C(6)	0.5635 (4)	0.5047 (3)	0.7990 (6)	3.5 (2)
C(7)	0.3012 (3)	0.5409 (3)	0.6098 (5)	3.0 (2)
C(8)	0.1924 (4)	0.5060 (4)	0.5007 (6)	3.9 (2)
C(9)	0.0795 (4)	0.5840 (4)	0.4312 (6)	4.2 (2)
C(10)	0.0725 (4)	0.6980 (3)	0.4733 (5)	3.4 (2)
C(11)	0.1774 (4)	0.7345 (3)	0.5831 (6)	3.9 (2)
C(12)	0.2918 (4)	0.6561 (3)	0.6504 (6)	3.5 (2)
C(13)	0.8038 (4)	0.1100 (4)	0.8184 (7)	4.4 (2)
C(14)	0.6616 (4)	0.0815 (3)	1.0709 (6)	3.6 (2)
C(15)	0.6553 (4)	0.0481 (4)	1.2361 (7)	4.2 (2)
C(16)	0.5433 (5)	0.0860 (4)	1.3059 (7)	4.6 (3)
C(17)	0.4350 (4)	0.1575 (3)	1.2068 (7)	4.0 (2)
C(18)	0.4365 (4)	0.1907 (3)	1.0414 (7)	4.1 (2)
C(19)	0.5499 (4)	0.1530 (3)	0.9718 (6)	3.8 (2)

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

S(1)—C(6)	1.809 (5)	C(3)—C(4)	1.386 (6)
S(1)—C(7)	1.764 (3)	C(4)—C(5)	1.374 (6)
S(2)—C(13)	1.808 (6)	C(5)—C(13)	1.510 (6)
S(2)—C(14)	1.759 (4)	C(7)—C(8)	1.392 (6)
O(1)—N(2)	1.218 (6)	C(7)—C(12)	1.379 (6)
O(2)—N(2)	1.215 (5)	C(8)—C(9)	1.370 (5)
O(3)—N(3)	1.217 (5)	C(9)—C(10)	1.370 (6)
O(4)—N(3)	1.223 (8)	C(10)—C(11)	1.370 (6)
N(1)—C(1)	1.340 (5)	C(11)—C(12)	1.378 (5)
N(1)—C(5)	1.346 (4)	C(14)—C(15)	1.389 (7)
N(2)—C(10)	1.462 (5)	C(14)—C(19)	1.401 (5)
N(3)—C(17)	1.464 (7)	C(15)—C(16)	1.371 (7)
C(1)—C(2)	1.381 (6)	C(16)—C(17)	1.380 (6)
C(1)—C(6)	1.507 (5)	C(17)—C(18)	1.373 (8)
C(2)—C(3)	1.375 (5)	C(18)—C(19)	1.384 (7)
C(6)—S(1)—C(7)	102.7 (2)	S(1)—C(7)—C(12)	126.6 (3)
C(13)—S(2)—C(14)	104.9 (2)	C(8)—C(7)—C(12)	118.5 (3)
C(1)—N(1)—C(5)	117.9 (3)	C(7)—C(8)—C(9)	121.0 (4)
O(1)—N(2)—O(2)	123.1 (4)	C(8)—C(9)—C(10)	119.5 (4)
O(1)—N(2)—C(10)	117.9 (4)	N(2)—C(10)—C(9)	119.3 (4)
O(2)—N(2)—C(10)	119.0 (4)	N(2)—C(10)—C(11)	120.0 (4)
O(3)—N(3)—O(4)	122.9 (5)	C(9)—C(10)—C(11)	120.7 (3)
O(3)—N(3)—C(17)	118.9 (5)	C(10)—C(11)—C(12)	119.9 (4)
O(4)—N(3)—C(17)	118.2 (4)	C(7)—C(12)—C(11)	120.5 (4)
N(1)—C(1)—C(2)	122.7 (3)	S(2)—C(13)—C(5)	114.8 (3)
N(1)—C(1)—C(6)	116.4 (3)	S(2)—C(14)—C(15)	115.9 (3)
C(2)—C(1)—C(6)	120.9 (4)	S(2)—C(14)—C(19)	125.0 (4)
C(1)—C(2)—C(3)	118.9 (4)	C(15)—C(14)—C(19)	119.1 (4)
C(2)—C(3)—C(4)	119.0 (4)	C(14)—C(15)—C(16)	121.4 (4)
C(3)—C(4)—C(5)	118.9 (3)	C(15)—C(16)—C(17)	118.5 (5)
N(1)—C(5)—C(4)	122.6 (4)	N(3)—C(17)—C(16)	119.2 (5)
N(1)—C(5)—C(13)	115.2 (3)	N(3)—C(17)—C(18)	118.9 (4)
C(4)—C(5)—C(13)	122.1 (3)	C(16)—C(17)—C(18)	121.9 (4)
S(1)—C(6)—C(1)	109.6 (3)	C(17)—C(18)—C(19)	119.6 (4)
S(1)—C(7)—C(8)	114.9 (3)	C(14)—C(19)—C(18)	119.6 (5)

S(1)—C(6)—C(1)—N(1)	6.8 (5)
S(1)—C(6)—C(1)—C(2)	−174.3 (3)
S(2)—C(13)—C(5)—N(1)	−112.0 (4)
S(2)—C(13)—C(5)—C(4)	68.1 (5)
O(1)—N(2)—C(10)—C(9)	178.2 (4)
O(1)—N(2)—C(10)—C(11)	−3.4 (6)
O(2)—N(2)—C(10)—C(9)	−2.0 (6)
O(2)—N(2)—C(10)—C(11)	176.4 (4)
O(3)—N(3)—C(17)—C(16)	170.9 (4)
O(3)—N(3)—C(17)—C(18)	−9.3 (6)
O(4)—N(3)—C(17)—C(16)	−9.0 (7)
O(4)—N(3)—C(17)—C(18)	170.7 (5)
C(1)—C(6)—S(1)—C(7)	−179.9 (3)
C(5)—C(13)—S(2)—C(14)	67.7 (4)
C(6)—S(1)—C(7)—C(8)	174.8 (3)
C(6)—S(1)—C(7)—C(12)	−5.2 (4)
C(13)—S(2)—C(14)—C(15)	−166.0 (3)
C(13)—S(2)—C(14)—C(19)	16.0 (4)

The structure was solved by direct methods (Gilmore, 1984) and successive Fourier syntheses. Refinement was performed with full-matrix least-squares methods, with non-H atoms anisotropic and H atoms with fixed displacement parameters (1.2 × B_{eq} of the carrying atom). The calculations were performed with *TEXSAN* (Molecular Structure Corporation, 1989) software using a VAXstation 3520 computer. The figures were drawn using *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the IUCr (Reference: AB1100). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-(*o*-Methoxyphenoxy)-1-methylbenzimidazole, $C_{15}H_{14}N_2O_2$

K. T. Vasudevan and Puttaraja

Department of Physics, Bangalore University, Bangalore 560056, India

and M. V. Kulkarni

Department of Chemistry, Central College, Bangalore University, Bangalore 560001, India

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Abstract

The benzimidazole ring is planar and makes a dihedral angle of 79.02 (2)° with the aryloxy ring. The N—CH_3 and O—CH_3 groups are *anti* to each other. The molecules are held together in the crystal by van der Waals interactions.

Comment

Several 1-dialkylaminoalkyl-2-aryloxybenzimidazoles have been found to exhibit excellent muscle-relaxant

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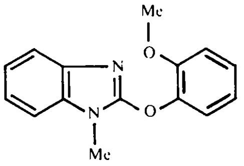
(Marshal, Jones & Weaver, 1964) and analgesic properties (Aka, Sasajima & Watanabe, 1965, 1966). The present compound, (I), is one of several *N*-methyl-2-aryloxybenzimidazoles possessing the pharmacologically useful iminoether configuration. The title compound was synthesized by the reaction of guaiacol (*o*-methoxyphenol) with 2-chloro-*N*-methylbenzimidazole. Its chemical structure along with its ^{1}H NMR and mass spectral fragmentation have been reported (Kulkarni & Patil, 1981). In order to establish its structure in the solid state, an X-ray analysis of this compound was undertaken.



(I)

The benzimidazole ring is coplanar with the N—CH$_3$ group. The dihedral angle between the benzene and imidazole rings is 0.7 (1)°. The aryloxy group at C(2) makes a dihedral angle of 79.0 (2)° with the benzimidazole ring and is bound equatorially. The bond lengths observed for N(1)—C(2) [1.360 (2) Å] and C(2)—N(3) [1.295 (2) Å] are in agreement with the values reported for benzimidazole (Escande & Galigne, 1974) and 2-chloro-1-(β-D-ribofuranosyl)benzimidazole (Sprang & Sundaralingam, 1973). The observed bond length C(2)—O(11) of 1.348 (2) Å is normal for a C_{sp^2}—O bond, showing the expected residual bond contraction of 0.07 Å due to oxygen (Ferguson, 1969). However, the C(12)—O(11) bond length of 1.410 (2) Å is comparatively longer. This has been observed by many groups (Kálmán & Argay, 1982; Brianso, 1980). The orientation of the methoxy group attached to aromatic systems has been of spectroscopic (Hoffer, 1975; Markriannis & Knittel, 1979) and crystallographic (Sakaki *et al.*, 1976) interest. Furthermore, the conformations of the individual arylmethoxy groups relative to the aromatic ring have been of vital importance in the study of the hallucinogenic potency of polymethoxyphenylamines (Braun, Braun, Jacob, Nicholas & Shulgin, 1978). The methoxy group at C(13) is *anti* to the N—CH$_3$ group of benzimidazole. The O(18)—C(19) bond of the methoxy group is *cis* to the aromatic C(13)—C(14) bond. The O(18)—C(13)—C(14) angle [125.2 (2)°] is larger than the O(18)—C(13)—C(12) angle [116.9 (2)°]. Similar observations have been reported for 5-methoxyindole derivatives and have been attributed to the repulsion between C(19) and C(14) (Sakaki, Wakahara, Fujiwara & Tomita, 1975; Chandrakantha, Nirmala, Puttaraja & Nethaji, 1990).

The methoxy C atom is −0.192 (3) Å out of the plane formed by the atoms C(12), C(13), C(14), C(15), C(16), C(17), O(18) and C(19). The C(19)—O(18)—C(13)—C(14) torsion angle of −7.6 (2)° indicates a synperiplanar arrangement. Furthermore, it is interesting to note the difference in bond lengths between O(18)—C(19) [1.436 (3) Å] and O(11)—C(12) [1.410 (2) Å] due to the fact that O(18) and O(11) are linked to methyl and benzimidazole moieties, respectively. The two *ortho* atoms O(11) and O(18) are separated by 2.695 (2) Å.

The conformation of the molecule about the C(2)—O(11) bond is *gauche* and the N(3)—C(2)—O(11)—C(12) [1.92 (27)°] and O(18)—C(13)—C(12)—O(11) [1.08 (27)°] torsion angles indicate a synperiplanar arrangement. There are no intermolecular hydrogen bonds, except for a contact of 2.48 (3) Å between H(10) and O(11); the molecules are mainly held together in the crystal by van der Waals forces.

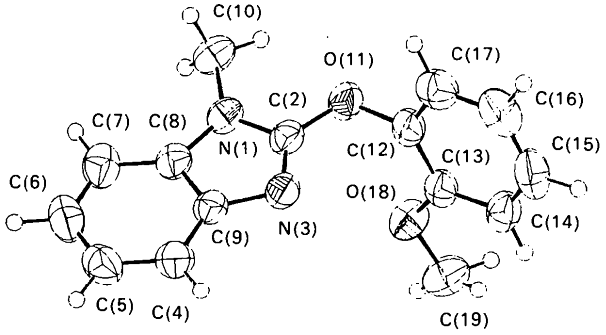


Fig. 1. View of the molecule (*PLATON*; Spek, 1990) with displacement ellipsoids plotted at the 50% probability level. H atoms are represented by spheres of arbitrary size.

Experimental

Crystal data

$C_{15}H_{14}N_2O_2$
M_r = 254.28
Monoclinic
$P2_1/n$
a = 7.388 (3) Å
b = 10.432 (1) Å
c = 16.929 (2) Å
β = 100.29 (2)°
V = 1283.9 (1) Å^3
Z = 4
D_x = 1.313 Mg m^{-3}
D_m = 1.29 (4) Mg m^{-3}
D_m measured by flotaion in KI solution

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 0–20°
μ = 0.052 mm^{-1}
T = 300 K
Needles
0.5 × 0.25 × 0.17 mm
Colourless
Crystal source: grown from chloroform

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2852 measured reflections
2510 independent reflections
1829 observed reflections [$I \geq 3\sigma(I)$]
$R_{\text{int}} = 0.045$
$\theta_{\max} = 27°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 20$
2 standard reflections monitored every 140 reflections intensity variation: no significant variation

Refinement

Refinement on F
$R = 0.051$
$wR = 0.051$
1829 reflections
228 parameters
H atoms refined isotropically
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.056$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Atomic scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965)

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

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

	x	y	z	U_{eq}
N(1)	0.7351 (2)	0.0474 (2)	0.0215 (1)	0.0463 (5)
C(2)	0.7347 (2)	0.1731 (2)	0.0428 (1)	0.0449 (6)
N(3)	0.7693 (2)	0.2548 (2)	−0.0102 (1)	0.0493 (5)
C(4)	0.8337 (3)	0.2113 (2)	−0.1485 (1)	0.0570 (7)
C(5)	0.8529 (3)	0.1150 (3)	−0.2018 (1)	0.0665 (9)
C(6)	0.8349 (3)	−0.0138 (3)	−0.1818 (1)	0.0691 (9)
C(7)	0.7955 (3)	−0.0499 (2)	−0.1085 (1)	0.0594 (8)
C(8)	0.7758 (2)	0.0479 (2)	−0.0555 (1)	0.0464 (6)
C(9)	0.7957 (2)	0.1770 (2)	−0.0743 (1)	0.0462 (6)
C(10)	0.7098 (2)	−0.0631 (2)	0.0706 (2)	0.0558 (8)
O(11)	0.6983 (2)	0.2000 (1)	0.1162 (1)	0.0554 (5)
C(12)	0.6934 (3)	0.3317 (2)	0.1346 (1)	0.0484 (7)
C(13)	0.5363 (3)	0.4023 (2)	0.1051 (1)	0.0469 (6)
C(14)	0.5345 (3)	0.5310 (2)	0.1252 (1)	0.0569 (8)
C(15)	0.6836 (4)	0.5841 (2)	0.1749 (2)	0.0700 (10)
C(16)	0.8365 (4)	0.5121 (3)	0.2052 (1)	0.0730 (10)
C(17)	0.8407 (3)	0.3846 (2)	0.1839 (1)	0.0627 (8)
O(18)	0.3960 (2)	0.3388 (1)	0.0583 (1)	0.0595 (5)
C(19)	0.2250 (3)	0.4060 (3)	0.0356 (2)	0.0694 (10)

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

C(10)—N(1)	1.452 (3)	C(2)—O(11)	1.348 (2)
C(2)—N(1)	1.360 (2)	O(11)—C(12)	1.410 (2)
C(2)—N(3)	1.295 (2)	C(12)—C(17)	1.365 (3)
C(9)—N(3)	1.397 (2)	C(17)—C(16)	1.380 (3)
C(9)—C(8)	1.398 (3)	C(16)—C(15)	1.377 (4)
C(8)—C(7)	1.383 (3)	C(15)—C(14)	1.378 (3)
C(7)—C(6)	1.376 (3)	C(14)—C(13)	1.386 (3)
C(6)—C(5)	1.399 (4)	C(13)—O(18)	1.360 (2)
C(5)—C(4)	1.375 (3)	O(18)—C(19)	1.436 (3)
C(4)—C(9)	1.383 (3)		
C(10)—N(1)—C(2)	127.4 (2)	N(3)—C(2)—O(11)	126.7 (2)
C(10)—N(1)—C(8)	127.7 (2)	C(2)—O(11)—C(12)	115.1 (1)
N(1)—C(8)—C(7)	132.2 (2)	C(13—O(18)—C(19)	117.4 (2)
C(8)—C(7)—C(6)	116.6 (2)	C(13)—C(14)—C(15)	119.8 (2)
C(7)—C(6)—C(5)	121.8 (2)	C(14)—C(15)—C(16)	121.5 (2)
C(6)—C(5)—C(4)	121.1 (2)	C(15)—C(16)—C(17)	118.9 (2)
C(5)—C(4)—C(9)	118.0 (2)	C(16)—C(17)—C(12)	119.8 (2)
C(4)—C(9)—C(8)	120.1 (2)	C(17)—C(12)—C(13)	121.9 (2)
C(9)—C(8)—C(7)	122.4 (2)	C(12)—C(13)—C(14)	118.0 (2)
C(9)—C(8)—N(1)	105.4 (2)	C(8)—C(9)—N(3)	110.4 (3)
C(8)—N(1)—C(2)	104.8 (1)	C(9)—N(3)—C(2)	103.2 (2)
N(1)—C(2)—N(3)	116.2 (2)		

The structure was solved by direct methods using *MULTAN*80 (Main *et al.*, 1980) and *SHELX*76 (Sheldrick, 1976). All H atoms were located from difference Fourier maps.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates and a molecular packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71707 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1052]

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