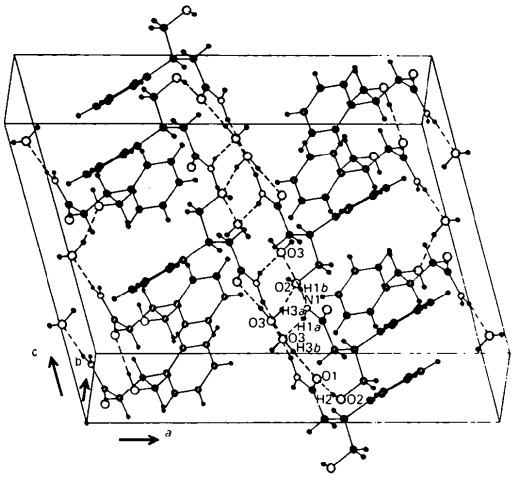

Fig. 3. A perspective drawing of the contents of one unit cell. The dashed lines indicate the intermolecular hydrogen bonds.

We are indebted to Consejo Nacional de Ciencia y Tecnologia, CONACYT, for funds which enabled us to obtain a single-phase uninterruptible power system to protect our diffractometer. We thank Mr Abelardo Cuellar for technical assistance. Project No. PCCBBNA-021262.

References

BAVIN, P. M. G., HANSELL, D. P. & SPICKETT, R. G. W. (1964). *J. Chem. Soc.* pp. 4535–4538.

BRISSE, F. & SYGUSCH, J. (1974). *Acta Cryst.* B**30**, 480–486.

CARVAJAL, G., RUSSEK, M., TAPIA, R. & MASSIEU, G. (1964). *Biochem. Pharmacol.* **13**, 1059–1060.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

JOSEPH-NATHAN, P., MASSIEU, G., CARVAJAL, G. & TAPIA, R. (1978). *Rev. Latinoam. Quim.* **9**, 90–92.

SHELDRICK, G. M. (1981). *SHELXTL, revision* 3. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.

SUTTON, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions.* Spec. Publ. No. 18. London: The Chemical Society.

Acta Cryst. (1984). C**40**, 2108–2109

Structure of 2,5-Dimethyl-1,3,4,6-tetraazacycl[3.3.3]azine,* $C_{10}H_9N_5$

BY GUY LAURENT,† JEAN-POL BOUTIQUE,‡§ GUY EVRARD,† JACQUES J. VERBIST‡ AND FRANÇOIS DURANT†

Laboratoire de Chimie Moléculaire Structurale and Laboratoire de Spectroscopie Electronique, Facultés Universitaires Notre-Dame de la Paix, 61 rue de Bruxelles, B-5000 Namur, Belgium

AND GEOFFREY J. ASHWELL

Sheffield City Polytechnic, Pond Street, Sheffield S1 1WB, England

(*Received* 6 *August* 1983; *accepted* 10 *September* 1984)

Abstract. $M_r = 199{\cdot}22$, monoclinic, $P2_1/m$, $a = 4{\cdot}022\ (1)$, $b = 17{\cdot}640\ (1)$, $c = 6{\cdot}568\ (1)$ Å, $\beta = 96{\cdot}02\ (1)°$, $V = 463{\cdot}42$ Å^3, $Z = 2$ (special positions), $D_x = 1{\cdot}43$ g cm^{-3}, Cu $K\bar{\alpha}$, $\lambda = 1{\cdot}54178$ Å, $\mu = 6{\cdot}73$ cm^{-1}, $F(000) = 208$, $T = 298$ K, final $R_w = 0{\cdot}045$ for 770 observed $[I \geq 2{\cdot}5\sigma(I)]$ reflections. The structure is planar. By comparison with the unmethylated compound the presence of methyl groups has no influence on either the bond lengths and angles, or the planarity of the molecule.

Introduction. The title compound belongs to the family of azacyclazines, which are particularly interesting for their special aromatic character, due to the π-electron delocalization of the nitrogens onto the whole molecule. Its crystal structure has been studied by X-ray diffraction and compared with the corresponding unmethylated derivative (Lindqvist, Ljungström, Andréasson & Ceder, 1978) to characterize the influence of methyl substituents on geometrical properties.

Experimental. Red-purple crystals obtained by slow evaporation from a chloroform solution. Crystal $0{\cdot}31 \times 0{\cdot}43 \times 0{\cdot}27$ mm, D_m not measured, 24 reflections ($62° \leq 2\theta \leq 80°$) used for measuring lattice parameters. 963 intensities collected, four-circle Enraf–

* 2,5-Dimethyl-1,3,4,6,9b-pentaazaphenalene.

† Laboratoire de Chimie Moléculaire Structurale.

‡ Laboratoire de Spectroscopie Electronique.

§ Research Assistant of the FNRS, Belgium.

0108-2701/84/122108-02$01.50

Table 1. *Atomic coordinates* ($\times 10^4$) *with standard deviations in parentheses and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a^*_i a^*_j \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}
N(1)	6001 (5)	2500	5229 (3)	3·1
C(2)	7742 (6)	2500	3484 (4)	3·3
N(3)	8573 (4)	1846 (1)	2657 (2)	4·0
C(4)	7592 (5)	1205 (1)	3530 (3)	3·9
N(5)	6003 (4)	1149 (1)	5177 (3)	4·2
C(6)	5201 (5)	1806 (1)	6073 (3)	3·6
C(7)	3571 (5)	1821 (1)	7829 (3)	4·5
C(8)	2769 (8)	2500	8692 (5)	4·8
C(9)	8392 (6)	483 (1)	2495 (4)	5·6

Nonius diffractometer (CAD-4 system, graphite monochromator, ω–2θ scan method, $2° \le \theta \le 74°$, $0 \le h \le 5$, $0 \le k \le 22$, $-8 \le l \le 8$). No absorption corrections. 770 observed reflections [$I \ge 2{\cdot}5\sigma(I)$], no significant variation in intensity of standard reflection $18\bar{4}$. Direct methods [*MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980)]. An E map calculated from the set of phases with the highest figure of merit revealed the whole asymmetric unit, *i.e.* half the molecule, due to the presence of a mirror plane containing the central N(1) atom of the tricyclic system and two axial carbon atoms [C(2), C(8)] in special positions (x, $\frac{1}{4}$, z; $\bar{x}$, $\frac{3}{4}$, $\bar{z}$). Full-matrix refinement on F by *SHELX* (Sheldrick, 1976). Anisotropic temperature factors for the C and N atoms, isotropic for H. H positions from a difference map. $w = 1/\sigma_F^2$. $R = 0{\cdot}046$, $S = 0{\cdot}41$; $(\Delta/\sigma)_{max} = 0{\cdot}186$ [for x of C(9)] [0·673 for z of H(91)]. $-0{\cdot}28 \le \Delta\rho \le 0{\cdot}16$ e Å^{-3} in final difference map. Scattering factors as in *SHELX*.

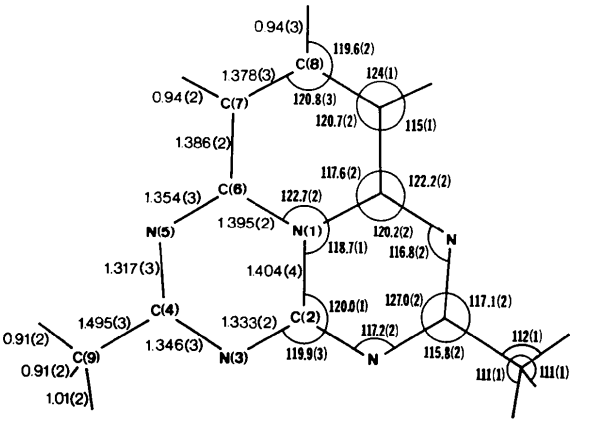

Fig. 1. Molecular structure of 2,5-dimethyl-1,3,4,6-tetraazacycl-[3.3.3]azine: atomic numbering, bond distances (Å) and angles (°) (e.s.d.'s in parentheses).

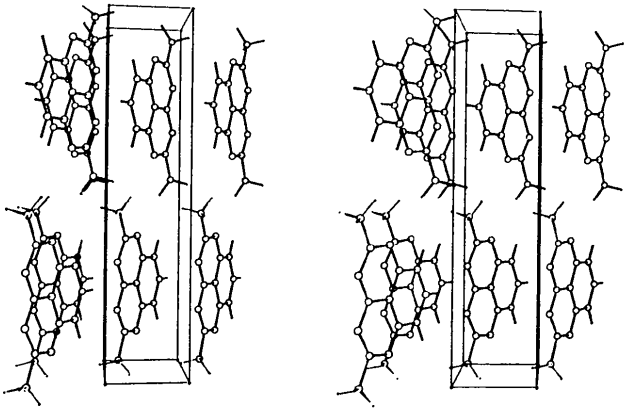

Fig. 2. Molecular packing of 2,5-dimethyl-1,3,4,6-tetraazacycl-[3.3.3]azine. Hydrogens are also represented.

Discussion. Table 1 gives final atomic parameters, Fig. 1 bond distances and angles.*

The bond pattern presents a strong aromatic character at the periphery of the molecule, while the bond lengths involving the central N atom are somewhat longer. This is in complete agreement with the structure of the unmethylated compound (Lindqvist, Ljungström, Andréasson & Ceder, 1978), indicating that the methyl group has no influence on the geometrical properties.

The molecule is planar (Fig. 2). The deviation of N(1) from the mean plane including the non-H atoms of the molecule is 0·009 (20) Å (0·033 Å for the unmethylated compound, σ not given). The intermolecular contacts between packed molecules are of the same order of magnitude (namely 3·4 Å) as in the unmethylated compound; this stacking distance is quite comparable to that in graphite (3·35 Å) (Nelson & Riley, 1945).

J-PB and GJA are respectively grateful to the FNRS, Belgium, and SERC, England, for financial support.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39713 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

LINDQVIST, O., LJUNGSTRÖM, E., ANDRÉASSON, E. & CEDER, O. (1978). *Acta Cryst.* B**34**, 1667–1670.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J. P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-La-Neuve, Belgium.

NELSON, J. B. & RILEY, D. P. (1945). *Proc. Phys. Soc. London*, **57**, 477.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal-structure determination. Univ. of Cambridge, England.