyl plane. This boat conformation is more pronounced on the bromide side of the molecule than on the other, with dihedral angles of 40.6 (9)° between the phenyl ring and the plane formed by the atoms C(8), C(9) and C(10), and 11 (3)° between the phenyl ring and the plane formed by the atoms O(2), C(1) and O(1). The X-ray structure analysis shows that intermolecular hydrogen

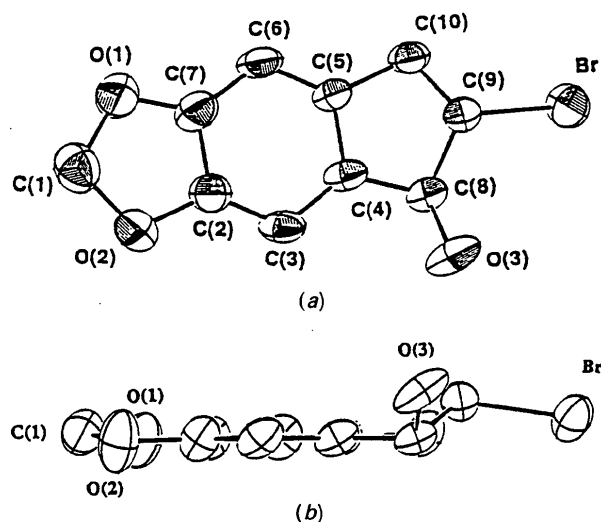


Fig. 1. ORTEP (Johnson, 1965) views of compound (2) showing (a) the atom-numbering scheme and (b) the boat conformation of the molecule. H atoms are not shown.

bonds are formed between hydroxyl groups with $O(3^i) \cdots O(3^{ii}) = 2.66$ (1) Å [symmetry codes: (i) x, y, z ; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$] (Fig. 2).

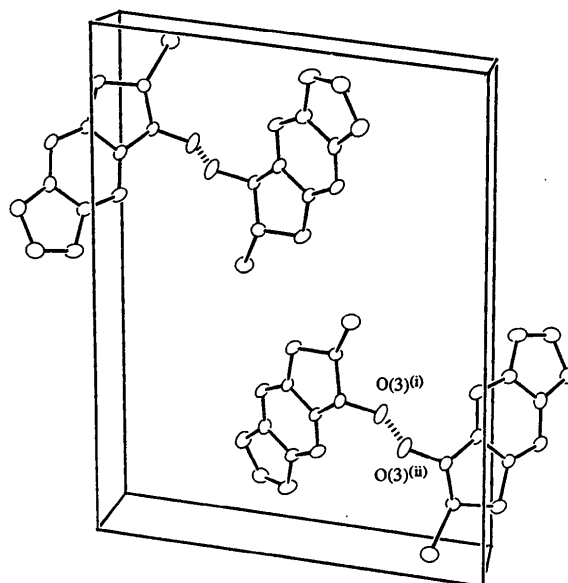


Fig. 2. An ORTEP (Johnson, 1965) packing view of compound (2) showing the unit-cell contents and the intermolecular hydrogen bond between the hydroxyl groups (H atoms are not shown) [symmetry codes: (i) x, y, z ; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$].

Experimental

Crystal data

$C_{10}H_9BrO_3$
 $M_r = 257.1$
 Monoclinic
 $P2_1/n$
 $a = 12.596$ (4) Å
 $b = 4.831$ (2) Å
 $c = 16.153$ (6) Å
 $\beta = 96.57$ (5)°
 $V = 976$ (1) Å³
 $Z = 4$
 $D_x = 1.749$ Mg m⁻³
 $D_m = 1.8$ Mg m⁻³
 D_m measured by flotation in 1,5-dibromopentane

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 25 reflections
 $\theta = 12-15^\circ$
 $\mu = 4.149$ mm⁻¹
 $T = 300$ K
 Rectangular block
 $0.4 \times 0.2 \times 0.1$ mm
 Brown
 Crystal source: grown from dichloromethane

Data collection

Enraf-Nonius four-circle diffractometer
 $\theta/2\theta$ scans
 Absorption correction: none
 1814 measured reflections
 1678 independent reflections
 1019 observed reflections
 $[I > 3\sigma(I)]$

$R_{int} = 0.069$
 $\theta_{max} = 24^\circ$
 $h = -14 \rightarrow 14$
 $k = 0 \rightarrow 5$
 $l = 0 \rightarrow 18$
 22 standard reflections
 intensity variation: 2%

Refinement

Refinement on <i>F</i>	(Δ/σ) _{max} = 0.01
<i>R</i> = 0.076	$\Delta\rho_{\text{max}} = 0.58 \text{ e } \text{\AA}^{-3}$
<i>wR</i> = 0.087	$\Delta\rho_{\text{min}} = -0.49 \text{ e } \text{\AA}^{-3}$
<i>S</i> = 2.96	Extinction correction: none
991 reflections	Atomic scattering factors
127 parameters	from <i>International Tables</i>
H-atom parameters not refined	for <i>X-ray Crystallography</i>
<i>w</i> = 1/ σ^2 (<i>F</i>)	(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{\text{eq}} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
Br	0.6383 (2)	0.4596 (3)	0.4548 (1)	5.20 (3)
O(1)	0.3021 (8)	-0.429 (3)	0.1175 (6)	5.7 (3)
O(2)	0.4326 (9)	-0.281 (3)	0.0399 (6)	6.0 (3)
O(3)	0.7089 (7)	0.256 (2)	0.2631 (7)	5.0 (2)
C(1)	0.347 (1)	-0.473 (3)	0.041 (1)	5.1 (4)
C(2)	0.452 (1)	-0.166 (3)	0.1193 (9)	4.1 (3)
C(3)	0.530 (1)	0.004 (3)	0.1488 (8)	3.8 (3)
C(4)	0.528 (1)	0.097 (3)	0.2315 (8)	3.4 (3)
C(5)	0.4474 (9)	0.002 (3)	0.2783 (8)	3.3 (3)
C(6)	0.369 (1)	-0.176 (3)	0.2467 (9)	4.2 (3)
C(7)	0.371 (1)	-0.263 (3)	0.1650 (9)	4.4 (3)
C(8)	0.602 (1)	0.286 (3)	0.2801 (8)	3.2 (3)
C(9)	0.586 (1)	0.188 (3)	0.3680 (8)	3.6 (3)
C(10)	0.470 (1)	0.124 (3)	0.3653 (8)	4.1 (3)

Table 2. Selected geometric parameters (\AA , °)

C(1)—O(1)	1.43 (2)	C(5)—C(6)	1.36 (2)
C(1)—O(2)	1.42 (2)	C(5)—C(10)	1.52 (2)
C(2)—C(3)	1.33 (2)	C(6)—C(7)	1.39 (2)
C(2)—C(7)	1.41 (2)	C(7)—O(1)	1.35 (2)
C(2)—O(2)	1.39 (2)	C(8)—C(9)	1.53 (2)
C(3)—C(4)	1.41 (2)	C(8)—O(3)	1.41 (2)
C(4)—C(5)	1.41 (2)	C(9)—C(10)	1.49 (2)
C(4)—C(8)	1.47 (2)	C(9)—Br	1.98 (1)
C(1)—O(1)—C(7)	106 (1)	C(1)—O(2)—C(2)	107 (1)
C(2)—C(3)—C(4)	116 (1)	C(2)—C(7)—C(6)	119 (1)
C(2)—C(7)—O(1)	111 (1)	C(3)—C(2)—C(7)	125 (1)
C(3)—C(2)—O(2)	128 (1)	C(3)—C(4)—C(5)	120 (1)
C(3)—C(4)—C(8)	129 (1)	C(4)—C(5)—C(10)	108 (1)
C(4)—C(5)—C(6)	123 (1)	C(4)—C(8)—C(9)	99 (1)
C(4)—C(8)—O(3)	113 (1)	C(5)—C(4)—C(8)	112 (1)
C(5)—C(6)—C(7)	117 (1)	C(5)—C(10)—C(9)	101 (1)
C(6)—C(5)—C(10)	130 (1)	C(6)—C(7)—O(1)	130 (1)
C(7)—C(2)—O(2)	107 (1)	C(8)—C(9)—Br	113 (1)
C(8)—C(9)—C(10)	106 (1)	C(9)—C(8)—O(3)	112 (1)
C(10)—C(9)—Br	114 (1)	O(1)—C(1)—O(2)	107 (1)

Program used for data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Programs used for data reduction and structure solution: *SDP* (Frenz, 1978) via direct methods with *MULTAN80* (Main *et al.*, 1980) and difference Fourier syntheses. Program used for molecular graphics: *ORTEP* (Johnson, 1965). H atoms were introduced at idealized positions before the last cycle of refinement, which included anisotropic displacement parameters for all non-H atoms.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1083). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4-Amino-3-chlorobenzophenone

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Abstract

The title compound, C₁₃H₁₀ClNO, has two phenyl groups, one of which has three substituents. The dihedral angles between the plane of the carbonyl group and the planes of the substituted and unsubstituted phenyl rings are 151.4 (3) and 149.7 (3)°, respectively.

Comment

It is reported that 4-aminobenzophenone (ABP) has a large second-harmonic generation (SHG) efficiency (Frazier & Cockerham, 1987). We have synthesized ABP and its derivatives in order to investigate their non-linear optical properties. The title compound, (I), is one such compound, but has no SHG efficiency, which depends on the conjugation of donor and acceptor.