Acta Cryst. (1991). C**47**, 2697–2698

Structure of Dimetridazole at 293 K

BY HENDRIK L. DE BONDT,* N. M. BLATON, O. M. PEETERS AND C. J. DE RANTER†

Laboratorium voor Analytische Chemie en Medicinale Fysicochemie, Instituut voor Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium

(*Received* 20 *January* 1991; *accepted* 21 *June* 1991)

Abstract. 1,2-Dimethyl-5-nitroimidazole, $C_5H_7N_3O_2$, $M_r = 141{\cdot}13$, orthorhombic, *Pmcn*, $a = 6{\cdot}5847\ (8)$, $b = 9{\cdot}1511\ (10)$, $c = 10{\cdot}9548\ (8)$ Å, $V = 660{\cdot}1\ (1)$ Å^3, $Z = 4$, $D_m = 1{\cdot}41$, $D_x = 1{\cdot}420$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}105$ mm^{-1}, $F(000) = 296$, $T = 293$ K, final $R = 0{\cdot}042$ for 601 unique observed [$F \geq 4\sigma(F)$] reflections. The non-H atoms of the molecule lie in the crystallographic *m*-mirror plane. Two different hydrogen bonds are present.

Experimental. Colourless crystals obtained from saturated Me_2SO solution and used for density measurements by flotation in *n*-heptane/CCl_4. Prismatic crystal, with approximate dimensions 0·7 × 0·3 × 0·3 mm, was mounted on Stoe STADI-4 four-circle diffractometer with graphite-monochromated Mo $K\alpha$ (reflection 200) radiation. Crystal decay reduced by surrounding the crystal with a glue layer. Space group, *Pmcn*, determined from observed symmetry and systematic absences *h*0*l* ($l = 2n$), *hk*0 ($h + k = 2n$). Non-centrosymmetric equivalent positions of this space group are $\frac{1}{2} - x, y, z$; $x, \frac{1}{2} - y, \frac{1}{2} + z$; and $\frac{1}{2} + x, \frac{1}{2} + y, \frac{1}{2} - z$. *Pnma* is standard. Unit-cell dimensions obtained by least-squares refinement of accurately determined 2θ values of 26 reflections with $18 \leq 2\theta \leq 25°$. These values were calculated as the differences between the ω values corresponding to the measured positive and negative 2θ's. X-ray intensities were collected in the $\omega/2\theta$ scan mode up to max. $(\sin\theta)/\lambda = 0{\cdot}65$ Å^{-1} and for $0 \leq h \leq 9$, $-12 \leq k \leq 12$, $-14 \leq l \leq 0$. Friedel equivalents were not collected. For all reflections, peak profiles of 96 steps were stored. Intensities of three standard reflections (400, 04$\bar{1}$, 11$\bar{6}$), monitored every 2 h of radiation, showed an average decrease in intensity of 4·2% in 38 h radiation. The reflection intensities were rescaled using a cubic regression curve. 1691 reflections were measured. Symmetry-related reflections were averaged to give 824 unique reflections of which 601 were considered observed with $F \geq 4\sigma(F)$; $R_{int} = 0{\cdot}020$. Data reduction with the peak-profile analysis program *DREAM* (Blessing, 1987). Lorentz and polarization corrections were applied. Structure factors were calculated with scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) and contracted hydrogen form factors from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were performed for all non-H atoms (Ibers & Hamilton, 1964). The phase problem was successfully solved by direct methods using *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) which revealed the positions of all non-H atoms. Full-matrix least-squares refinements were performed on *F*, first isotropically and next anisotropically. All H atoms were located in a difference map. All methyl H atoms were given a temperature factor, B_{iso}, of 8·0 Å^2. For H4, B_{iso} was fixed at 4·0 Å^2. All H-atom positions were refined. An isotropic extinction parameter, $g = 4{\cdot}0\ (3) \times 10^{-5}$, defined as $F_{c,\ \text{corrected}} = F_{c,\ \text{uncorrected}}/(1 + g\mathrm{L}\,\mathrm{p}F^2_{c,\ \text{uncorrected}})$, was also refined. $w = 4F^2/[\sigma^2(F^2) + (0{\cdot}02F^2)^2]$. Final $R = 0{\cdot}042$, $wR = 0{\cdot}059$, with $S = 2{\cdot}59$. Largest parameter shift/e.s.d. = 0·03. The residual electron density varies between −0·15 and 0·21 e Å^{-3}. The number of reflections per refined parameter 601/74 = 8·1. All calculations were performed on a Digital PDP-11/73 and MicroVAX 2000 microcomputer using *SDP/VAX* (B. A. Frenz & Associates Inc., 1985) and *PARST* (Nardelli, 1983). An *ORTEP* (Johnson, 1976) view 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.‡ Fig. 2 shows the stacking along the *a* axis and the electrostatic packing forces N3($\delta-$)···N51′($\delta+$).

* Research Assistant of the National Fund for Scientific Research (Belgium).

† To whom correspondence should be addressed.

‡ Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54400 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/91/122697-02$03.00

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

E.s.d.'s on the least significant digit are given in parentheses. Starred temperature factors are isotropic and were fixed.

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

	x	y	z	U_{eq}
N1	0·250	−0·0479 (2)	0·8703 (1)	3·77 (4)
C2	0·250	−0·1670 (2)	0·9436 (2)	4·16 (5)
N3	0·250	−0·1311 (2)	1·0613 (1)	4·60 (4)
C4	0·250	0·0167 (2)	1·0647 (2)	4·18 (5)
C5	0·250	0·0699 (2)	0·9491 (2)	3·89 (4)
C11	0·250	−0·0517 (3)	0·7368 (2)	6·07 (6)
C21	0·250	−0·3196 (3)	0·8978 (2)	6·45 (6)
N51	0·250	0·2177 (2)	0·9127 (2)	5·13 (5)
O52	0·250	0·3099 (2)	0·9938 (2)	7·82 (5)
O53	0·250	0·2488 (2)	0·8045 (2)	8·44 (6)
H11*A*	0·250	−0·143 (4)	0·708 (3)	10·1*
H11*B*	0·150 (3)	0·004 (2)	0·705 (1)	10·1*
H21*A*	0·250	−0·386 (4)	0·961 (3)	10·1*
H21*B*	0·337 (3)	−0·331 (2)	0·841 (2)	10·1*
H4	0·250	0·067 (2)	1·143 (2)	5·1*

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

N1—C2	1·353 (2)	N51—O52	1·225 (2)
C2—N3	1·330 (2)	N51—O53	1·219 (2)
N3—C4	1·353 (3)	C11—H11*A*	0·89 (4)
C4—C5	1·358 (3)	C11—H11*B*	0·90 (2)
C5—N1	1·381 (2)	C21—H21*A*	0·92 (3)
N1—C11	1·463 (3)	C21—H21*B*	0·85 (2)
C2—C21	1·485 (3)	C4—H4	0·98 (2)
C5—N51	1·410 (3)		

C2—N1—C5	104·9 (1)	C4—C5—N51	127·4 (2)
C2—N1—C11	125·0 (1)	N1—C11—H11*A*	112 (2)
C5—N1—C11	130·0 (1)	N1—C11—H11*B*	112 (1)
N1—C2—N3	112·1 (1)	H11*A*—C11—H11*B*	113 (2)
N1—C2—C21	123·8 (2)	C2—C21—H21*A*	112 (2)
N3—C2—C21	124·0 (2)	C2—C21—H21*B*	111 (1)
C2—N3—C4	105·9 (2)	H21*A*—C21—H21*B*	118 (2)
N3—C4—C5	109·4 (1)	C5—N51—O52	117·1 (2)
N3—C4—H4	120 (1)	C5—N51—O53	119·9 (2)
C5—C4—H4	131 (1)	O52—N51—O53	123·0 (1)
N1—C5—C4	107·6 (2)		
N1—C5—N51	124·9 (2)	N1—C2—C21—H21*B*	46 (1)
		C2—N1—C11—H11*B*	128 (1)

Inspection of non-bonding distances shorter than the sum of the van der Waals radii reveals the hydrogen bonds summarized in Table 3.

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

The authors are indebted to J.-P. Van Cuyck for his valuable help in preparing the pictures and to the Merck, Sharp & Dohme Research Laboratories, Rahway, New Jersey, USA, for providing the title compound.

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

E.s.d.'s on the least significant digit are given in parentheses. H-atom positions are normalized to C—H = 1·06 and 1·08 Å.*

D—H···*A*	*D*—H	*D*···*A*	H···*A*	*D*—H···*A*
C11—H11*A*···N3^{i}	1·06	3·482 (3)	2·459 (2)	161·8 (1)
C4—H4···O53^{ii}	1·08	3·392 (3)	2·341 (2)	163·7 (1)

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

* Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987).

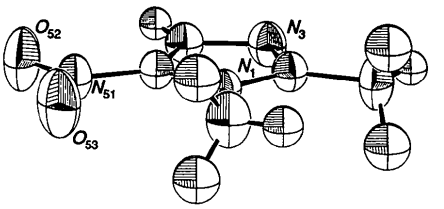

Fig. 1. *ORTEP* (Johnson, 1965) plot of the title compound with atomic numbering scheme. The ellipsoids enclose 50% probability.

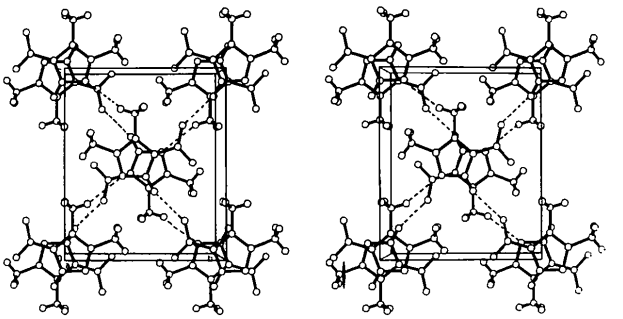

Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) plot of the crystal showing the packing along **a**. Dashed lines indicate hydrogen bonds.

References

ALLEN, F. H., KENNARD, O., WATSON, D. G., BRAMMER, L., ORPEN, A. G. & TAYLOR, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

B. A. FRENZ & ASSOCIATES, INC. (1985). *SDP-Plus Structure Determination Package*, College Station, Texas, USA.

BLESSING, R. H. (1987). *Crystallogr. Rev.* **1**, 3–58.

EDWARDS, D. I. (1981). *Prog. Med. Chem.* **18**, 87–116.

IBERS, J. A. & HAMILTON, W. C. (1964). *Acta Cryst.* **17**, 781–782.

JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., FISKE, S., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MCMULLAN, R. K., EPSTEIN, J., RUBLE, J. R. & CRAVEN, B. M. (1979). *Acta Cryst.* B**35**, 688–691.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175—3187.