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Structure of *N,N'*-Diacetylparabanic Acid*

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Abstract. $C_7H_6N_2O_5$, $M_r = 198{\cdot}14$, orthorhombic, *Pnma*, $a = 8{\cdot}130\,(3)$, $b = 19{\cdot}120\,(9)$, $c = 5{\cdot}243\,(2)$ Å, $V = 815{\cdot}0$ Å^3, $Z = 4$, D_m (displacement of benzene) $= 1{\cdot}59$, $D_x = 1{\cdot}614$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}131$ mm^{-1}, $F(000) = 408$, room temperature, final $R = 0{\cdot}067$ for 304 unique observed reflections; crystal selected from synthetic material. The structure contains almost planar molecules which lie perpendicular to crystallographic mirror planes and are closely packed in layers parallel to them.

Introduction. In the course of study of the title compound it was found that the compound underwent hydrolysis in two stages, first to *N*-acetylparabanic acid and then by ring opening to *N*-acetyloxaluric acid. The crystal structure determination was undertaken in an attempt to discover why this should be so.

Experimental. The compound was synthesized by acetylation of the disodium salt of parabanic acid (Murray, 1963) with acetyl chloride and recrystallization from a chloroform/acetone mixture provided crystals suitable for further study (m.p. 433–435 K).

Colourless triangular crystal fragment of 0·15 mm side and 0·08 mm thickness; Nicolet *P*3 diffractometer with Mo$K\alpha$ radiation and $\omega/2\theta$ scan method with $\Delta 2\theta$ 2·40–2·66° and scan rate 5·33 ($I < 150$) to 58·6° 2θ min^{-1} ($I > 2500$); cell refined from 071, 002 and 311 reflections with $15 < 2\theta < 18°$; no correction for absorption or extinction; $(\sin\theta)/\lambda \le 0{\cdot}4813$ Å^{-1}; $0 \le h \le 7$, $0 \le k \le 15$, $0 \le l \le 5$; no significant variation in the intensities of the standard reflections 071 and 311; 342 unique reflections including 38 with $I < 3\sigma(I)$ which were excluded from further calculations. Structure solved in the space group $Pn2_1a$ using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) but results clearly compatible with the space group *Pnma*, consistent with *E*-value statistics. Refinement of all non-H atoms vibrating anisotropically in *Pnma* minimizing $\sum w(|F_o| - |F_c|)^2$ with $w = \{1 + [(|F_o| - 16)/9]^2\}^{-1/2}$ gave $R = 0{\cdot}093$, $wR = 0{\cdot}113$. Further refinement including H from a difference map vibrating isotropically gave final $R = 0{\cdot}067$, $wR = 0{\cdot}074$ and $S = 0{\cdot}844$ for 80 refined

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* Parabanic acid is imidazolidinetrione.

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parameters. At this stage the maximum shift/e.s.d. was 0·47 and 0·9 for non-H and H respectively and in the difference map $|\Delta\rho|$ was $<0{\cdot}25$ e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on the Honeywell 66/80 of the Computing Centre of the University of Aberdeen using for the most part the *NRC* program (Ahmed, Hall, Pippy & Huber, 1966).

Discussion. Atomic coordinates are given in Table 1.* The molecular structure with bond lengths and angles is shown in Fig. 1, and the packing of the molecules in Fig. 2.

The bond lengths and angles are in good agreement with the results obtained for similar molecules, *e.g.* acetamide (Hamilton, 1966) and parabanic acid (Craven & McMullan, 1979). Least well determined are the values involving H; C(4)—H distances in the range 0·91 (8)–1·07 (6), mean 0·99 Å; C(3)—C(4)—H angles in the range 110 (3)–118 (4), mean 115°; H—C(4)—H′ angles in the range 89 (6)–114 (6), mean 102°. The large range of these last in particular is ascribed to limitations in the intensity data rather than to an error in the choice of space group or the effect of disorder.

Whereas C—O distances fall in the relatively narrow range 1·178 (10)–1·207 (7) Å the distribution of C—N [1·381 (7)–1·454 (9) Å] is rather broader. The longest of these is the N(1)—C(3) (acetyl) distance. Since the work of Hamilton (1966) implies a relationship between the length and reactivity of the C—N bond in acetamide this result agrees with the observation that the first stage in the hydrolysis of *N,N′*-diacetylparabanic acid is by the loss of an acetyl group to yield *N*-acetylparabanic acid. The present results do not, however, give any clear indication of the route to be followed in the second stage of hydrolysis. It is not unlikely that the relative C—N bond lengths are significantly different in the monoacetyl compared with the diacetyl derivative. Therefore, it is our intention to continue the present study by investigating the structure of *N*-acetylparabanic acid in an attempt to clarify this matter.

Ignoring H the molecule is close to planar. The maximum deviation from planarity within the five-membered heterocyclic ring is C(1) with $\Delta = -0{\cdot}0082$ Å when $\sum\Delta^2 = 0{\cdot}00017$ Å^2 for the ring. Of the atoms attached to the ring, O(1) and O(2) with $\Delta = -0{\cdot}0102$ and $-0{\cdot}0171$ Å respectively do not deviate significantly from a planar arrangement. The atoms of the acetyl groups, O(3), C(3) and C(4) with $\Delta = 0{\cdot}0588$, $0{\cdot}1106$ and $0{\cdot}3254$ Å respectively, show some departure from a completely planar arrangement. The lack of planarity arises from two effects. In part there is evidence of a small amount of pyramidal coordination at N(1) [this atom is 0·0293 Å out of the C(1)—C(2)—C(3) plane]. This is consistent with

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* ($\times 10^2$ Å^2)

$B_{eq} = \frac{4}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33})$, where the correction for anisotropic thermal vibration is of the form

$$\exp[-(h^2B_{11} + k^2B_{22} + l^2B_{33} + hkB_{12} + hkB_{13} + klB_{23})].$$

	x	y	z	B_{eq}
N(1)	1955 (5)	3098 (3)	5404 (8)	353
C(1)	1374 (10)	$\frac{1}{4}$	6756 (15)	380
C(2)	2891 (7)	2892 (3)	3339 (10)	397
C(3)	1497 (7)	3809 (4)	6083 (12)	478
C(4)	1990 (10)	4354 (4)	4257 (14)	597
O(1)	551 (8)	$\frac{1}{4}$	8606 (11)	509
O(2)	3599 (5)	3252 (2)	1799 (8)	521
O(3)	724 (7)	3897 (3)	7988 (9)	678

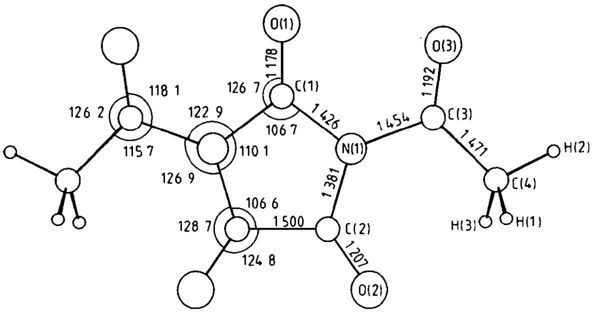

Fig. 1. Atomic arrangement of the molecule showing atom designations, bond lengths (e.s.d.'s 0·007 to 0·010 Å) and bond angles (e.s.d.'s approximately 0·5°).

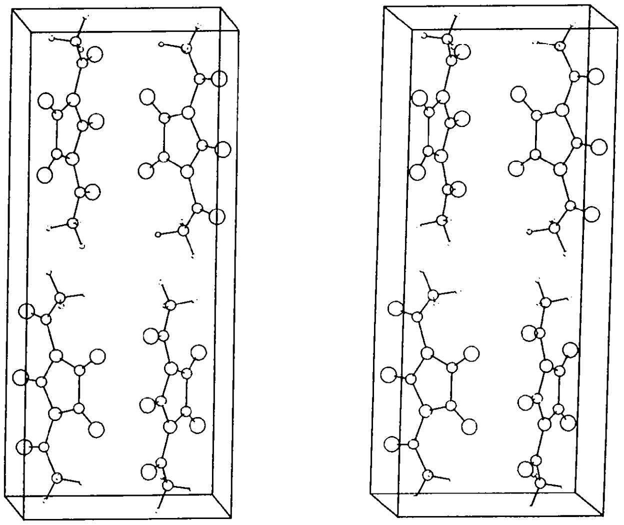

Fig. 2. Stereoscopic view of the molecular packing along **c**.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and molecular geometry data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44668 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

reduced conjugation with the acetyl group as indicated by the relatively long N(1)–C(3) bond. Further, there is a rotation of the acetyl group about the N(1)–C(3) bond [the C(1)–C(2)–C(3) and N(1)–O(3)–C(4) planes lie at an angle of about 8°].

As shown in Fig. 2 the almost planar molecules lie perpendicular to crystallographic mirror planes passing through C(1), O(1) and the midpoint of the C(2)–C(2') bond and are closely packed in a herring-bone pattern to form layers parallel to these planes. The closeness of the intra-layer packing is shown by the presence of relatively short intermolecular contacts [H(3)–O(3'), O(1)–C(2'), H(1)–O(3'), O(2)–N(1') and O(2)–C(3') of 2·68 (8), 2·794 (8), 2·80 (6), 2·977 (6) and 2·994 (8) Å respectively] and is reflected in the relatively high density of the material. The separation between the layers of molecules is determined primarily by intermolecular H–H' contacts ranging from 2·49 (9) to 2·61 (10) Å.

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Structure of Antischistosome Compounds. III. (3-Cyanopropyl)triphenylphosphonium Bromide

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Abstract. $C_{22}H_{21}NP^+.Br^-$, $M_r = 410·3$, monoclinic, $P2_1/c$, $a = 11·284$ (1), $b = 10·236$ (1), $c = 17·392$ (2) Å, $\beta = 105·33$ (45)°, $V = 1937·37$ Å^3, $Z = 4$, $D_x = 1·407$ g cm^{-3}, graphite-monochromatized Cu $K\alpha$ radiation, $\lambda = 1·5418$ Å, $\mu = 37·1$ cm^{-1}, $F(000) = 840$, $T = 292$ K. Final $R = 0·044$ for 3335 observed reflections. Structure solved by direct methods. The cyanopropyl moiety is in an extended conformation. However, the C–C–C–C torsion angle in this group is *gauche*$^+$, which points the cyano group in a direction similar to the direction of the N–Br1 vector observed in the (2-aminoethyl)triphenylphosphonium bromide hydrobromide structure and the C–Br vector in the (3-bromopropyl)triphenylphosphonium bromide structure. This suggests a basis for the biological activities of these three compounds.

Introduction. This communication is the third structure to be reported in a series of 17 triphenylphosphonium compounds which exhibit varying effects on the cholinergic nervous system of *Schistosoma mansoni* (McAllister, Dotson, Grim & Hillman, 1980). The differences in the biological effects of these 17 compounds cannot be explained solely on the basis of the differences in the chemical properties of the substituted moiety. Differences in the torsion angles in the substituted moieties may be the reason for the observed biological activities of these 17 triphenylphosphonium compounds. In order to ascertain if there is a structural basis for the observed biological activities of these triphenylphosphonium compounds, the crystal-structure determinations of the compounds in this series were initiated.

Experimental. Sample provided by Professor G. Hillman, synthesized as described in McAllister *et al.* (1980); colorless crystals (from ethanol), 0·20 × 0·30 × 0·70 mm; Enraf–Nonius CAD-4 diffractometer, cell parameter from 2θ values for 25 reflections from least-squares refinement with $11 \leq 2\theta \leq 64°$; ω–2θ scan, width $(1·00 + 0·15\tan\theta)°$, $[(\sin\theta)/\lambda]_{\max} = 0·6092$ Å^{-1}, $-14 \leq h \leq 0$, $-13 \leq k \leq 0$, $-22 \leq l \leq 22$; intensities of three standard reflections monitored every 7200 s showed a linear decline in intensity of 3·0%, correction applied; 3672 unique reflections measured, 3335 reflections with $I > 3\sigma(I)$; Lp correc-

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