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

BY MICHAL DUNAJ-JURČO

*Department of Inorganic Chemistry, Slovak Technical University, CS-*81237 *Bratislava, Czechoslovakia*

DUŠAN MIKLOŠ

*Institute of Inorganic Chemistry, Slovak Academy of Sciences, CS-*84236 *Bratislava, Czechoslovakia*

AND JÁN JENDRICHOVKSÝ

Drug Research Institute Modra, Horná 36, *CS-*90001 *Modra, Czechoslovakia*

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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}, λ(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 $\bar{1}\bar{1}2$) 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-

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Table 1. *Atomic coordinates and equivalent isotropic temperature parameters* ($Å^2$) *for non-H atoms with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}
Br	1.0095 (1)	0.71221 (8)	−0.20705 (9)	4.52 (3)
N(1)	0.1800 (8)	0.8674 (5)	0.2099 (5)	2.70 (16)
N(2)	0.2758 (8)	0.6485 (5)	0.3031 (6)	2.64 (16)
C(1)	0.7718 (10)	0.7222 (6)	−0.0922 (7)	3.13 (21)
C(2)	0.6551 (11)	0.8362 (7)	−0.0845 (8)	3.53 (24)
C(3)	0.4868 (11)	0.8439 (6)	−0.0005 (7)	3.39 (22)
C(4)	0.4352 (9)	0.7392 (6)	0.0782 (6)	2.40 (19)
C(5)	0.5610 (12)	0.6253 (7)	0.0670 (8)	3.38 (23)
C(6)	0.7272 (11)	0.6159 (7)	−0.0171 (8)	3.35 (22)
C(7)	0.2463 (10)	0.7483 (6)	0.1654 (7)	2.56 (20)
C(8)	0.2715 (9)	0.8365 (5)	0.3503 (7)	2.50 (19)
C(9)	0.2973 (12)	0.9136 (7)	0.4343 (8)	3.57 (25)
C(10)	0.3836 (12)	0.8633 (8)	0.5686 (8)	3.92 (26)
C(11)	0.4402 (11)	0.7372 (8)	0.6218 (8)	3.79 (26)
C(12)	0.4091 (11)	0.6578 (7)	0.5411 (8)	3.42 (23)
C(13)	0.3262 (10)	0.7098 (6)	0.4035 (7)	2.63 (20)
C(14)	0.1014 (13)	0.6023 (8)	0.3522 (9)	4.02 (27)
C(15)	0.9061 (15)	0.6933 (10)	0.3168 (14)	6.59 (41)
C(16)	0.8682 (15)	0.8182 (10)	0.3325 (15)	6.23 (40)
C(17)	0.9635 (12)	0.9086 (9)	0.2222 (10)	4.31 (28)

Table 2. *Interatomic distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Br—C(1)	1.894 (7)	C(7)—N(1)—C(8)	101.3 (4)
C(1)—C(2)	1.351 (10)	—C(17)	112.0 (7)
C(2)—C(3)	1.354 (10)	C(8)—N(1)—C(17)	111.6 (6)
C(3)—C(4)	1.370 (10)	C(7)—N(2)—C(13)	100.9 (5)
C(4)—C(5)	1.380 (9)	—C(14)	114.4 (5)
C(5)—C(6)	1.343 (11)	C(13)—N(2)—C(14)	112.3 (6)
C(6)—C(1)	1.353 (10)	Br—C(1)—C(2)	118.5 (6)
C(4)—C(7)	1.486 (9)	—C(6)	119.4 (5)
C(7)—N(1)	1.462 (9)	C(2)—C(1)—C(6)	122.1 (7)
—N(2)	1.478 (7)	C(1)—C(2)—C(3)	118.8 (7)
N(1)—C(8)	1.414 (8)	C(2)—C(3)—C(4)	121.5 (6)
C(8)—C(9)	1.361 (12)	C(3)—C(4)—C(5)	117.2 (6)
C(9)—C(10)	1.348 (11)	—C(7)	121.2 (6)
C(10)—C(11)	1.363 (11)	C(5)—C(4)—C(7)	121.6 (6)
C(11)—C(12)	1.374 (13)	C(4)—C(5)—C(6)	122.2 (7)
C(12)—C(13)	1.367 (9)	C(5)—C(6)—C(1)	118.3 (7)
C(13)—C(8)	1.368 (8)	C(4)—C(7)—N(1)	111.2 (6)
—N(2)	1.406 (10)	—N(2)	110.1 (5)
N(2)—C(14)	1.457 (11)	N(1)—C(7)—N(2)	107.2 (5)
C(14)—C(15)	1.476 (12)	N(1)—C(8)—C(9)	129.1 (5)
C(15)—C(16)	1.421 (17)	—C(13)	110.4 (6)
C(16)—C(17)	1.525 (15)	C(9)—C(8)—C(13)	120.5 (6)
C(17)—N(1)	1.460 (10)	C(8)—C(9)—C(10)	119.0 (7)
C(2)—H(2)	0.92 (9)	C(9)—C(10)—C(11)	120.8 (9)
C(3)—H(3)	1.01 (7)	C(10)—C(11)—C(12)	121.1 (7)
C(5)—H(5)	0.83 (7)	C(11)—C(12)—C(13)	117.5 (7)
C(6)—H(6)	0.94 (7)	C(8)—C(13)—C(12)	121.1 (7)
C(7)—H(7)	0.93 (8)	—N(2)	111.1 (5)
C(9)—H(9)	0.87 (8)	N(2)—C(13)—C(12)	127.8 (6)
C(10)—H(10)	1.01 (9)	N(2)—C(14)—C(15)	116.2 (7)
C(11)—H(11)	1.08 (8)	C(14)—C(15)—C(16)	121.9 (11)
C(12)—H(12)	0.89 (6)	C(15)—C(16)—C(17)	119.1 (10)
C(14)—H(141)	0.98 (8)	N(1)—C(17)—C(16)	115.1 (6)
—H(142)	0.96 (9)		
C(15)—H(151)	0.87 (7)	Shortest intermolecular contacts	
—H(152)	1.05 (18)	Br···C(11′)	3.343 (9)
C(16)—H(161)	1.18 (12)	Br···C(12′)	3.519 (8)
—H(162)	0.97 (8)	C(1)···C(11′)	3.587 (12)
C(17)—H(171)	0.89 (7)		
—H(172)	0.83 (9)		

tity was $\sum w(\Delta|F|)^2$, where $w = [\sigma^2(F_o) + 0.03F_o^2]^{-1}$. Initial calculations were performed on a NOVA 1200 computer with the *XTL/XTLE Structure Determination System* (Syntex, 1976), and final calculations were performed on a PC with the *SDS* system (Petříček & Malý, 1988).

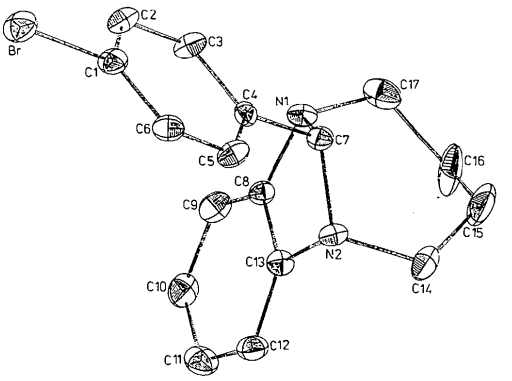

Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of a molecule of the title compound with atom labeling.

All atoms except the H atoms were refined anisotropically. All H atoms were located from the difference synthesis and refined isotropically. Scattering factors including the anomalous-scattering corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). $(\Delta/\sigma)_{max} = 0.13$. $-1.59 \le \Delta\rho \le 1.03$ e $Å^{-3}$ with extreme values near the Br atom. Atomic parameters are given in Table 1.* Bond distances and angles are given in Table 2. A view of the molecule is presented in Fig. 1.

Related literature. For previously reported *N,N′*-bridged benzodiazonines and benzodiazecines (Hayward & Meth-Cohn, 1975) the *exo* form was proposed by the interpretation of ^{1}H NMR spectra. For the title compound, the existence of only one conformer (either the *exo* or *endo* form) was observed by the interpretation of ^{1}H and ^{13}C NMR spectra (Jendrichovský, 1979), but it was not possible to decide which one.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55568 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0296]

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