References

Byriel, K. A., Kennard, C. H. L., Lynch, D. E., Smith, G. & Thompson, J. G. (1992). *Aust. J. Chem.* **45**, 969–981.

Davies, E. S. & Hartshorne, N. H. (1934). *J. Chem. Soc.* pp. 1830–1836.

Dhaneshwar, N. N., Kulkarni, A. G., Tavole, S. S. & Part, L. M. (1975). *Acta Cryst.* B**31**, 1978–1980.

Dhaneshwar, N. N., Tavole, S. S. & Part, L. M. (1974). *Acta Cryst.* B**30**, 583–587.

Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.

Lynch, D. E., Smith, G., Byriel, K. A. & Kennard, C. H. L. (1992). *Acta Cryst.* C**48**, 1265–1267.

Lynch, D. E., Smith, G., Byriel, K. A. & Kennard, C. H. L. (1994). *Aust. J. Chem.* In the press.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures.* Univ. of Cambridge, England.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination.* Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.

Acta Cryst. (1994). C**50**, 1294–1295

2-(3-Benzyloxy-4-nitrophenyl)oxirane, an Intermediate in the Synthesis of Formoterol

K. Chandra Mohan and K. Ravikumar

Laboratory of Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India

T. V. S. K. Vittal and C. K. Gido

Bio-Organic Laboratory, Indian Institute of Chemical Technology, Hyderabad 500 007, India

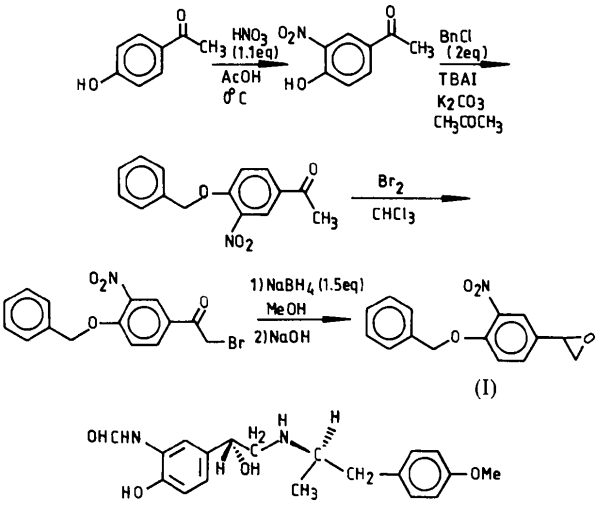
(*Received* 29 *September* 1993; *accepted* 23 *December* 1993)

Abstract

In the title compound, $C_{15}H_{13}NO_4$, the two planar nitrophenyl and benzyloxy groups are inclined at 152.3 (2)°. The torsion angle about the central C—O bond is 177.5 (5)°, giving an extended C—C—O—C chain. The nitro group is twisted out of the plane of the phenyl ring by 28.4 (3)° to diminish steric hindrance; O atoms of the nitro and benzyloxy groups are separated by only 2.625 (7) Å. The dihedral angles between the epoxy ring and the two aromatic rings are 98.3 (3) and 102.6 (3)°. There is a possible C—H···O intermolecular interaction.

Comment

The title compound, (I), is one of the intermediates in the synthesis of the anti-asthmatic agent formoterol, (II), a β-adrenoreceptor-stimulating catecholamine analogue with selective brochodilator activity (Murase, Mase, Ida, Takahashi & Murakami, 1977; Trofast, Osterberg, Kallstrom & Waldeck, 1991). It was prepared in four stages as shown in the scheme below.



The structure determination of (I) was undertaken to gain insight into the reaction pathways. In all essential details the geometry of the molecule is normal. The dihedral angles between the plane defined by C(6)—O(4)—C(9)—C(10) and the two aromatic rings present in the structure are 2.7 (3) and 149.6 (4)°. The nitro group is twisted 28.4 (3)° out of the plane of the phenyl ring and this gives a short O(4)···O(3) intramolecular contact of 2.625 (7) Å; the sum of van der Waals radii for O is 2.80 Å (Pauling, 1960). The O(4)···O(3)—N(1) angle is 86.1 (7)°.

One C—H···O intermolecular contact [C(9)—H···O(1)($y-\frac{3}{4}$, $-x+\frac{3}{4}$, $-z+\frac{3}{4}$) 3.437 (8) Å] is geometrically appropriate for a C—H···O hydrogen bond (Berkovitch-Yellin & Leiserowitz, 1984). Other contacts are all of van der Waals type.

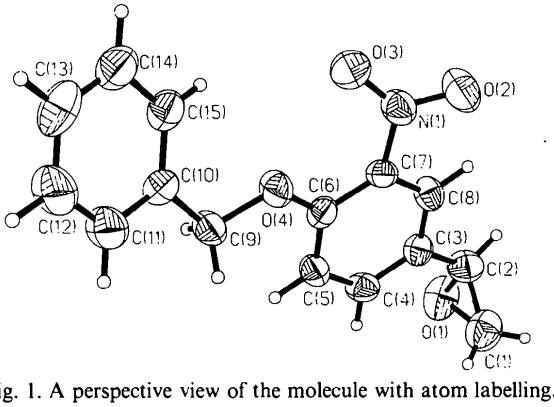


Fig. 1. A perspective view of the molecule with atom labelling.

Experimental

Crystal data

$C_{15}H_{13}NO_4$
$M_r = 271.3$
Tetragonal
$I4_1/a$
$a = 19.531$ (3) Å
$c = 14.131$ (3) Å
$V = 5390.4$ (2) Å^3
$Z = 16$
$D_x = 1.337$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 9$–$22°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Cube
0.23 × 0.20 × 0.17 mm
White
Crystal source: recrystallization from methanol

Data collection

Siemens *R3m/V* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1994 measured reflections
1776 independent reflections
969 observed reflections [$I \geq 3\sigma(I)$]
$R_{int} = 0.0316$
$\theta_{max} = 22.5°$
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 15$
2 standard reflections monitored every 98 reflections
intensity variation: <1%

Refinement

Refinement on F
$R = 0.050$
$wR = 0.058$
$S = 1.15$
969 reflections
181 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0018|F|^2]$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1991)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

Origin of coordinates at $\bar{1}$ on glide plane *b*.

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O(1)	0.1200 (3)	0.7612 (3)	0.5601 (3)	0.069 (2)
O(2)	0.3122 (3)	0.5873 (3)	0.2914 (3)	0.069 (2)
O(3)	0.2439 (3)	0.5670 (3)	0.1758 (4)	0.096 (3)
O(4)	0.1773 (2)	0.6774 (2)	0.1230 (3)	0.051 (3)
N(1)	0.2601 (3)	0.5996 (3)	0.2458 (4)	0.050 (2)
C(1)	0.1802 (3)	0.8020 (3)	0.5560 (4)	0.072 (3)
C(2)	0.1800 (3)	0.7336 (4)	0.5183 (4)	0.057 (3)
C(3)	0.1779 (3)	0.7207 (3)	0.4129 (4)	0.040 (2)
C(4)	0.1368 (3)	0.7593 (3)	0.3523 (4)	0.046 (3)
C(5)	0.1345 (3)	0.7457 (3)	0.2554 (4)	0.044 (3)
C(6)	0.1740 (3)	0.6936 (3)	0.2163 (4)	0.040 (2)
C(7)	0.2154 (3)	0.6557 (3)	0.2782 (4)	0.039 (2)
C(8)	0.2173 (3)	0.6690 (3)	0.3755 (4)	0.043 (2)
C(9)	0.1329 (3)	0.7141 (3)	0.0581 (4)	0.048 (2)
C(10)	0.1449 (3)	0.6837 (3)	−0.0398 (4)	0.043 (2)
C(11)	0.1360 (4)	0.7249 (4)	−0.1179 (4)	0.063 (3)
C(12)	0.1451 (4)	0.6987 (3)	−0.2079 (4)	0.073 (3)
C(13)	0.1638 (3)	0.6313 (3)	−0.2205 (4)	0.072 (3)
C(14)	0.1736 (3)	0.5887 (3)	−0.1433 (4)	0.064 (3)
C(15)	0.1625 (3)	0.6158 (3)	−0.0515 (4)	0.053 (3)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(1)	1.421 (8)	O(1)—C(2)	1.420 (8)
O(2)—N(1)	1.228 (8)	O(3)—N(1)	1.217 (8)
O(4)—C(6)	1.357 (7)	O(4)—C(9)	1.451 (7)
N(1)—C(7)	1.476 (8)	C(1)—C(2)	1.438 (9)
C(2)—C(3)	1.510 (8)	C(3)—C(4)	1.395 (9)
C(3)—C(8)	1.376 (9)	C(4)—C(5)	1.395 (9)
C(5)—C(6)	1.391 (9)	C(6)—C(7)	1.402 (8)
C(7)—C(8)	1.399 (8)	C(9)—C(10)	1.523 (8)
C(10)—C(11)	1.377 (9)	C(10)—C(15)	1.380 (9)
C(11)—C(12)	1.382 (9)	C(12)—C(13)	1.378 (9)
C(13)—C(14)	1.385 (9)	C(14)—C(15)	1.416 (9)
C(1)—O(1)—C(2)	60.8 (4)	C(6)—O(4)—C(9)	118.1 (5)
O(2)—N(1)—O(3)	122.7 (6)	O(3)—N(1)—C(7)	119.1 (6)
O(2)—N(1)—C(7)	118.2 (5)	O(1)—C(1)—C(2)	59.5 (4)
O(1)—C(2)—C(1)	59.6 (4)	C(1)—C(2)—C(3)	121.4 (6)
O(1)—C(2)—C(3)	116.8 (5)	C(2)—C(3)—C(8)	119.1 (5)
C(2)—C(3)—C(4)	122.1 (6)	C(4)—C(3)—C(8)	118.8 (6)
C(3)—C(4)—C(5)	121.2 (6)	C(4)—C(5)—C(6)	120.8 (6)
O(4)—C(6)—C(5)	125.7 (5)	C(5)—C(6)—C(7)	117.2 (6)
O(4)—C(6)—C(7)	117.1 (5)	N(1)—C(7)—C(6)	122.7 (5)
C(6)—C(7)—C(8)	122.1 (6)	N(1)—C(7)—C(8)	115.3 (5)
C(3)—C(8)—C(7)	119.9 (6)	O(4)—C(9)—C(10)	106.9 (5)
C(9)—C(10)—C(15)	121.4 (6)	C(9)—C(10)—C(11)	118.8 (6)
C(11)—C(10)—C(15)	119.8 (6)	C(10)—C(11)—C(12)	120.4 (6)
C(11)—C(12)—C(13)	120.4 (6)	C(12)—C(13)—C(14)	120.5 (6)
C(13)—C(14)—C(15)	118.4 (6)	C(10)—C(15)—C(14)	120.4 (6)

A $\Delta\rho$ map showed the positions of all H atoms, which were placed in idealized positions and included in the least-squares refinement with fixed isotropic displacement parameters. Structure solution and refinement were performed using *SHELXTL-Plus* (Sheldrick, 1991). Geometric parameters were calculated using the program *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HA1084). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Berkovitch-Yellin, Z. & Leiserowitz, L. (1984). *Acta Cryst.* **B40**, 159–165.

Murase, K., Mase, T., Ida, H., Takahashi, K. & Murakami, M. (1977). *Chem. Pharm. Bull.* **25**, 1368–1377.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Pauling, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.

Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Trofast, J., Osterberg, K., Kallstrom, B. L. & Waldeck, B. (1991). *Chirality*, **3**, 443–450.