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Structure of 5-Benzylamino-1-methyl-4-nitroimidazole

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Abstract. $C_{11}H_{12}N_4O_2$, $M_r = 232.24$, orthorhombic, *Pbca*, $a = 6.962\ (4)$, $b = 13.754\ (7)$, $c = 22.974\ (12)$ Å, $V = 2200\ (2)$ Å^3, $Z = 8$, $D_m = 1.40$, $D_x = 1.402$ Mg m^{-3}, graphite-monochromated Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å, $\mu = 0.794$ mm^{-1}, $F(000) = 976$, $T = 293$ K, final $R = 0.035$ for 1143 unique observed $[F \geq 4\sigma(F)]$ reflections. The phenyl ring and the nitro group make angles of 50.8 and 3.6°, respectively, to the imidazole ring. An intermolecular hydrogen bond exists parallel to the *b* axis.

Experimental. Yellow crystals were obtained from 1,4-dioxane and were used for density measurements by flotation in *n*-heptane/CCl_4. A prismatic crystal with approximate dimensions $0.1 \times 0.2 \times 0.4$ mm was mounted for X-ray diffraction data collection on a Stoe Stadi-4 four-circle diffractometer with graphite-monochromated Cu $K\alpha$ (reflection 200) radiation. Lattice and space group, *Pbca*, were determined from cell dimensions, observed symmetry and systematic absences. Unit-cell dimensions were obtained by least-squares refinement of setting angles of 30 reflections with $30 \leq 2\theta \leq 50°$. X-ray intensities were collected in the $\omega/2\theta$ scan mode up to a maximum $\sin\theta/\lambda = 0.588$ Å^{-1}, corresponding to $2\theta_{\max} = 130°$, and for $0 \leq h \leq 8$, $0 \leq k \leq 16$, $-27 \leq l \leq 27$. Friedel pairs were also collected. Intensities of four standard reflections ($\bar{2}\bar{2}0$, $\bar{2}\bar{1}\bar{1}$, $\bar{1}0\bar{4}$, $00\bar{2}$), monitored every 60 min, showed an average decrease in intensity of 1.92% in 96 h of radiation. The reflection intensities were rescaled using a linear regression. A total of 5911 reflections were measured. Symmetry-related reflections were averaged to give 1816 unique reflections ($R_{\text{int, observed},F} = 0.027$) of which 1143 were considered observed with $F \geq 4\sigma(F)$. Data reduction was with a locally adapted Stoe & Co. (1985) *REDU4* program. Lorentz and polarization corrections were applied, and absorption corrections were performed by the method of North, Phillips & Mathews (1968) based on the observed absorption of five reflections (302, 210, $\bar{2}00$, $\bar{2}\bar{1}\bar{1}$, 111) as a function of ψ. Transmission factors varied between 0.81 and 1.00. Structure factors were calculated with scattering factors from Cromer & Waber (1974), and contracted H-atom form factors from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were performed for all non-H atoms (Ibers & Hamilton, 1964). The phase problem was succesfully solved by direct methods using *MULTAN82* (Main *et al.*, 1982) which revealed the positions of all non-H atoms. Full-matrix least-squares refinements were performed on *F*, first isotropically and then anisotropically. The 12 H atoms were located in a difference map. H-atom positions were refined and U_{iso} was fixed at 0.05 Å^2. $w = 4F^2/[\sigma^2(F^2) + (0.03F^2)^2]$. Final $R = 0.035$, $wR = 0.035$, with $S = 2.05$. Largest parameter shift/e.s.d. = 0.03. The ratio of reflections to refined parameters was 5.8. The residual electron density varied between -0.11 and 0.13 e Å^{-3}. All calculations were performed on a Digital PDP-11/73 and a MicroVAX 2000 microcomputer using *SDP/VAX* (Enraf–Nonius, 1985) and *PARST* (Nardelli, 1983). An

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*ORTEP*II view (Johnson, 1976) of the title compound with the atomic numbering scheme is shown in Fig. 1. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1. Bond lengths, bond angles and selected torsion angles are given in Table 2*.

The imidazole ring and the phenyl ring are almost planar with maximum deviations from the best planes through the corresponding moieties of 0.005 (3) and 0.008 (3) Å. The dihedral angle between the least-squares planes through the imidazole and the phenyl rings is 50.8 (1)°. C52 definitely does not lie in the phenyl plane ($\Delta/\sigma = 21$). The nitro group and the planar amine N51 lie almost in the imidazole plane. The dihedral angles are 3.6 (2) and 1.6 (3)°, respectively. The alignment of the planar amine parallel to the imidazole ring is stabilized by an intramolecular hydrogen bond between N51 and O43. Inspection of non-bonding distances shorter than the sum of the van der Waals radii revealed the hydrogen bonds summarized in Table 3. The intermolecular hydrogen bond is parallel to the *b* axis. Fig. 2 shows the stacking of the imidazole rings perpendicular to the *a* axis.

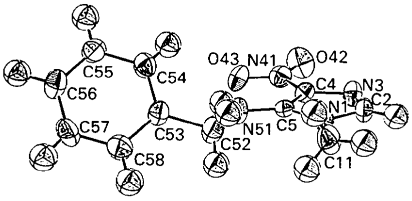

Fig. 1. An *ORTEP*II plot (Johnson, 1976) of the title compound with atomic numbering scheme. The ellipsoids enclose 50% probability.

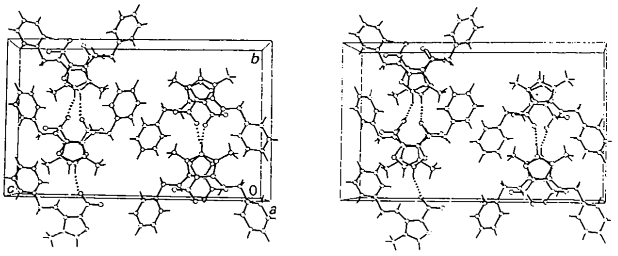

Fig. 2. A *PLUTO* plot (Motherwell & Clegg, 1978) of the crystal packing (viewed along **a**). Dotted lines indicate intermolecular hydrogen bonds.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* ($Å^2 \times 10^2$)

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

	x	*y*	*z*	U_{eq}
N1	0.0352 (4)	0.7429 (2)	0.3008 (1)	3.46 (7)
C2	0.0095 (5)	0.7834 (2)	0.2457 (2)	4.43 (9)
N3	0.0523 (5)	0.7263 (2)	0.2032 (1)	4.44 (7)
C4	0.1086 (5)	0.6425 (2)	0.2309 (1)	3.44 (9)
C5	0.1012 (5)	0.6505 (2)	0.2916 (1)	3.08 (9)
C11	−0.0110 (6)	0.7945 (2)	0.3545 (1)	5.4 (1)
N41	0.1627 (4)	0.5602 (2)	0.2005 (1)	4.47 (9)
O42	0.1553 (4)	0.5592 (2)	0.1465 (1)	7.12 (9)
O43	0.2221 (4)	0.4876 (2)	0.2292 (1)	5.05 (7)
N51	0.1440 (4)	0.5830 (2)	0.3312 (1)	4.12 (9)
C52	0.1393 (6)	0.5894 (2)	0.3939 (2)	5.4 (1)
C53	0.1454 (5)	0.4907 (2)	0.4221 (1)	3.86 (9)
C54	0.0393 (6)	0.4141 (2)	0.4017 (1)	4.45 (9)
C55	0.0408 (6)	0.3252 (2)	0.4300 (1)	4.57 (9)
C56	0.1464 (6)	0.3144 (2)	0.4803 (1)	5.0 (1)
C57	0.2503 (6)	0.3903 (3)	0.5013 (1)	5.2 (1)
C58	0.2514 (6)	0.4783 (3)	0.4726 (1)	4.86 (9)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

N1—C2	1.393 (4)	N41—O43	1.266 (3)
N1—C5	1.368 (4)	N51—C52	1.444 (4)
N1—C11	1.461 (4)	C52—C53	1.505 (5)
C2—N3	1.287 (4)	C53—C54	1.369 (5)
N3—C4	1.373 (4)	C54—C55	1.386 (5)
C4—C5	1.399 (4)	C55—C56	1.377 (5)
C4—N41	1.382 (4)	C56—C57	1.358 (5)
C5—N51	1.334 (4)	C57—C58	1.378 (5)
N41—O42	1.242 (3)	C58—C53	1.386 (5)
C2—N1—C5	105.9 (2)	C4—N41—O43	118.2 (3)
C2—N1—C11	123.1 (2)	O42—N41—O43	121.7 (3)
C5—N1—C11	131.0 (3)	C5—N51—C52	129.4 (3)
N1—C2—N3	114.5 (3)	N51—C52—C53	112.0 (3)
C2—N3—C4	103.1 (3)	C52—C53—C54	122.1 (3)
N3—C4—C5	112.6 (3)	C52—C53—C58	119.1 (3)
N3—C4—N41	122.1 (3)	C54—C53—C58	118.7 (3)
C5—C4—N41	125.3 (3)	C53—C54—C55	120.9 (3)
N1—C5—C4	103.9 (3)	C54—C55—C56	119.5 (3)
N1—C5—N51	128.0 (3)	C55—C56—C57	120.0 (3)
C4—C5—N51	128.1 (3)	C56—C57—C58	120.6 (3)
C4—N41—O42	120.1 (3)	C53—C58—C57	120.3 (3)
N3—C4—N41—O42	−2.4 (5)	C4—C5—N51—C52	179.4 (4)
N3—C4—N41—O43	176.4 (3)	C5—N51—C52—C53	164.3 (3)
C5—C4—N41—O42	176.4 (3)	C52—C53—C54—C55	−177.0 (3)
C5—C4—N41—O43	−4.7 (4)	C52—C53—C58—C57	176.1 (3)

Table 3. *Geometry of intra- and intermolecular hydrogen bonds* (Å, °)

H-atom positions are normalized to C—H = 1.08 and N—H = 1.01 Å (Allen *et al.*, 1987).

Donor—H	Donor···Acceptor	H···Acceptor	Donor—H···Acceptor
N51—H51	N51···O43^{i}	H51···O43^{i}	N51—H51···O43^{i}
1.01	2.740 (4)	1.986 (3)	129.4 (2)
C2—H2	C2···O43^{ii}	H2···O43^{ii}	C2—H2···O43^{ii}
1.08	3.289 (4)	2.260 (3)	158.6 (2)

Symmetry code: (i) x, y, z; (ii) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Related literature. Nitroimidazoles are generally known as antiprotozoic and antibiotic drugs (Edwards, 1981). Accurate structural parameters on unsubstituted imidazole have been reported by McMullan, Epstein, Ruble & Craven (1979).

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[*] Lists of structure factors, anisotropic thermal parameters, bond distances and angles involving H atoms and details of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55674 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR1000]

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Structure of 11-(4-Bromophenyl)-1,2,3,4,5,6-hexahydro-1,6-methano-1,6-benzodiazocine

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Abstract. $C_{17}H_{17}BrN_2$, $M_r = 329.25$, triclinic, $P\bar{1}$, $a = 6.981\,(7)$, $b = 11.443\,(9)$, $c = 9.255\,(11)$ Å, $\alpha = 76.15\,(8)$, $\beta = 85.75\,(9)$, $\gamma = 74.13\,(7)°$, $V = 690.4\,(12)$ Å^3, $Z = 2$, $D_m = 1.57\,(6)$, $D_x = 1.58$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 31.4$ cm^{-1}, $F(000) = 336$, room temperature, $R = 0.057$, $wR = 0.069$ for 1534 reflections with $|F_o| \geq 3\sigma(F_o)$. The crystal structure determination of the title compound establishes the conformation as *endo*.

Experimental. The compound was prepared by the reaction of 1,2,3,4,5,6-hexahydro-1,6-benzodiazocine with 4-bromobenzaldehyde. A colourless crystal with dimensions 0.5 × 0.3 × 0.2 mm (D_m by flotation in CCl_4 and n-hexane) was used for data collection on a Syntex $P2_1$ diffractometer. Unit-cell parameters were determined from least-squares treatment of 15 reflections with $7.58 \leq 2\theta \leq 14.84°$. Intensities were measured by ω–2θ scans for $0 < 2\theta < 55°$ and from $[2\theta(\text{Mo } K\alpha_1) - 1]$ to $[2\theta(\text{Mo } K\alpha_2) + 1]°$ with variable scan speed 4.88–29.30° min^{-1} ($0 \leq h \leq 9$, $-14 \leq k \leq 14$, $-12 \leq l \leq 12$). 3184 unique reflections were measured, of which 1534 observed reflections with $|F_o| > 3\sigma(|F_o|)$ and maximum $(\sin\theta/\lambda) \leq 0.55$ Å^{-1} ($0 \leq h \leq 7$, $-11 \leq k \leq 12$, $-9 \leq l \leq 10$) were used for the structure solution and refinement. Two standards, ($0\bar{3}0$ and $\overline{1}\overline{1}\overline{2}$) measured after every 100 reflections, showed no appreciable trends. Lorentz and polarization corrections were applied, as well as an absorption correction based on ψ scans of seven reflections with $20.75 \leq 2\theta \leq 43.25°$ (transmission factor $A = 0.283$–1.0). No extinction correction was made.

The structure was solved by the heavy-atom method and refined by full-matrix least squares to $R = 0.057$, $wR = 0.069$, $S = 1.79$. The minimized quan-