$C_{21}H_{30}N_3O_3S^+.Br_3^-$

mouvement selon l'axe de symétrie du système et de direction perpendiculaire à ce dernier pour les atomes de brome terminaux.

Je remercie Marcel Pierrot, Directeur du Service de Cristallochimie de la Faculté des Sciences et Techniques de Saint-Jérôme, pour les moyens mis à ma disposition et André Baldy, Ingénieur de Recherche, pour son assistance technique.

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Structure of Pipemidic Acid

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Abstract. 8-Ethyl-5,8-dihydro-5-oxo-2-(1-piperazinyl)-pyrido[2,3-*d*]pyrimidine-6-carboxylic acid, $C_{14}H_{17}N_5O_3.3H_2O$, $M_r = 357.37$, triclinic, $P\bar{1}$, $a = 8.584$ (1), $b = 12.571$ (1), $c = 8.519$ (1) Å, $\alpha = 94.87$ (1), $\beta = 113.93$ (1), $\gamma = 88.61$ (1)°, $V = 837.2$ (2) Å^3, $Z = 2$, $D_x = 1.416$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 9.035$ cm^{-1}, $F(000) = 380$, room temperature, final $R = 0.053$ for 2410 independent reflections. The resolution of the structure showed that the apical N atom of piperazine has a quaternary character: the molecule is in a 'zwitterion' state. There is a three-dimensional network of hydrogen bonds in which all available proton donors are utilized.

Introduction. Pipemidic acid, which is used clinically in human therapy (de Lajudie, Horvath, Leriche & Pattle, 1974) belongs, like the related compounds nalidixic acid (Achari & Neidle, 1976), aminooxolinic acid (Czugler, Argay, Frank, Mészáros, Kutschabsky & Reck, 1976) and oxolinic acid (Cygler & Huber, 1985), to a group

of antibacterial agents that act by inhibiting DNA synthesis. With the aim of improving the understanding of the drug action of these compounds, it seems worthwhile to obtain the maximum information about them; for this reason we have undertaken the study of the crystal structure of the title compound.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* U_{eq} (Å² × 10⁴) *for the title compound*

$$U_{eq} = \tfrac{1}{3}\sum_i \sum_j U_{ij}a_i^* a_j^* a_i a_j \cos(a_i, a_j).$$

	x	y	z	U_{eq}
N(1)	1·0034 (3)	0·0473 (2)	0·1943 (3)	241 (8)
C(2)	0·8621 (3)	0·0959 (2)	0·0945 (3)	233 (9)
N(3)	0·7078 (3)	0·0494 (2)	−0·0022 (3)	293 (9)
C(4)	0·7001 (3)	−0·0549 (2)	0·0072 (4)	275 (10)
C(9)	0·8376 (3)	−0·1160 (2)	0·1065 (3)	237 (10)
C(10)	0·9894 (3)	−0·0584 (2)	0·1979 (3)	221 (9)
N(8)	1·1325 (3)	−0·1107 (2)	0·3010 (3)	252 (8)
C(7)	1·1236 (4)	−0·2183 (2)	0·3055 (3)	261 (10)
C(6)	0·9808 (3)	−0·2799 (2)	0·2194 (3)	256 (10)
C(5)	0·8212 (3)	−0·2299 (2)	0·1181 (4)	269 (10)
C(11)	0·9967 (4)	−0·3983 (2)	0·2370 (4)	279 (10)
O(12)	0·8844 (3)	−0·4586 (2)	0·1267 (3)	445 (9)
O(13)	1·1225 (3)	−0·4311 (2)	0·3613 (3)	350 (8)
N(14)	0·8743 (3)	0·2028 (2)	0·0856 (3)	262 (8)
C(15)	0·7230 (4)	0·2698 (2)	0·0278 (4)	299 (11)
C(16)	0·6938 (4)	0·3196 (2)	0·1821 (4)	327 (11)
N(17)	0·8507 (3)	0·3788 (2)	0·3049 (3)	312 (9)
C(18)	1·0071 (4)	0·3117 (2)	0·3573 (4)	303 (11)
C(19)	1·0286 (3)	0·2636 (2)	0·1988 (4)	279 (10)
O(20)	0·6807 (3)	−0·2756 (2)	0·0497 (3)	454 (9)
C(21)	1·2905 (3)	−0·0511 (2)	0·4123 (4)	292 (11)
C(22)	1·2780 (5)	−0·0013 (4)	0·5749 (4)	518 (15)
O(1)	0·3449 (3)	0·4612 (2)	0·2248 (3)	481 (10)
O(2)	0·3784 (4)	0·3980 (3)	0·6960 (4)	704 (13)
O(3)	0·4608 (4)	0·2735 (3)	0·4356 (5)	758 (15)

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

N(1)—C(2)	1·336 (3)	C(6)—C(5)	1·452 (4)
N(1)—C(10)	1·342 (4)	C(6)—C(11)	1·505 (4)
C(2)—N(3)	1·358 (3)	C(5)—O(20)	1·237 (3)
C(2)—N(14)	1·360 (3)	C(11)—O(12)	1·252 (3)
N(3)—C(4)	1·325 (4)	C(11)—O(13)	1·258 (3)
C(4)—C(9)	1·398 (4)	N(14)—C(15)	1·463 (4)
C(9)—C(10)	1·398 (4)	N(14)—C(19)	1·469 (3)
C(9)—C(5)	1·458 (4)	C(15)—C(16)	1·517 (5)
C(10)—N(8)	1·377 (3)	C(16)—N(17)	1·495 (3)
N(8)—C(7)	1·362 (4)	N(17)—C(18)	1·495 (4)
N(8)—C(21)	1·478 (3)	C(18)—C(19)	1·511 (5)
C(7)—C(6)	1·363 (4)	C(21)—C(22)	1·514 (5)
C(2)—N(1)—C(10)	115·8 (2)	C(7)—C(6)—C(5)	119·6 (3)
N(1)—C(2)—N(14)	116·8 (3)	C(5)—C(6)—C(11)	122·8 (3)
N(1)—C(2)—N(3)	126·7 (2)	C(9)—C(5)—C(6)	113·7 (3)
N(3)—C(2)—N(14)	116·4 (3)	C(6)—C(5)—O(20)	125·3 (3)
C(2)—N(3)—C(4)	115·2 (3)	C(9)—C(5)—O(20)	121·0 (3)
N(3)—C(4)—C(9)	124·3 (3)	C(6)—C(11)—O(13)	118·2 (3)
C(4)—C(9)—C(5)	122·3 (3)	C(6)—C(11)—O(12)	118·2 (3)
C(4)—C(9)—C(10)	114·7 (2)	O(12)—C(11)—O(13)	123·6 (3)
C(10)—C(9)—C(5)	123·0 (3)	C(2)—N(14)—C(19)	121·1 (2)
N(1)—C(10)—C(9)	123·4 (3)	C(2)—N(14)—C(15)	121·4 (3)
C(9)—C(10)—N(8)	119·5 (2)	C(15)—N(14)—C(19)	112·2 (2)
N(1)—C(10)—N(8)	117·1 (2)	N(14)—C(15)—C(16)	109·9 (2)
C(10)—N(8)—C(21)	121·1 (2)	C(15)—C(16)—N(17)	109·8 (3)
C(10)—N(8)—C(7)	118·7 (2)	C(16)—N(17)—C(18)	113·1 (2)
C(7)—N(8)—C(21)	120·2 (2)	N(17)—C(18)—C(19)	109·7 (2)
N(8)—C(7)—C(6)	125·2 (3)	N(14)—C(19)—C(18)	109·6 (3)
C(7)—C(6)—C(11)	117·5 (3)	N(8)—C(21)—C(22)	110·8 (3)

Experimental. Pipemidic acid was recrystallized from a mixture of chloroform, methanol and water at room temperature (crystals kindly supplied by Dr M. D. Veiga, Departamento de Farmacia Galénica, Universidad Complutense, Madrid), Prismatic crystal, approximate dimensions 0·26 × 0·16 × 0·50 mm. Philips PW 1100 four-circle diffractometer, graphite-monochromatized Cu $K\alpha$ radiation. Cell parameters by least squares based on 73 reflections in the range $11\cdot0 < 2\theta < 87°$. Cell parameters found by Cholin, Clanet, Ghemard, Souleau & Khodadad (1979) from powder diffraction data, reduced by program *TRACER* (Lawton & Jacobson, 1965), were similar. Intensities measured for $2 < \theta < 65°$ (index range: $-9 < h < 10, -14 < k < 15, 0 < l < 10$). Two standard reflections (300, $\bar{3}00$) monitored every 90 measurements, no significant change in intensity; 2831 independent reflections were collected with $\omega/2\theta$ scan technique, 2410 reflections considered observed with $|I| > 2\sigma(I)$. Intensities corrected for Lorentz and polarization effects. Absorption corrections not applied. Initial attempts to solve the structure using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) failed; E map always gave a so-called chicken-wire solution; structure solved finally using *MITHRIL* (Gilmore, 1983). Structure refined by full-matrix least squares with isotropic thermal parameters first, anisotropic later, for non-hydrogen atoms. The difference map showed no H atom in the neighbourhood of the O atoms of the carboxylic group, but two peaks with similar density values (0·40, 0·33 e Å⁻³) appeared near the apical N atom of the piperazine ring; the remaining H atoms were found at the expected positions; H atoms included in mixed refinement with positional and isotropic thermal parameters fixed; convenient weighting scheme to obtain flat dependence in $\langle \omega\Delta^2 F \rangle$ vs $\langle F_o \rangle$ and vs $\langle \sin\theta/\lambda \rangle$ (Martínez-Ripoll & Cano, 1975) was used. Final values: $R(F) = 0\cdot053$, $wR = 0\cdot056$, for 2410 observed reflections, max. $\Delta/\sigma = 0\cdot0008$, $\Delta\rho$ max. $= 0\cdot25$, min. $= -0\cdot27$ e Å⁻³, $S = 5\cdot71$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations performed with the *XRAY70* system of crystallographic programs (Stewart, Kundell & Baldwin, 1970) and *PARST* (Nardelli, 1983a) on the VAX 11/750 computer at Instituto Rocasolano.

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1 and molecular dimensions in Table 2.* Fig. 1 shows a view of the title compound with the numbering of the atoms.

The molecule contains a pyridine ring joined to a pyrimidine ring. This fused ring is nearly planar; the dihedral angle between the least-squares planes is 0·90 (7)°. The pyrimidine ring has attached at C(2) a piperazine ring, which adopts a chair conformation: $\Delta C_s^{N(14)} = 0·005$ (1), $\Delta C_2^{N(14)-C(19)} = 0·007$ (1) and $\Delta C_2^{C(15)-C(16)} = 0·020$ (1) in terms of the Nardelli (1983b) asymmetry parameters. The average torsion angle around the C(2)–N(14) bond is 13·8 (3)°. All bond lengths and angles in the three rings are consistent with the literature values.

The N(17) presents a quaternary character: this quaternization is produced by proton transfer from the carboxylic group to N(17); consequently, a 'zwitterion' structure is present, with the carboxyl group deprotonated and the piperazine N protonated. As a consequence, the C–O distances in the carboxyl group have similar values: 1·258 (3) and 1·252 (3) Å. This structure is stabilized by the interactions N(17)–H(171)···O(12)(x, y + 1, z), 2·735 (4) Å, and N(17)–H(172)···O(13)(−x + 2, −y, −z + 1), 2·780 (4) Å.

The substituents in the pyrimidine ring at C(5), C(6) and N(8) are the same as those supported by the pyrimidine ring in oxolinic acid (Cygler & Huber, 1985), nalidixic acid (Huber, Sake Gowda & Acharya, 1980) and 5-aminooxolinic acid (Czugler et al., 1976). Here, the geometry of such substituents is similar to that observed in the compounds mentioned above; the only differences are the C–O distance of 1·237 (3) Å and the lack, owing to deprotonation of the carboxyl group, of an intramolecular hydrogen bond between the carboxylic oxygen, O(12) and O(20), the adjacent carbonyl oxygen.

The N-ethyl substituent is here, as in oxolinic, aminooxolinic and nalidixic acids, almost perpendicular to the pyrimidine ring and slightly rotated about the C–N bond away from the carboxyl group. The value of the C(7)–N(8)–C(21)–C(22) torsion angle is −97·3 (3)°, in good agreement with −102·3 (2), −97·5 and −90·0° in oxolinic, aminooxolinic and nalidixic acids, respectively.

Cygler & Huber (1985) presented a very good review on a group of highly active antibacterial agents, all presenting a strong intramolecular hydrogen bond between an O of the carboxylic group and an O of the adjacent carbonyl group. Most of these compounds have an N-ethyl group in the pyridine ring. Pipemidic acid has antibacterial properties to a higher degree than nalidixic acid; nevertheless, it has no intramolecular hydrogen bond but the same N-ethyl substituent. This common part in the molecule should probably be the active group, in agreement with Cygler & Huber (1985).

There are eight crystallographically independent hydrogen bonds in the crystal structure in which all possible proton donors are involved; the molecules of the acid are linked head-to-tail by hydrogen bonds through the N(17) protons and the oxygens of the carboxyl group forming infinite ribbons running along y; these ribbons are held together by the water molecules giving rise to an intricate three-dimensional network. Table 3 and Fig. 2 show the geometry of the hydrogen bonds.

Table 3. *Hydrogen-bond geometry* (*distances in* Å, *angles in* °)

D–H···A	D···A	D–H	H···A	D–H···A
O(3)–H(31)···O(1^0)	2·989 (4)	0·96	2·04 (4)	172
O(3)–H(32)···O(2^0)	2·916 (5)	0·82	2·29 (5)	133
N(17)–H(172)···O(13^i)	2·780 (4)	0·97	1·85 (4)	160
O(1)–H(12)···O(12^{ii})	2·837 (3)	0·93	1·92 (4)	166
N(17)–H(171)···O(12^{iii})	2·735 (4)	1·01	1·73 (5)	173
O(1)–H(11)···O(13^{iv})	2·871 (4)	0·89	1·98 (5)	175
O(2)–H(21)···O(1^v)	2·818 (4)	0·99	1·87 (5)	158
O(2)–H(22)···O(20^{vi})	2·969 (5)	0·99	2·03 (5)	159

Symmetry code: (0) x, y, z; (i) −x + 2, −y, −z + 1; (ii) −x + 1, −y, −z; (iii) x, y + 1, z; (iv) x − 1, y + 1, z; (v) −x + 1, −y + 1, −z + 1; (vi) −x + 1, −y, −z + 1.
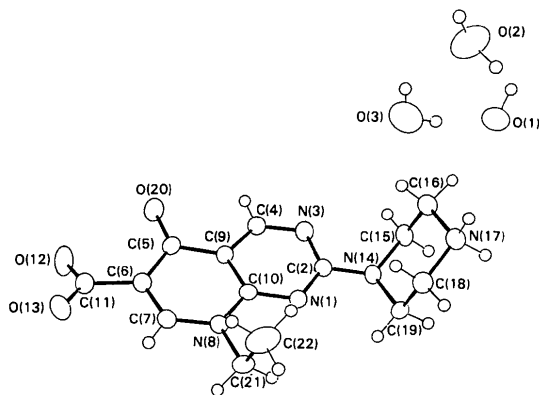

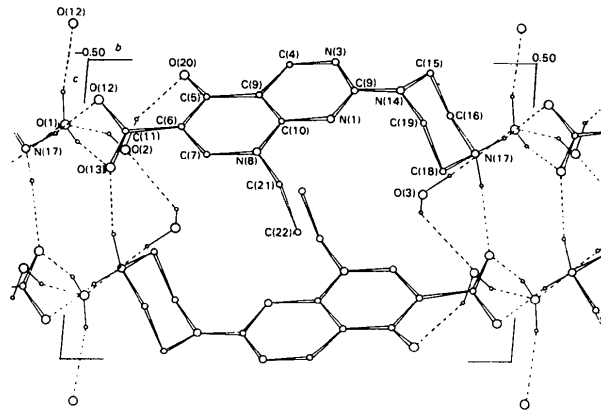Fig. 1. View of the molecule showing the atomic numbering.

Fig. 2. A view along [100] of the unit-cell contents. Hydrogen bonds are indicated by dashed lines.

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Structure of (R,R)-(+)-3,6-Bis[1-hydroxy-1-(4-methylphenyl)ethyl]-1,2,4,5-tetrazine

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Abstract. $C_{20}H_{22}N_4O_2$, $M_r = 350.4$, monoclinic, $P2_1$, $a = 11.322$ (5), $b = 5.655$ (3), $c = 14.802$ (8) Å, $\beta = 96.54$ (5)°, $U = 941.5$ Å^3, $Z = 2$, $D_x = 1.24$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.048$ mm^{-1}, $F(000) = 372$, $T = 293$ K, $R = 0.071$ for 893 unique reflections. Bond lengths and angles lie within normal ranges. The tetrazine and phenyl rings are planar. The structure contains one probable H bond, $O(1A)\cdots N(4)(x, 1 + y, z)$ 3.19 (1) Å.

Introduction. The title compound, m.p. 390–391 K, was prepared from (−)-4-methylatrolactamidinium chloride, $[\alpha]_{5461}$ −60.0° (in water) (Neilson, Mahmood & Watson, 1973). Its intense red colour precluded any measurement of its optical rotation. Some 1,2,4,5-tetrazines have proved of interest in the field of liquid crystal displays (Isenberg, Kruecke, Pelzl, Zashke & Demus, 1983) and our studies hope to show why some tetrazines show these properties and others do not.

Experimental. Crystals grown from water. Intense red prisms elongated along **b**. Crystal dimensions *ca* 0.3 × 0.7 × 0.3 mm. Cell dimensions from Weissenberg photographs and by diffractometry. Data collected in the range $k = 0$–5 from the *b* axis on a Stoe Stadi II two-circle diffractometer, $0 < \theta < 27°$, Mo $K\alpha$ radiation, range of indices: $-14 < h < 14$, $0 < k < 5$, $-18 < l < 18$. Standard reflections measured every 100 reflections, these varied by less than 5%. No absorption corrections were applied. 3916 reflections measured, 893 unique with $I > 3\sigma(I)$ regarded as observed, $R_{int} = 0.022$. Structure solved by direct methods with *MITHRIL* (Gilmore, 1984) using E values produced by *SHELX*76 (Sheldrick, 1976). The resulting E map revealed all the atoms in the molecule. Refinement (on F) by blocked-matrix least squares with *SHELX*76 (Sheldrick, 1976). Non-H atoms refined anisotropically; H atoms, attached to C atoms, included at calculated positions; hydroxyl H atoms fixed at positions approximately 0.9 Å from parent atom, these positions being based on significant peaks on difference map. H atoms were given isotropic temperature factors approximately 1.5 times those of their parent atoms. 235 parameters refined, $R = 0.071$, $wR = 0.084$, $w = 2.85/[\sigma(F)^2 + 0.0011F^2]$. Max. $\Delta/\sigma = 0.076$; max. difference map peak 0.35, min. −0.25 e Å^{-3}. The highest final difference map peaks lie at approximately 0.5 Å from atom $O(1A)$ suggesting that the H atom connected to this atom has a certain amount of rotational disorder (peak used as H atom in the calculation was 50% larger than the other relevant peaks). Other programs used, *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO*