

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$)*

$$B_{\text{eq}} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B_{eq}
O1A	0.7749 (2)	0.0674 (2)	0.0912 (1)	4.32 (4)
C2A	0.9373 (4)	0.0593 (3)	0.1283 (2)	4.34 (6)
O2A	0.9486 (3)	-0.0345 (3)	0.1741 (1)	6.20 (6)
C3A	1.0748 (4)	0.1884 (3)	0.1029 (2)	4.53 (6)
C4A	0.9972 (3)	0.2664 (3)	0.0544 (2)	3.91 (6)
C5A	0.7979 (3)	0.1977 (3)	0.0441 (2)	3.52 (5)
O6A	0.7086 (2)	0.1083 (2)	-0.0445 (1)	3.44 (4)
C7A	0.6647 (3)	0.2107 (3)	-0.0982 (2)	3.32 (5)
O8A	0.8263 (2)	0.3123 (2)	-0.1180 (1)	3.54 (4)
C9A	0.8134 (3)	0.2735 (3)	-0.2053 (2)	3.80 (5)
O9A	0.9240 (3)	0.3476 (3)	-0.2393 (1)	5.26 (5)
C10A	0.6411 (3)	0.1315 (3)	-0.2482 (2)	3.98 (6)
C11A	0.5577 (3)	0.0956 (3)	-0.1866 (2)	3.77 (6)
C12A	0.5797 (3)	0.3333 (3)	-0.0549 (2)	4.15 (6)
O13A	0.6921 (3)	0.4331 (2)	0.0282 (1)	4.63 (4)
C14A	0.7130 (4)	0.3246 (4)	0.0832 (2)	4.76 (7)
O1B	0.4665 (2)	0.4320 (2)	0.6281 (1)	3.92 (4)
C2B	0.4331 (3)	0.4318 (3)	0.7092 (2)	3.92 (6)
O2B	0.5349 (3)	0.5313 (3)	0.7743 (1)	5.90 (6)
C3B	0.2619 (3)	0.2954 (3)	0.6988 (2)	3.97 (6)
C4B	0.1974 (3)	0.2226 (3)	0.6147 (2)	3.69 (5)
C5B	0.3249 (3)	0.3007 (3)	0.5621 (2)	3.34 (5)
O6B	0.2543 (2)	0.3923 (2)	0.5083 (1)	3.46 (4)
C7B	0.1486 (3)	0.2929 (3)	0.4245 (2)	3.38 (5)
O8B	-0.0244 (2)	0.1945 (2)	0.4358 (1)	3.78 (4)
C9B	-0.1524 (3)	0.2393 (3)	0.3893 (2)	4.04 (6)
O9B	-0.3090 (2)	0.1691 (3)	0.3844 (2)	5.78 (6)
C10B	-0.0667 (4)	0.3759 (3)	0.3497 (2)	4.40 (6)
C11B	0.1064 (3)	0.4095 (3)	0.3716 (2)	4.05 (6)
C12B	0.2298 (3)	0.1682 (3)	0.3798 (2)	4.00 (6)
O13B	0.2763 (2)	0.0695 (2)	0.4358 (1)	4.30 (4)
C14B	0.4052 (4)	0.1813 (3)	0.5126 (2)	4.33 (6)

Table 2. *Selected geometric parameters ($\text{\AA}$, $^\circ$)*

O1A—C2A	1.374 (4)	O1B—C2B	1.359 (3)
O1A—C5A	1.432 (3)	O1B—C5B	1.437 (2)
C2A—O2A	1.188 (4)	C2B—O2B	1.201 (3)
C2A—C3A	1.480 (4)	C2B—C3B	1.480 (3)
C3A—C4A	1.305 (4)	C3B—C4B	1.309 (3)
C4A—C5A	1.511 (3)	C4B—C5B	1.510 (4)
C5A—O6A	1.423 (3)	C5B—O6B	1.419 (3)
C5A—C14A	1.508 (4)	C5B—C14B	1.503 (4)
O6A—C7A	1.404 (3)	O6B—C7B	1.414 (2)
C7A—O8A	1.455 (3)	C7B—O8B	1.460 (3)
C7A—C11A	1.503 (3)	C7B—C11B	1.489 (4)
C7A—C12A	1.506 (4)	C7B—C12B	1.506 (4)
O8A—C9A	1.344 (3)	O8B—C9B	1.360 (3)
C9A—O9A	1.199 (3)	C9B—O9B	1.211 (3)
C9A—C10A	1.499 (3)	C9B—C10B	1.471 (4)
C10A—C11A	1.308 (4)	C10B—C11B	1.314 (4)
C12A—O13A	1.423 (3)	C12B—O13B	1.411 (4)
O13A—C14A	1.410 (4)	O13B—C14B	1.429 (3)
C2A—O1A—C5A	110.4 (2)	C9A—C10A—C11A	107.9 (2)
O1A—C2A—O2A	121.2 (3)	C7A—C11A—C10A	110.3 (2)
O1A—C2A—C3A	107.1 (2)	C7A—C12A—O13A	109.4 (2)
O2A—C2A—C3A	131.6 (3)	C12A—O13A—C14A	109.8 (2)
C2A—C3A—C4A	109.1 (2)	C5A—C14A—O13A	111.2 (2)
C3A—C4A—C5A	109.9 (2)	C7B—O8B—C9B	108.5 (2)
O1A—C5A—C4A	103.5 (2)	O8B—C9B—O9B	121.4 (3)
O1A—C5A—O6A	104.8 (2)	O8B—C9B—C10B	108.9 (2)
O1A—C5A—C14A	107.7 (2)	O9B—C9B—C10B	129.7 (3)
C4A—C5A—O6A	111.9 (2)	C9B—C10B—C11B	108.3 (3)
C4A—C5A—C14A	116.0 (2)	C2B—O1B—C5B	109.9 (2)
O6A—C5A—C14A	111.9 (2)	O1B—C2B—O2B	121.2 (2)
C5A—O6A—C7A	115.4 (2)	O1B—C2B—C3B	108.3 (2)
O6A—C7A—O8A	109.0 (2)	O2B—C2B—C3B	130.5 (3)
O6B—C7B—C11B	109.2 (2)	C2B—C3B—C4B	108.1 (2)
O6B—C7B—C12B	112.4 (2)	C3B—C4B—C5B	110.2 (2)
O8B—C7B—C11B	104.0 (2)	O1B—C5B—C4B	103.4 (2)
O8B—C7B—C12B	108.2 (2)	O1B—C5B—O6B	104.2 (2)

C11B—C7B—C12B	114.3 (2)	O1B—C5B—C14B	107.7 (2)
O6A—C7A—C11A	108.7 (2)	C4B—C5B—O6B	112.6 (2)
O6A—C7A—C12A	112.7 (2)	C4B—C5B—C14B	115.6 (2)
O8A—C7A—C11A	103.6 (2)	O6B—C5B—C14B	112.1 (2)
O8A—C7A—C12A	107.7 (2)	C5B—O6B—C7B	115.7 (2)
C11A—C7A—C12A	114.7 (2)	O6B—C7B—O8B	108.3 (2)
C7A—O8A—C9A	109.8 (2)	C7B—C11B—C10B	110.2 (2)
O8A—C9A—O9A	123.0 (2)	C7B—C12B—O13B	111.1 (2)
O8A—C9A—C10A	108.3 (2)	C12B—O13B—C14B	109.3 (2)
O9A—C9A—C10A	128.6 (2)	C5B—C14B—O13B	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 Diffraction 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]

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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

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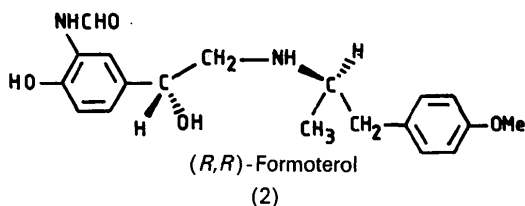
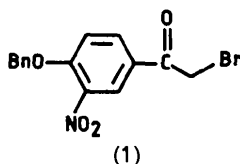
Abstract

In the title compound, 4-benzyloxy-3-nitrophenacyl bromide, $\text{C}_{15}\text{H}_{12}\text{BrNO}_4$, the two planar nitrophenyl and benzyloxy groups are inclined at a dihedral angle of $17.1 (1)^\circ$. The NO_2 group is twisted out of the

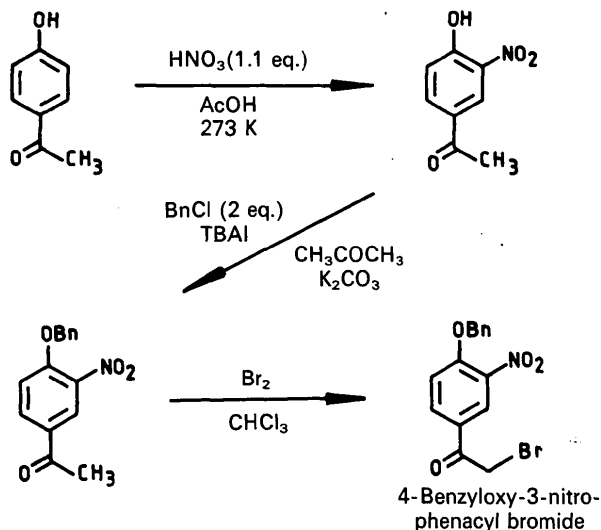
plane of the phenyl ring by 20.4 (3)° to minimize steric hindrance between O(4) and O(3); the O(4)···O(3) separation is 2.600 (5) Å and the angle O(4)···O(3)—N(1) is 88.2 (4)°.

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 bronchodilator activity (Ida, 1976; Murase, Mase, Ida, Takahashi & Murakami, 1977; Trofast, Osterberg, Kallstrom & Waldeck, 1991).



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



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 NO₂ 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.

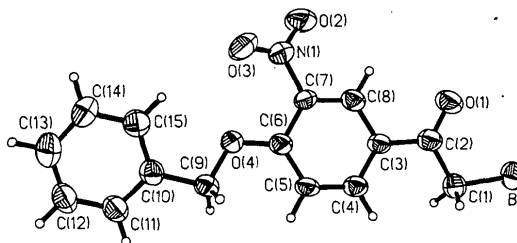


Fig. 1. A perspective view of the molecule with atom labelling.

Experimental

Crystal data

C₁₅H₁₂BrNO₄

M_r = 350.17

Monoclinic

*P*2₁/*c*

a = 7.877 (1) Å

b = 12.600 (2) Å

c = 14.326 (2) Å

β = 96.68 (1)°

V = 1412.2 (4) Å³

Z = 4

D_x = 1.638 (3) Mg m⁻³

Mo *K*α 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* ≥ 3σ(*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%

RefinementRefinement on F $R = 0.031$ $wR = 0.038$ $S = 1.06$

1219 reflections

190 parameters

 $w = 1/[\sigma^2(F) + 0.0030|F|^2]$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Atomic scattering factors

from *SHELXTL-Plus*

(Sheldrick, 1991)

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]

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

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

	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 ($\text{\AA}$, $^\circ$)

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).

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2-Methyl-2-(4-nitrophenylazo)-1,3-indandione, $\text{C}_{16}\text{H}_{11}\text{N}_3\text{O}_4$

SÜHEYLA ÖZBEY AND ENGİN KENDİ

Department of Physics Engineering,
 Faculty of Engineering, Hacettepe University,
 06532 Beytepe, Ankara, Turkey

NERMİN HOCAOĞLU AND TAHSİN UYAR

Chemistry Department, Art and Science Faculty,
 Gazi University, 06503 Ankara, Turkey

THOMAS C. W. MAK

Department of Chemistry, The Chinese University of
 Hong Kong, Shatin, New Territories, Hong Kong

GIOVANNI LUCA CASCARANO

Inst. di Ricerca per lo Sviluppo di Metod Crist,
 70124 Bari Campus Univ., Bari, Italy

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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