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

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
O1*A*	0.7749 (2)	0.0674 (2)	0.0912 (1)	4.32 (4)
C2*A*	0.9373 (4)	0.0593 (3)	0.1283 (2)	4.34 (6)
O2*A*	0.9486 (3)	−0.0345 (3)	0.1741 (1)	6.20 (6)
C3*A*	1.0748 (4)	0.1884 (3)	0.1029 (2)	4.53 (6)
C4*A*	0.9972 (3)	0.2664 (3)	0.0544 (2)	3.91 (6)
C5*A*	0.7979 (3)	0.1977 (3)	0.0441 (2)	3.52 (5)
O6*A*	0.7086 (2)	0.1083 (2)	−0.0445 (1)	3.44 (4)
C7*A*	0.6647 (3)	0.2107 (3)	−0.0982 (2)	3.32 (5)
O8*A*	0.8263 (2)	0.3123 (2)	−0.1180 (1)	3.54 (4)
C9*A*	0.8134 (3)	0.2735 (3)	−0.2053 (2)	3.80 (5)
O9*A*	0.9240 (3)	0.3476 (3)	−0.2393 (1)	5.26 (5)
C10*A*	0.6411 (3)	0.1315 (3)	−0.2482 (2)	3.98 (6)
C11*A*	0.5577 (3)	0.0956 (3)	−0.1866 (2)	3.77 (6)
C12*A*	0.5797 (3)	0.3333 (3)	−0.0549 (2)	4.15 (6)
O13*A*	0.6921 (3)	0.4331 (2)	0.0282 (1)	4.63 (4)
C14*A*	0.7130 (4)	0.3246 (4)	0.0832 (2)	4.76 (7)
O1*B*	0.4665 (2)	0.4320 (2)	0.6281 (1)	3.92 (4)
C2*B*	0.4331 (3)	0.4318 (3)	0.7092 (2)	3.92 (6)
O2*B*	0.5349 (3)	0.5313 (3)	0.7743 (1)	5.90 (6)
C3*B*	0.2619 (3)	0.2954 (3)	0.6988 (2)	3.97 (6)
C4*B*	0.1974 (3)	0.2226 (3)	0.6147 (2)	3.69 (5)
C5*B*	0.3249 (3)	0.3007 (3)	0.5621 (2)	3.34 (5)
O6*B*	0.2543 (2)	0.3923 (2)	0.5083 (1)	3.46 (4)
C7*B*	0.1486 (3)	0.2929 (3)	0.4245 (2)	3.38 (5)
O8*B*	−0.0244 (2)	0.1945 (2)	0.4358 (1)	3.78 (4)
C9*B*	−0.1524 (3)	0.2393 (3)	0.3893 (2)	4.04 (6)
O9*B*	−0.3090 (2)	0.1691 (3)	0.3844 (2)	5.78 (6)
C10*B*	−0.0667 (4)	0.3759 (3)	0.3497 (2)	4.40 (6)
C11*B*	0.1064 (3)	0.4095 (3)	0.3716 (2)	4.05 (6)
C12*B*	0.2298 (3)	0.1682 (3)	0.3798 (2)	4.00 (6)
O13*B*	0.2763 (2)	0.0695 (2)	0.4358 (1)	4.30 (4)
C14*B*	0.4052 (4)	0.1813 (3)	0.5126 (2)	4.33 (6)

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

O1*A*—C2*A*	1.374 (4)	O1*B*—C2*B*	1.359 (3)
O1*A*—C5*A*	1.432 (3)	O1*B*—C5*B*	1.437 (2)
C2*A*—O2*A*	1.188 (4)	C2*B*—O2*B*	1.201 (3)
C2*A*—C3*A*	1.480 (4)	C2*B*—C3*B*	1.480 (3)
C3*A*—C4*A*	1.305 (4)	C3*B*—C4*B*	1.309 (3)
C4*A*—C5*A*	1.511 (3)	C4*B*—C5*B*	1.510 (4)
C5*A*—O6*A*	1.423 (3)	C5*B*—O6*B*	1.419 (3)
C5*A*—C14*A*	1.508 (4)	C5*B*—C14*B*	1.503 (4)
O6*A*—C7*A*	1.404 (3)	O6*B*—C7*B*	1.414 (2)
C7*A*—O8*A*	1.455 (3)	C7*B*—O8*B*	1.460 (3)
C7*A*—C11*A*	1.503 (3)	C7*B*—C11*B*	1.489 (4)
C7*A*—C12*A*	1.506 (4)	C7*B*—C12*B*	1.506 (4)
O8*A*—C9*A*	1.344 (3)	O8*B*—C9*B*	1.360 (3)
C9*A*—O9*A*	1.199 (3)	C9*B*—O9*B*	1.211 (3)
C9*A*—C10*A*	1.499 (3)	C9*B*—C10*B*	1.471 (4)
C10*A*—C11*A*	1.308 (4)	C10*B*—C11*B*	1.314 (4)
C12*A*—O13*A*	1.423 (3)	C12*B*—O13*B*	1.411 (4)
O13*A*—C14*A*	1.410 (4)	O13*B*—C14*B*	1.429 (3)
C2*A*—O1*A*—C5*A*	110.4 (2)	C9*A*—C10*A*—C11*A*	107.9 (2)
O1*A*—C2*A*—O2*A*	121.2 (3)	C7*A*—C11*A*—C10*A*	110.3 (2)
O1*A*—C2*A*—C3*A*	107.1 (2)	C7*A*—C12*A*—O13*A*	109.4 (2)
O2*A*—C2*A*—C3*A*	131.6 (3)	C12*A*—O13*A*—C14*A*	109.8 (2)
C2*A*—C3*A*—C4*A*	109.1 (2)	C5*A*—C14*A*—O13*A*	111.2 (2)
C3*A*—C4*A*—C5*A*	109.9 (2)	C7*B*—O8*B*—C9*B*	108.5 (2)
O1*A*—C5*A*—C4*A*	103.5 (2)	O8*B*—C9*B*—O9*B*	121.4 (3)
O1*A*—C5*A*—O6*A*	104.8 (2)	O8*B*—C9*B*—C10*B*	108.9 (2)
O1*A*—C5*A*—C14*A*	107.7 (2)	O9*B*—C9*B*—C10*B*	129.7 (3)
C4*A*—C5*A*—O6*A*	111.9 (2)	C9*B*—C10*B*—C11*B*	108.3 (3)
C4*A*—C5*A*—C14*A*	116.0 (2)	C2*B*—O1*B*—C5*B*	109.9 (2)
O6*A*—C5*A*—C14*A*	111.9 (2)	O1*B*—C2*B*—O2*B*	121.2 (2)
C5*A*—O6*A*—C7*A*	115.4 (2)	O1*B*—C2*B*—C3*B*	108.3 (2)
O6*A*—C7*A*—O8*A*	109.0 (2)	O2*B*—C2*B*—C3*B*	130.5 (3)
O6*B*—C7*B*—C11*B*	109.2 (2)	C2*B*—C3*B*—C4*B*	108.1 (2)
O6*B*—C7*B*—C12*B*	112.4 (2)	C3*B*—C4*B*—C5*B*	110.2 (2)
O8*B*—C7*B*—C11*B*	104.0 (2)	O1*B*—C5*B*—C4*B*	103.4 (2)
O8*B*—C7*B*—C12*B*	108.2 (2)	O1*B*—C5*B*—O6*B*	104.2 (2)
C11*B*—C7*B*—C12*B*	114.3 (2)	O1*B*—C5*B*—C14*B*	107.7 (2)
O6*A*—C7*A*—C11*A*	108.7 (2)	C4*B*—C5*B*—O6*B*	112.6 (2)
O6*A*—C7*A*—C12*A*	112.7 (2)	C4*B*—C5*B*—C14*B*	115.6 (2)
O8*A*—C7*A*—C11*A*	103.6 (2)	O6*B*—C5*B*—C14*B*	112.1 (2)
O8*A*—C7*A*—C12*A*	107.7 (2)	C5*B*—O6*B*—C7*B*	115.7 (2)
C11*A*—C7*A*—C12*A*	114.7 (2)	O6*B*—C7*B*—O8*B*	108.3 (2)
C7*A*—O8*A*—C9*A*	109.8 (2)	C7*B*—C11*B*—C10*B*	110.2 (2)
O8*A*—C9*A*—O9*A*	123.0 (2)	C7*B*—C12*B*—O13*B*	111.1 (2)
O8*A*—C9*A*—C10*A*	108.3 (2)	C12*B*—O13*B*—C14*B*	109.3 (2)
O9*A*—C9*A*—C10*A*	128.6 (2)	C5*B*—C14*B*—O13*B*	109.4 (2)

The structure was solved with the Enraf-Nonius *SDP* system (B. A. Frenz & Associates, Inc., 1982) using a DEC MicroVAX 3100-80 computer, at the Centre de Diffractométrie Automatique, Université Lyon I.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71679 (46 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1070]

References

Bernasconi, C., Cottier, L., Descotes, G., Nigay, H., Parron, J. C. & Wisniewski, A. (1984). *Bull. Soc. Chim. Fr.* II, pp. 323–328.

Cottier, L., Descotes, G., Nigay, H., Parron, J. C. & Gregoire, V. (1986). *Bull. Soc. Chim. Fr.* II, pp. 844–850.

B. A. Frenz & Associates, Inc. (1982). *SDP Structure Determination Package*. College Station, Texas, USA.

Moriarty, R. M., Romain, C. R., Karle, I. L. & Karle, J. (1965). *J. Am. Chem. Soc.* **87**, 3251–3252.

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

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An Intermediate in the Synthesis of Formoterol

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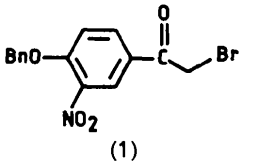
Abstract

In the title compound, 4-benzyloxy-3-nitrophenacyl bromide, $C_{15}H_{12}BrNO_4$, the two planar nitrophenyl and benzyloxy groups are inclined at a dihedral angle of 17.1 (1)°. The NO_2 group is twisted out of the

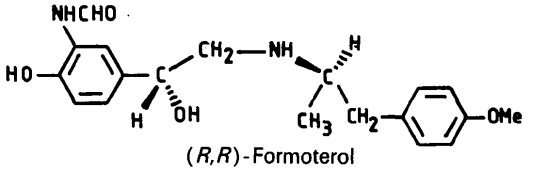
plane of the phenyl ring by 20.4 (3)° to minimize steric hindrance between O(4) and O(3); the O(4)···O(3) separation is 2.600 (5) Å and the angle O(4)···O(3)—N(1) is 88.2 (4)°.

Comment

The title compound, (1), is a key intermediate in the synthesis of an anti-asthmatic agent, formoterol, (2), a β-adrenoreceptor stimulating catecholamine analogue with selective brochodilator activity (Ida, 1976; Murase, Mase, Ida, Takahashi & Murakami, 1977; Trofast, Osterberg, Kallstrom & Waldeck, 1991).



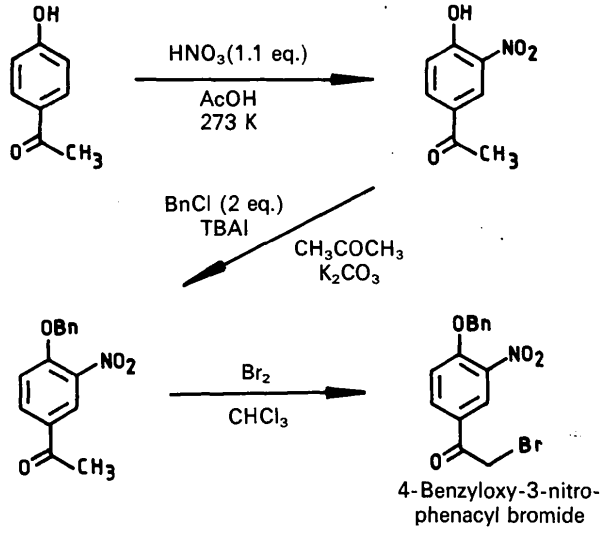
(1)



(*R,R*)-Formoterol

(2)

The title compound was prepared in three stages as shown below.



4-Benzyloxy-3-nitrophenacyl bromide

The structure determination of (1) was undertaken in order to gain insight into the successive reaction pathways. The atom labelling and the anisotropic displacement ellipsoids are shown in Fig. 1. In all essential details the geometry of the molecule shows normal values.

The C(7)—N(1) bond distance of 1.461 (6) Å is comparable to the average distance of 1.462 (3) Å for 2236 bond distances between sp^2-hybridized C atoms and nitro-group N atoms extracted from the 1986 release of the Cambridge Structural Database (Allen *et al.*, 1979).

The NO_2 group is twisted by 20.4 (3)° out of the plane of the phenyl ring, thus giving a short O(4)···O(3) intramolecular contact of 2.600 (5) Å; the sum of van der Waals radii for O atoms is 2.80 Å (Pauling, 1960). The O(4)···O(3)—N(1) angle is 88.2 (4)°.

The two phenyl rings are planar with a maximum deviation of 0.006 (5) Å; they are inclined at a dihedral angle of 17.1 (1)°. The deviations of Br, C(1), O(1) and C(2) from the mean plane of the phenyl ring to which they are attached are 0.490 (1), 0.212 (5), −0.125 (4) and 0.013 (5) Å, respectively. The dihedral angles between the plane defined by C(6)—O(4)—C(9)—C(10) and the two phenyl rings are 28.1 (3) and 11.1 (3)°. The packing contacts in the crystal are all of van der Waals type.

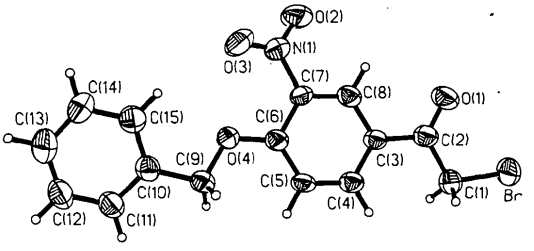


Fig. 1. A perspective view of the molecule with atom labelling.

Experimental

Crystal data

$C_{15}H_{12}BrNO_4$
M_r = 350.17
Monoclinic
$P2_1/c$
a = 7.877 (1) Å
b = 12.600 (2) Å
c = 14.326 (2) Å
β = 96.68 (1)°
V = 1412.2 (4) Å^3
Z = 4
D_x = 1.638 (3) Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71063 Å
Cell parameters from 25 reflections
θ = 8-20°
μ = 2.89 mm^{-1}
T = 293 K
Cube
0.21 × 0.19 × 0.15 mm
White

Data collection

Siemens *R3m/V* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2112 measured reflections
1840 independent reflections
1219 observed reflections [$I \geq 3\sigma(I)$]
R_{int} = 0.014
θ_{max} = 22.5°
h = 0 → 8
k = 0 → 12
l = −15 → 15
2 standard reflections monitored every 98 reflections
intensity variation: 1%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.001$
$R = 0.031$	$\Delta\rho_{max} = 0.33$ e Å^{-3}
$wR = 0.038$	$\Delta\rho_{min} = -0.21$ e Å^{-3}
$S = 1.06$	Extinction correction: none
1219 reflections	Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1991)
190 parameters	
$w = 1/[\sigma^2(F) + 0.0030\|F\|^2]$	

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

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

	x	y	z	U_{eq}
Br	0.5686 (1)	0.0535 (1)	0.61914 (4)	0.0649 (2)
O(1)	0.8543 (5)	−0.0620 (3)	0.7315 (3)	0.080 (2)
O(2)	1.4351 (5)	−0.1572 (3)	0.8333 (3)	0.092 (2)
O(3)	1.5903 (6)	−0.0533 (3)	0.9205 (4)	0.111 (2)
O(4)	1.5623 (4)	0.1473 (2)	0.8791 (2)	0.052 (1)
N(1)	1.4684 (6)	−0.0692 (3)	0.8641 (3)	0.057 (2)
C(1)	0.7723 (6)	0.1143 (4)	0.6856 (3)	0.051 (2)
C(2)	0.8944 (7)	0.0309 (4)	0.7294 (3)	0.047 (2)
C(3)	1.0660 (6)	0.0679 (3)	0.7694 (3)	0.040 (2)
C(4)	1.1147 (6)	0.1738 (4)	0.7791 (3)	0.045 (2)
C(5)	1.2775 (6)	0.2019 (4)	0.8164 (3)	0.045 (2)
C(6)	1.3989 (6)	0.1252 (4)	0.8454 (3)	0.043 (2)
C(7)	1.3503 (6)	0.0174 (3)	0.8357 (3)	0.042 (2)
C(8)	1.1876 (6)	−0.0096 (4)	0.7985 (3)	0.046 (2)
C(9)	1.6056 (6)	0.2548 (3)	0.9049 (3)	0.051 (2)
C(10)	1.7903 (6)	0.2565 (4)	0.9471 (3)	0.046 (2)
C(11)	1.8861 (6)	0.3481 (4)	0.9442 (3)	0.054 (2)
C(12)	2.0522 (7)	0.3522 (5)	0.9863 (4)	0.063 (2)
C(13)	2.1257 (7)	0.2650 (5)	1.0325 (4)	0.065 (2)
C(14)	2.0314 (6)	0.1726 (5)	1.0348 (4)	0.061 (2)
C(15)	1.8655 (6)	0.1675 (4)	0.9925 (3)	0.053 (2)

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

Br—C(1)	1.926 (5)	O(1)—C(2)	1.214 (6)
O(2)—N(1)	1.211 (6)	O(3)—N(1)	1.197 (6)
O(4)—C(6)	1.350 (5)	O(4)—C(9)	1.435 (5)
N(1)—C(7)	1.461 (6)	C(1)—C(2)	1.511 (7)
C(2)—C(3)	1.481 (7)	C(3)—C(4)	1.391 (6)
C(3)—C(8)	1.397 (7)	C(4)—C(5)	1.377 (6)
C(5)—C(6)	1.389 (6)	C(6)—C(7)	1.413 (6)
C(7)—C(8)	1.372 (7)	C(9)—C(10)	1.509 (7)
C(10)—C(11)	1.382 (7)	C(10)—C(15)	1.393 (7)
C(11)—C(12)	1.377 (7)	C(12)—C(13)	1.375 (8)
C(13)—C(14)	1.383 (8)	C(14)—C(15)	1.377 (7)
Br—C(1)—C(2)	112.5 (3)	C(6)—O(4)—C(9)	118.4 (3)
O(2)—N(1)—O(3)	121.4 (5)	O(3)—N(1)—C(7)	119.9 (4)
O(2)—N(1)—C(7)	118.6 (4)	O(1)—C(2)—C(1)	121.7 (5)
C(1)—C(2)—C(3)	116.7 (4)	O(1)—C(2)—C(3)	121.5 (4)
C(2)—C(3)—C(8)	117.3 (4)	C(2)—C(3)—C(4)	124.7 (4)
C(4)—C(3)—C(8)	118.0 (4)	C(3)—C(4)—C(5)	121.3 (4)
C(4)—C(5)—C(6)	120.9 (4)	O(4)—C(6)—C(5)	123.9 (4)
C(5)—C(6)—C(7)	118.0 (4)	O(4)—C(6)—C(7)	118.0 (4)
N(1)—C(7)—C(6)	122.2 (4)	C(6)—C(7)—C(8)	120.5 (4)
N(1)—C(7)—C(8)	117.3 (4)	C(3)—C(8)—C(7)	121.3 (4)
O(4)—C(9)—C(10)	107.8 (4)	C(9)—C(10)—C(15)	120.9 (4)
C(9)—C(10)—C(11)	120.3 (4)	C(11)—C(10)—C(15)	118.8 (5)
C(10)—C(11)—C(12)	120.9 (5)	C(11)—C(12)—C(13)	120.4 (5)
C(12)—C(13)—C(14)	119.1 (15)	C(13)—C(14)—C(15)	120.8 (5)
C(10)—C(15)—C(14)	119.9 (5)		

The $\Delta\rho$ map showed the positions of all H atoms. H atoms were placed in idealized positions and included in the least-squares refinement with fixed isotropic temperature factors. Structure solution and refinement were performed with *SHELXTL-Plus* (Sheldrick, 1991). Geometrical 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 British Library Document Supply Centre as Supplementary Publication No. SUP 71610 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1062]

References

Allen, F. H., Bellard, S., Brice, M. D., Cartwright, B. A., Doubleday, A., Higgs, H., Hummelink, T., Hummelink-Peters, B. G., Kennard, O., Motherwell, W. D. S., Rodgers, J. R. & Watson, D. G. (1979). *Acta Cryst.* B**35**, 2331–2339.

Ida, M. (1976). *Arzneim. Forsch. Drug Res.* **26**, 1337–1340.

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

Nardelli, M. (1983). *Comput. Chem.* 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., Karlsruhe, Germany.

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

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2-Methyl-2-(4-nitrophenylazo)-1,3-indandione, $C_{16}H_{11}N_3O_4$

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Abstract

The 2-methyl-1,3-indandione and 4-nitrophenyl groups are *trans* to each other. The five-membered ring of the indandione moiety adopts an envelope