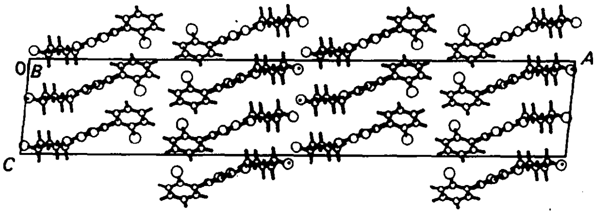

Fig. 2. Crystal packing viewed down **b**.

bond lengths in either structure. The C—O bond of the attached hydroxyl group is normal at 1·428 (4) Å.

The dihedral angle between the planes defining the phenyl and pyridazine rings is 45·55°. The relative orientation of the three ring moieties with respect to one another, and of the hydroxyl group with respect to the piperidine ring, are described by the torsion angles given in Table 2(*c*).

The packing arrangement of the molecule is shown in Fig. 2. This comprises molecules arranged as dimers across the centre of symmetry in a head-to-head fashion, hydrogen-bonded through the hydroxyl O atoms, while the same atoms generate infinite hydrogen-bonded ribbons through *c*-glide-related molecules (Table 2*d*).

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Structure of (—)-1-Phenylethylammonium Hydrogen (+)-Tartrate

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Abstract. $C_8H_{12}N^+.C_4H_5O_6^-$, $M_r = 271{\cdot}27$, monoclinic, $P2_1$, $a = 6{\cdot}352$ (2), $b = 14{\cdot}195$ (5), $c = 7{\cdot}507$ (2) Å, $\beta = 107{\cdot}08$ (2)°, $V = 647{\cdot}0$ (8) Å^3, $Z = 2$, $D_x = 1{\cdot}39$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 1·05 cm^{-1}, $F(000) = 288$, $T = 294$ K, $R = 0{\cdot}037$ for 941 observed reflections [$F > 2\sigma(F)$]. This (+)-tartrate structure is very similar to its *meso*-tartrate analogue. O(6) occupies an unusual antiperiplanar position relative to the carboxyl group. A strong hydrogen-bond network stabilizes the crystal packing.

Introduction. Preferential crystallization of diastereomeric salts is a classical procedure for the resolution of racemic bases and acids. (+)-Tartaric acid is, probably, the most popular acid employed in the resolution of amines. An investigation directed to exploiting its recognition abilities in solid-phase ('polymer supported') methodology is in progress. Within this context, it was considered that a detailed study of a pair of diastereomeric salts would afford information on the most convenient way in which tartaric acid should be anchored to the polymeric frame. In order to study the (+)-tartrate configuration in a chiral environment, we have undertaken the X-ray structural resolution of (+)-tartrate with the (—)-1-phenylethylammonium cation. A study of (—)-1-phenylethylammonium hydrogen *meso*-tartrate has been published (Kroon, Duisenberg & Peerdeman, 1984). Attempts to crystallize (+)-1-phenylethylammonium (+)-tartrate have been unsuccessful so far.

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Table 1. *Fractional atomic coordinates* ($\times 10^4$, *for* H $\times 10^3$) *with their e.s.d.'s and their isotropic temperature factors*

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

	x	y	z	B_{eq}/B_{iso}(Å^2)
O1	−2926 (4)	−676	−4848 (4)	3·30
O2	711 (4)	−676 (3)	−3474 (4)	3·42
O3	−2108 (5)	−1149 (3)	2219 (4)	3·25
O4	−4961 (5)	−236 (3)	811 (4)	3·73
O5	377 (4)	−247 (4)	−171 (4)	3·74
O6	−2160 (6)	−1979 (3)	−963 (4)	3·77
C1	−1225 (6)	−592 (4)	−3455 (5)	2·53
C2	−1633 (6)	−337 (4)	−1607 (5)	2·65
C3	−3093 (6)	−1068 (4)	−1061 (5)	2·72
C4	−3514 (6)	−779 (4)	763 (5)	2·59
N1	−2539 (6)	−4986 (4)	−3745 (5)	2·84
C5	−2411 (7)	−3924 (4)	−3605 (6)	3·23
C6	−3969 (7)	−3479 (4)	−5322 (6)	3·02
C7	−3699 (8)	−3598 (4)	−7071 (7)	3·68
C8	−5131 (9)	−3164 (5)	−8623 (7)	4·42
C9	−6803 (9)	−2611 (5)	−8441 (8)	4·78
C10	−7101 (8)	−2487 (5)	−6714 (9)	4·97
C11	−5693 (8)	−2920 (4)	−5142 (8)	3·94
C12	−13 (8)	−3630 (5)	−3281 (8)	4·08
H2	−242 (8)	26 (4)	−176 (7)	4·35
H3	−458 (8)	−110 (4)	−205 (7)	4·35
H5	−296 (8)	−377 (4)	−258 (8)	4·35
H7	−243 (8)	−398 (4)	−711 (7)	4·35
H8	−494 (9)	−337 (4)	−985 (7)	4·35
H9	−777 (8)	−231 (4)	−945 (7)	4·35
H10	−826 (9)	−213 (4)	−656 (7)	4·35
H11	−590 (8)	−285 (4)	−392 (7)	4·35
H121	90 (8)	−390 (4)	−211 (8)	4·35
H122	49 (8)	−383 (4)	−435 (7)	4·35
H123	14 (8)	−288 (2)	−319 (7)	4·35
HN1	−389 (9)	−518 (4)	−406 (7)	4·35
HN2	−186 (8)	−525 (4)	−254 (8)	4·35
HN3	−192 (7)	−522 (4)	−475 (7)	4·35
HO3	−258 (7)	−102 (4)	320 (7)	4·35
HO5	129 (8)	−33 (4)	−68 (7)	4·35
HO6	−142 (9)	−206 (4)	4 (8)	4·35

Experimental. Irregular prismatic crystal of approximate dimensions 0·20 × 0·18 × 0·35 mm; Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell constants from least-squares analysis of 25 high-angle reflections, intensity data collected by ω–2θ scan technique, no fluctuations in standard reflections (0·65%), $2\theta \leq 50°$, $-7 \leq h \leq 7$, $0 \leq k \leq 16$, $0 \leq l \leq 8$, 1317 reflections collected, 1194 unique reflections and 941 considered to be observed [$F > 2\sigma(F)$] and used in the structure analysis, Lp correction, no corrections for absorption.

The structure was solved by direct methods with a straightforward run of the *MULTAN*11/84 program (Main, Germain & Woolfson, 1984). A subsequent weighted Fourier synthesis gave the remaining non-H atoms. Full-matrix refinement on F was carried out with *SHELX*76 least-squares program (Sheldrick, 1976) using anisotropic thermal parameters for the non-H atoms and a global isotropic temperature factor for the H atoms, which were located by difference Fourier synthesis. The refinement process converged at $R = 0{\cdot}037$, $wR = 0{\cdot}035$ with $w = 1/[\sigma^2(F) + 0{\cdot}000452F^2]$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974) except for those of the H atoms (Stewart, Davidson & Simpson, 1965). No significant shift/e.s.d. values in the final cycle. Maximum and minimum final difference Fourier map peaks are 0·15 and −0·20 e Å^{-3}, respectively.

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

C1–O1	1·269 (4)	HO3–O3	0·89 (5)
C1–O2	1·240 (4)	HO5–O5	0·79 (5)
C4–O3	1·303 (5)	HO6–O6	0·77 (5)
C4–O4	1·208 (5)	H2–C2	0·97 (5)
C2–O5	1·414 (4)	H3–C3	1·02 (5)
C3–O6	1·415 (5)	HN1–N1	0·87 (5)
C2–C1	1·528 (5)	HN2–N1	0·95 (5)
C3–C2	1·526 (6)	HN3–N1	1·01 (5)
C4–C3	1·527 (5)	H5–C5	0·96 (5)
C5–N1	1·512 (6)	H7–C7	0·98 (5)
C6–C5	1·514 (6)	H8–C8	1·01 (5)
C12–C5	1·529 (6)	H9–C9	0·93 (5)
C7–C6	1·384 (6)	H10–C10	0·92 (5)
C11–C6	1·390 (6)	H11–C11	0·97 (5)
C8–C7	1·393 (7)	H121–C12	0·97 (5)
C9–C8	1·360 (8)	H122–C12	0·99 (5)
C10–C9	1·375 (8)	H123–C12	1·08 (5)
C11–C10	1·396 (7)		

O2–C1–O1	126·0 (3)	C6–C5–N1	110·5 (4)
C2–C1–O1	116·1 (3)	C12–C5–N1	108·3 (4)
C2–C1–O2	117·8 (3)	C12–C5–C6	113·1 (4)
C1–C2–O5	111·0 (3)	C7–C6–C5	121·6 (4)
C3–C2–O5	109·9 (3)	C11–C6–C5	119·5 (4)
C3–C2–C1	111·2 (3)	C11–C6–C7	118·9 (4)
C2–C3–O6	110·9 (3)	C8–C7–C6	120·5 (5)
C4–C3–O6	112·4 (3)	C9–C8–C7	120·5 (5)
C4–C3–C2	109·8 (3)	C10–C9–C8	119·8 (5)
O4–C4–O3	125·0 (3)	C11–C10–C9	120·6 (5)
C3–C4–O3	112·7 (3)	C10–C11–C6	119·7 (5)
C3–C4–O4	122·3 (3)		

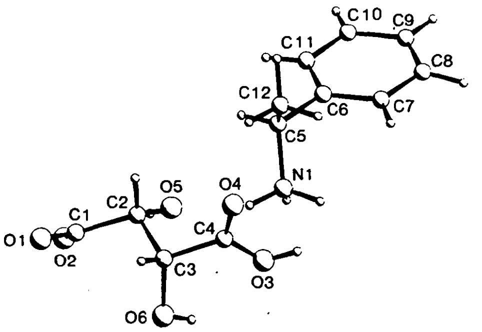

Fig. 1. Perspective view of the title compound with the atom numbering.

Discussion. Final atomic parameters are listed in Table 1.* Table 2 shows bond lengths and angles. Fig. 1 represents a perspective view of the asymmetric unit with the atom numbering. The general shape of the tartrate molecule can be defined by two different regions: the O(1), O(2), C(1), C(2), O(5) atoms lie in a plane (r.m.s.d. = 0·5%) while the O(3), O(4), O(6),

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51308 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

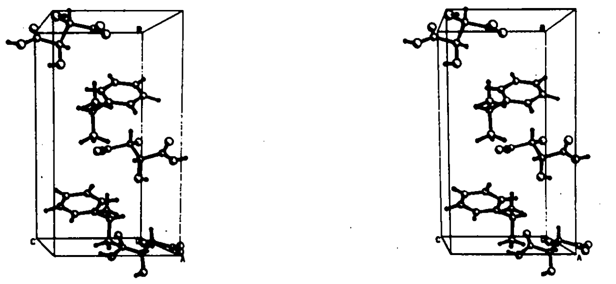

Fig. 2. Stereoscopic view of the unit cell.

Table 3. *Hydrogen-bond distances* (Å) *and angles* (°)

	O—H	H···O	O···O	O—H···O
$O3—HO3\cdots O1^{i}$	0·89	1·62	2·498	168
$N1—HN1\cdots O1^{ii}$	0·87	2·07	2·930	173
$N1—HN3\cdots O2^{iii}$	1·01	1·84	2·844	178
$N1—HN2\cdots O5^{iv}$	0·95	1·98	2·879	157
$O5—HO5\cdots O2$	0·79	2·08	2·622	126
$O5—HO5\cdots O4^{v}$	0·79	2·32	2·834	124
$O6—HO6\cdots O3$	0·77	2·22	2·655	116

Symmetry code: (i) $x, y, 1+z$; (ii) $-1-x, -0{\cdot}5-y, -1-z$; (iii) $-x, -0{\cdot}5+y, -1-z$; (iv) $-x, -0{\cdot}5+y, -z$; (v) $-1+x, y, z$.

C(3), C(4) atoms depart from an analogous plane (r.m.s.d. = 16·2%); this effect is mainly due to the repulsion between the β-hydroxyl groups and the neighbouring carboxyl O atoms.

The existence of dissymmetry in these two regions is similar to that in other reported X-ray structures (Kroon *et al.*, 1984; Moerman, Ouwerkerk & Kroon, 1985; Kroon, 1982). In particular, the title structure bears a striking resemblance to the *meso*-tartrate analogue, due to the approximate correspondence between their fractional coordinates (applying the adequate translation and symmetry operations), obviously except for atoms O(6) and H(3), which are interchanged as a consequence of the different configuration at C(3). The similarity between the (+)-tartrate and *meso*-tartrate structures lies in the fact that O(6) is antiperiplanar to the carboxyl group, which is very unusual, since it corresponds to an energetically unfavourable conformation. In this case the carboxyl groups are rotated from the C—C(α)—O(H) planes by 1·1 and 30·8°, which is more dissymmetric than in other reported tartrate moieties.

Crystal packing is drawn in Fig. 2 and Table 3 shows that the HO5 atom is involved in a bifurcated inter- and intramolecular hydrogen bond and is, as usual, coplanar with the donor and acceptor atoms (sum of the coordination angles is 360·0°).

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Structure of L-Phenylalanyl-L-proline Monohydrate

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Abstract. $C_{14}H_{18}N_2O_3.H_2O$, $M_r = 280{\cdot}3$, tetragonal, $P4_12_12$, $a = 8{\cdot}162\,(4)$, $c = 41{\cdot}41\,(3)$ Å, $V = 2758{\cdot}7$ Å^3, $Z = 8$, $D_m = 1{\cdot}32$, $D_x = 1{\cdot}35$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 7{\cdot}30$ cm^{-1}, $F(000) = 1200$, $R = 0{\cdot}037$, $wR = 0{\cdot}039$ for 840 unique reflections $[F > 2\sigma(F)]$. The peptide linkage is in *cis* conformation. The pyrrolidine ring exists as twist, ${}^{\beta}T_{\alpha}$. The crystal structure is stabilized by a three-dimensional network of N—H···O, O—H···O and C—H···O hydrogen bonds.

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