of 0·06 Å^2 for all H atoms; all non-hydrogen atoms anisotropic. Form I: 75 parameters refined, $\Delta_{max}/\sigma = 0{\cdot}007$ in final refinement cycle; difference-map excursions (max) 0·279 and (min) −0·381 Å^{-3}; $R_w = 0{\cdot}0471$, $R = 0{\cdot}0443$, $w^{-1} = \sigma^2(F) + 0{\cdot}001F^2$. Form II: 76 parameters refined, $\Delta_{max}/\sigma = 0$ in the final refinement cycle; difference-map excursions (max) 0·429 and (min) −0·538 Å^{-3}; $R_w = 0{\cdot}0857$, $R = 0{\cdot}0812$, $w^{-1} = \sigma^2(F) + 0{\cdot}001\,F^2$. Scattering factors of *SHELXTL* used.*

Discussion. Final atomic coordinates for forms I and II are presented in Table 1, with derived bond lengths and angles indicated on Fig. 1(*a*) and (*b*). Table 2 contains torsion angles (Allen & Rogers, 1969) for the nonhydrogen atoms of both forms.

The results of the structure determination indicated that the two polymorphs arise from a conformational difference along the ethylenediamine chain. This difference corresponds to a rotation about the N(1)—C(2) and C(2)—C(3) bonds. In both forms, the O···N intermolecular contact distances varying from 2·84 (1) to 2·93 (1) Å can be interpreted as O···H—N hydrogen bonds between neighboring molecules. No evidence for any intramolecular hydrogen bonding could be determined in either form.* Since the two polymorphs are related by simple rotations, it is interesting to note that in 13 different preparations of this material examined by diffraction, these are the only two polymorphs that have been observed.

We would like to thank G. L. Gaines Jr and O. H. LeBlanc Jr for supplying us with the N-(2-ammonioethyl)carbamate samples and for drawing the problem to our attention. We would also like to thank J. S. Kasper for discussions and advice with regard to this work.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and packing diagrams have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38783 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* Packing diagrams for each of the crystal structures have been deposited. See deposition footnote.

References

Allen, F. H. & Rogers, D. (1969). *Acta Cryst.* **B25**, 1326–1330.
Frahn, J. L. & Mills, J. A. (1964). *Aust. J. Chem.* **17**, 256–273.
Frank, A. W. (1982). *Appl. Spectrosc.* **36**, 282–285.
Jensen, A. & Christensen, R. (1955). *Acta Chem. Scand.* **9**, 486–492.
Katchalsky, E., Berliner, C. & Berger, A. (1951). *J. Am. Chem. Soc.* **73**, 1829–1831.
Schering, E. (1900). German Patent No. 123,138.
Schering, E. (1901). *Chem. Zentralbl.* **72**II, 519.
Sheldrick, G. M. (1981). *SHELXTL User's Manual,* Revision 3. Nicolet XRD Corporation, Fremont, CA, USA.

Acta Cryst. (1983). C**39**, 1686–1688

1,3-Dimethyl-5-(3-methyl-1-benzimidazolio)-4-barbiturate Monohydrate, $C_{14}H_{14}N_4O_3.H_2O$

By L. Prasad

National Research Council Canada, Ottawa, Ontario, Canada K1A 0R6

and Max R. Taylor*

School of Physical Sciences, Flinders University of South Australia, Bedford Park, SA 5042, Australia

(*Received* 21 *December* 1982; *accepted* 29 *July* 1983)

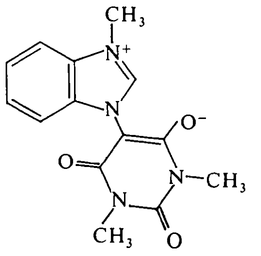
Abstract. $M_r = 304{\cdot}3$, $P\bar{1}$, $a = 7{\cdot}926$ (2), $b = 8{\cdot}626$ (2), $c = 11{\cdot}882$ (4) Å, $\alpha = 79{\cdot}00$ (4), $\beta = 100{\cdot}58$ (4), $\gamma = 62{\cdot}88$ (4)°, $U = 674{\cdot}87$ Å^3, $Z = 2$, $D_x = 1{\cdot}497$, $D_m = 1{\cdot}45$ (2) Mg m^{-3}, Ni-filtered Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}952$ mm^{-1}, $T = 298$ K, $F(000) = 320$, $R = 0{\cdot}045$ for 1276 data. There are marked deviations from hexagonal symmetry in the benzene ring of the benzimidazole fragment. The benzimidazole and barbiturate rings are both planar [interplanar angle 121·6 (3)°]. [The conventional setting of the unit cell ($a = 8{\cdot}626$, $b = 11{\cdot}882$, $c = 7{\cdot}926$ Å, $\alpha = 100{\cdot}58$, $\beta = 117{\cdot}12$, $\gamma = 101{\cdot}00$°) may be obtained from the above unit-cell parameters by the transformation: $010/00\bar{1}/\bar{1}00$.]

---

* Author to whom correspondence should be addressed.

0108-2701/83/121686-03$01.50

Introduction. The title compound results from the reaction of *o*-dimethylaminoaniline with alloxan (Clark-Lewis, Edgar, Shannon & Thompson, 1964; Clark-Lewis, Moody & Thompson, 1970). The crystal structure of the compound was determined in order to confirm that it had the molecular structure (I) previously assigned to it on the basis of synthetic studies.



(I)

Experimental. D_m measured by flotation in a mixture of toluene and CCl_4. Intensity data and unit-cell dimensions measured on a Supper automatic two-circle equi-inclination diffractometer using Ni-filtered Cu $K\alpha$ radiation. For each reflection the optimum scan speed [to make the ratio $\sigma(I)/I$ for all reflections approximately the same] was estimated from an initial fast scan. Minimum and maximum scan ranges were 1·6° and 4° in θ and scan speeds ranged from 0·025 to 0·33° s^{-1}. One crystal [0·55 (2) × 0·11 (1) × 0·08 (1) mm] was used to collect data from layers $k = 0$–5, and another [0·62 (2) × 0·24 (1) × 0·08 (1) mm] to collect layers $l = 0$–6; $2\theta_{\max} = 120°$. Absorption corrections calculated and applied (Coppens, Leiserowitz & Rabinovich, 1965). Data scaled by the method of Rae (1965) to give intensities for a set of 1917 independent reflections, of which 1276 had $I > 3\sigma(I)$, where $\sigma(I)$ is based on counting statistics. Standard deviations for observed reflections were calculated from a combination of counting statistics and mean deviations for those intensities measured more than once, and counting statistics alone for the remainder. Structure solved by application of *QTAN* (Langs & DeTitta, 1975) to 200 reflections with the largest E values. The resulting E map revealed the positions of 20 of the 22 non-hydrogen atoms and the positions of the remaining atoms were located in a Fourier map. After initial least-squares refinement the positions of all H atoms were located in a difference Fourier map. The structure was refined by block-diagonal least-squares calculations, minimizing $\sum w(\Delta F)^2$ where $w = \sigma^{-2}(|F_o|^2)$, using the 1276 'observed' data. Scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974); the curve for H was that calculated by Stewart, Davidson & Simpson (1965). The parameters refined included a scale factor, an extinction parameter (Larson, 1970), positional and anisotropic temperature factor coefficients for the non-hydrogen atoms and positional and isotropic thermal parameters for the H atoms. At the end of the refinement $wR = 0·048$, $S = 1·48$, and the extinction parameter $g = 1·48 \times 10^{-6}$ (1) for 1267 reflections; $(\Delta/\sigma)_{\max} = 0·2$, $(\Delta\rho)_{\max} = 0·3$ e Å^{-3}. The structure was solved and refined using the NRC system of programs for the PDP-11 and PDP8-E computers (Larson & Gabe, 1978).

Discussion. Final atomic parameters are listed in Table 1.* Atom labelling, bond lengths and angles are shown in Fig. 1.

The crystal structure analysis confirms the proposed structure (I). The compound exists as discrete molecules in the crystal held together by van der Waals forces and hydrogen bonds between the atoms O(1) and O(2) of different molecules and the water molecule (Fig. 1). There are no hydrogen bonds to O(3).

Table 1. *Atomic coordinates and isotropic thermal parameters*

B_{eq} is the arithmetic mean of the principal axes of the thermal ellipsoids.

	x	y	z	B_{eq}(Å^2)
N(1)	0·7386 (3)	0·7107 (3)	0·3716 (2)	2·0
N(2)	0·8164 (3)	0·5169 (3)	0·2688 (2)	2·0
N(3)	0·7728 (3)	0·4626 (3)	0·0313 (2)	1·9
N(4)	1·0736 (3)	0·2170 (3)	0·1029 (2)	1·9
C(1)	0·7796 (4)	0·6840 (4)	0·2721 (3)	2·0
C(2)	0·7971 (4)	0·4323 (4)	0·3746 (3)	1·7
C(3)	0·7533 (4)	0·5526 (4)	0·4409 (3)	1·9
C(4)	0·7355 (4)	0·5075 (4)	0·5536 (3)	2·3
C(5)	0·7588 (5)	0·3367 (4)	0·5970 (3)	2·7
C(6)	0·7929 (5)	0·2192 (4)	0·5258 (3)	2·6
C(7)	0·8142 (4)	0·2614 (4)	0·4138 (3)	2·4
C(8)	0·6867 (5)	0·8780 (4)	0·4042 (3)	2·6
C(9)	0·8614 (4)	0·4448 (4)	0·1738 (3)	1·8
C(10)	0·7225 (4)	0·5421 (4)	0·0580 (3)	1·8
C(11)	0·9430 (4)	0·2997 (4)	0·0114 (3)	2·0
C(12)	1·0405 (4)	0·2817 (4)	0·2004 (3)	2·1
C(13)	0·6338 (5)	0·5534 (4)	0·1537 (3)	2·8
C(14)	1·2561 (5)	0·0407 (4)	0·1235 (3)	3·2
O(1)	0·5646 (3)	0·6892 (3)	0·0328 (2)	2·8
O(2)	0·9753 (3)	0·2351 (3)	0·0929 (2)	2·7
O(3)	1·1675 (3)	0·1945 (3)	0·3011 (2)	2·5
O(W)	0·2223 (3)	0·9387 (3)	0·8373 (2)	3·6
H(1)	0·786 (4)	0·781 (4)	0·208 (2)	3·7 (7)
H(4)	0·715 (4)	0·595 (4)	0·602 (2)	4·1 (7)
H(5)	0·741 (5)	0·295 (4)	0·672 (3)	6·0 (9)
H(6)	0·801 (4)	0·110 (4)	0·561 (3)	5·1 (8)
H(7)	0·853 (4)	0·172 (4)	0·364 (2)	3·7 (7)
H(1)C(8)	0·551 (4)	0·922 (4)	0·398 (3)	4·9 (8)
H(2)C(8)	0·718 (5)	0·954 (4)	0·353 (2)	4·5 (7)
H(3)C(8)	0·757 (4)	0·849 (4)	0·495 (2)	5·1 (8)
H(1)C(13)	0·704 (8)	0·529 (7)	0·211 (4)	15·3 (16)
H(2)C(13)	0·551 (6)	0·676 (6)	0·166 (3)	10·3 (12)
H(3)C(13)	0·548 (7)	0·517 (6)	0·162 (4)	12·2 (14)
H(1)C(14)	1·323 (5)	0·067 (4)	0·084 (3)	6·3 (8)
H(2)C(14)	1·320 (6)	0·005 (6)	0·213 (3)	10·0 (11)
H(3)C(14)	1·222 (6)	0·041 (5)	0·096 (3)	8·8 (11)
HW(1)	0·119 (6)	1·041 (5)	0·869 (3)	9·4 (11)
HW(2)	0·344 (7)	0·839 (6)	0·921 (4)	11·5 (13)

---

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38746 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

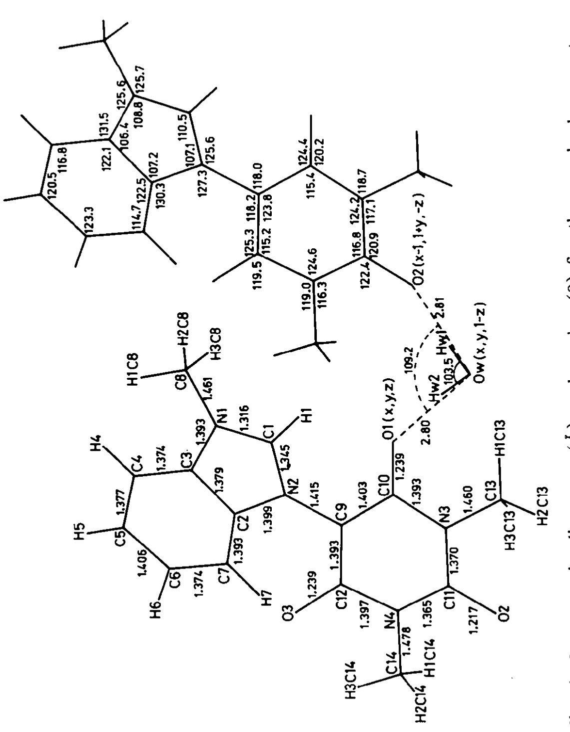


Fig. 1. Interatomic distances (Å) and angles (°) for the non-hydrogen atoms. Standard deviations are 0·004 Å in distances and 0·3° in angles.

The bond lengths and angles found in this structure are similar to those found in the structure of 5-(6-bromo-3-ethyl-2-methylbenzimidazolio)-4-barbiturate hydrate (Matthews, 1965). The N(2)—C(9) length of 1·415 (4) Å indicates considerable double-bond character and suggests significant conjugation between the benzimidazole and barbiturate ring systems. The bond lengths of the imidazole ring in the region of N(1) are similar to those found in a number of structures where the benzimidazole moiety is present (Quick & Williams, 1976, and references therein) but where it does not have a substituent bonded to N(1). This suggests that the majority of the positive charge is localized on N(1), and that the canonical form (I) is a principal contributor to the resonance hybrid so far as the benzimidazole moiety is concerned. The bond lengths C(12)—O(3) and C(10)—O(1) are equal and are significantly longer than C(11)—O(2) indicating that (I) and the canonical structure with the negative charge on O(3) contribute equally to the resonance hybrid so far as the barbiturate moiety is concerned. The benzene ring shows a large distortion from a regular hexagonal shape, being elongated along the C(4)—C(7) line in much the same manner as occurs in the bromo derivative referred to above. The benzimidazole ring and the barbiturate ring are both planar within experimental error, with the dihedral angle between the two planes being 121·6 (3)°.

We wish to thank Professor J. W. Clark-Lewis for supplying the crystals of the compound, Dr Y. Le Page of the National Research Council, Canada, for useful discussions, and Dr G. T. DeTitta for assistance in applying *QTAN* to this problem. One of the authors (LP) would like to thank the Natural Sciences and Engineering Research Council Canada for the award of a Visiting Fellowship.

References

Clark-Lewis, J. W., Edgar, J. A., Shannon, J. S. & Thompson, M. J. (1964). *Aust. J. Chem.* **17**, 877–893.

Clark-Lewis, J. W., Moody, K. & Thompson, M. J. (1970). *Aust. J. Chem.* **23**, 1249–1273.

Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–98. Birmingham: Kynoch Press.

Langs, D. A. & DeTitta, G. T. (1975). *Acta Cryst.* A**31**, *S*16.

Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, pp. 291–294. Copenhagen: Munksgaard.

Larson, A. C. & Gabe, E. J. (1978). *Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Gassi, p. 81. Delft Univ. Press.

Matthews, B. W. (1965). *Acta Cryst.* **18**, 151–157.

Quick, A. & Williams, D. J. (1976). *Can. J. Chem.* **54**, 2482–2487.

Rae, A. D. (1965). *Acta Cryst.* **19**, 683–684.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1983). C**39**, 1688–1690

Structure of 3-Deazauracil (4-Hydroxy-2-pyridone), $C_5H_5NO_2$

By J. N. Low and C. C. Wilson

*Carnegie Laboratory of Physics, University of Dundee, Dundee DD*1 4*HN, Scotland*

(*Received* 24 *May* 1983; *accepted* 9 *August* 1983)

Abstract. $M_r = 111{\cdot}1$, $P2_12_12_1$, $a = 8{\cdot}638$ (6), $b = 5{\cdot}279$ (5), $c = 11{\cdot}220$ (8) Å, $U = 511{\cdot}61$ Å^3, $Z = 4$, $D_x = 1{\cdot}442$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}072$ mm^{-1}, $F(000) = 232{\cdot}0$, $T = 293$ K, $R = 0{\cdot}065$ for 1133 observed reflections (Friedel pairs not merged). The molecule exists in the 4-enol form. There is an intermolecular N—H···O hydrogen bond.

Introduction. In order to make electron spin resonance studies of free radicals produced by ionizing radiations

0108-2701/83/121688-03$01.50