étudié correspond donc à celle des deux formes énantiomères possibles:

(*R*) triméthylène-2,1′
[(*S*) α-phényl-α-hydroxypropyl]-1 ferrocène
ou

(*S*) triméthylène-2,1
[(*R*) α-phényl-α-hydroxypropyl]-1 ferrocène .

V. Empilement moléculaire

Nous avons reporté sur la Fig. 5 les distances intermoléculaires les plus courtes. On remarque une distance de 2,85 Å entre les oxygènes de deux molécules proches voisines mais étant donné que les spectres infrarouges montrent que le proton de la fonction alcool dans chaque molécule est lié à l'atome de fer, il ne peut y avoir de liaison hydrogène intermoléculaire. Par ailleurs on remarque une distance de 3,35 Å entre les carbones C(22) et C(4). Toutes les autres distances sont supérieures à 3,50 Å.

Conclusion

Connaissant la configuration relative de la forme $F=122\,^{\circ}C$, on en déduit immédiatement celle de l'alcool diastéréoisomère $F=105\,^{\circ}C$ obtenu concuremment lors de l'action de C_6H_5MgBr sur la cétone II.

Or c'est l'isomère $F=122\,^{\circ}C$ qui se forme en majorité dans cette réaction. D'autre part nous avons signalé, dans l'introduction, qu'il y a inversion de la configuration de l'alcool prépondérant lorsqu'on change la chronologie de l'introduction des deux radicaux portés sur le carbone alcoolique (l'action de C_2H_5MgBr sur la cétone benzoylée donne un excédent de l'alcool $F=105\,^{\circ}C$).

On peut interpréter l'ensemble de ces phénomènes en supposant que l'encombrement par un pont triméthylène est suffisamment important pour imposer une inclinaison spécifique du groupe cétonique dans l'état de transition. Cette inclinaison doit être toujours la même quel que soit le radical alcoyle porté par la cétone.

Par contre, si l'induction est imposée par un groupe méthyle cette inclinaison varie avec l'encombrement du radical *R* porté par la cétone substrat.

Nous avons indiqué ailleurs les modèles proposés dans chacun des cas pour les états de transition (Moïse *et al.*, 1972).

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The Crystal Structure of Ammonium Tartrate

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Piezoelectric ammonium tartrate, $(NH_4)_2C_4H_4O_6$, crystallizes in the monoclinic space group $P2_1$ with $a=7{\cdot}083$ (1), $b=6{\cdot}128$ (3), $c=8{\cdot}808$ (1) Å; $\beta=92{\cdot}42$ (1)° and $Z=2$. The crystal structure has been determined from three-dimensional X-ray diffraction data by the symbolic addition method and refined by full-matrix least squares to an R index of 0·086. The tartrate ion consists of two planar halves, each having a carboxyl group, a tetrahedral carbon atom and a hydroxyl oxygen atom. The two parts are found to be identical within experimental error, with an interplanar angle of 62°. The structure is stabilized by a three-dimensional network of hydrogen bonds.

Introduction

The conformation of the tartrate ion has been determined in a number of structures (Beevers & Hughes, 1941; Sadanaga, 1950; van Bommel & Bijvoet, 1958; Ambady & Kartha, 1968; Ambady, 1968). The ion consists of two planar halves, each containing a carboxyl group, a tetrahedral carbon atom and a hydroxyl oxygen atom. It has been observed by Okaya, Stemple & Kay (1966) that although the two parts are similar in overall shape, there exist slight differences in certain equivalent bond lengths, bond angles or planarity. Whether such an asymmetry in these seemingly equivalent groups is due to packing forces or is inherent in the tartrate ion itself is not clear. To establish the conformation of the tartrate ion and to study the intermolecular hydrogen bonds, the present structure investigation was undertaken.

Experimental

Crystals of ammonium tartrate were grown by slow evaporation of an aqueous solution of the salt. The cell constants determined from Weissenberg photographs compare well with the more precise data (Table 1) obtained by Zhdanov, Umanski, Barfolomeeva, Ezhkova & Zolina (1956).

Table 1. *Crystal data*

Molecular formula	$(NH_4)_2C_4H_4O_6$
Lattice constants	$a=7{\cdot}083$ (1) Å
	$b=6{\cdot}128$ (3)
	$c=8{\cdot}808$ (1)
	$\beta=92{\cdot}42$ (1)°
Density	$d=1{\cdot}611$ g cm^{-3}
Molecules per unit cell	$Z=2$
Systematic absences	$0k0$: $k=2n+1$
Space group	$P2_1$ (No. 4, C_2^2), monoclinic

Three-dimensional intensities were collected by the multiple-film equi-inclination Weissenberg technique with nickel-filtered Cu $K\alpha$ radiation. The intensities were collected for $h0l$ to $h3l$ and $0kl$ to $4kl$ from approximately cylindrical crystals having diameters 0·28 and 0·42 mm respectively. The intensities of 675 independent reflexions were estimated visually by comparison with a standard scale. The unobserved reflexions were estimated as $I=\frac{1}{2}I_{min}$. Corrections for absorption, polarization and Lorentz factors were made in the usual way. Interlayer scaling was achieved using cross-layer data and the corrected intensities were placed on an absolute scale by Wilson's method.

Structure determination

The structure was determined by the symbolic addition method (Karle & Karle, 1966). Normalized structure amplitudes, $|E|$, were calculated and rescaled to set the averages of $|E|^2$ equal to 1. The statistical averages of $|E|$'s obtained are given in Table 2 and confirm that the structure is non-centric.

Table 2. *Statistical averages of* $|E|$*'s*

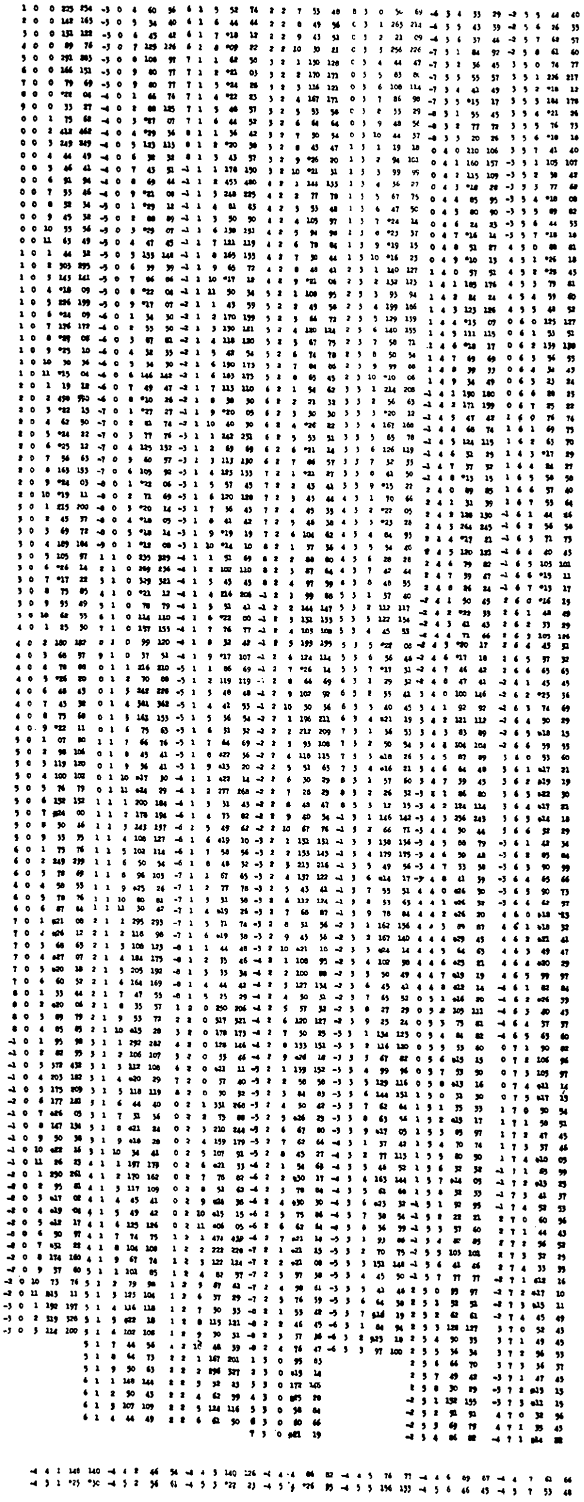
	Experimental	Theoretical	
		Non-centric	Centric
$\langle\lvert E\rvert\rangle$	0·84	0·886	0·798
$\langle\lvert E\rvert^2\rangle$	1·00	1·00	1·00
$\langle\lvert E\rvert^2-1\rangle$	0·81	0·736	0·968

Table 3. *Initial phase assignments*

h	k	l	E	Phase	
−1	0	7	1·955	0	origin
−7	0	4	1·631	0	
−6	1	2	2·729	0	
−4	5	5	3·450	a	
6	0	2	2·588	b	
−2	0	8	2·204	c	

Table 4. *The observed and calculated structure amplitudes*

Successive columns contain h, k, l, $|F_o|$ and F_c. An asterisk with $|F_o|$ means unobserved reflexion.



Three linearly independent and highly interacting reflexions with large E values were chosen and given signs arbitrarily to fix the origin. Three more reflexions with large E values were assigned symbols. This basic set of phases for the application of the $\sum_2$ formula is given in Table 3.

Phases for 70 strong E values ($E \geq 1 \cdot 6$) were obtained in terms of these symbols using the relationship

$$\varphi_{\mathbf{h}} = \langle \varphi_{\mathbf{k}} + \varphi_{\mathbf{h}-\mathbf{k}} \rangle_{\mathbf{kr}}.$$

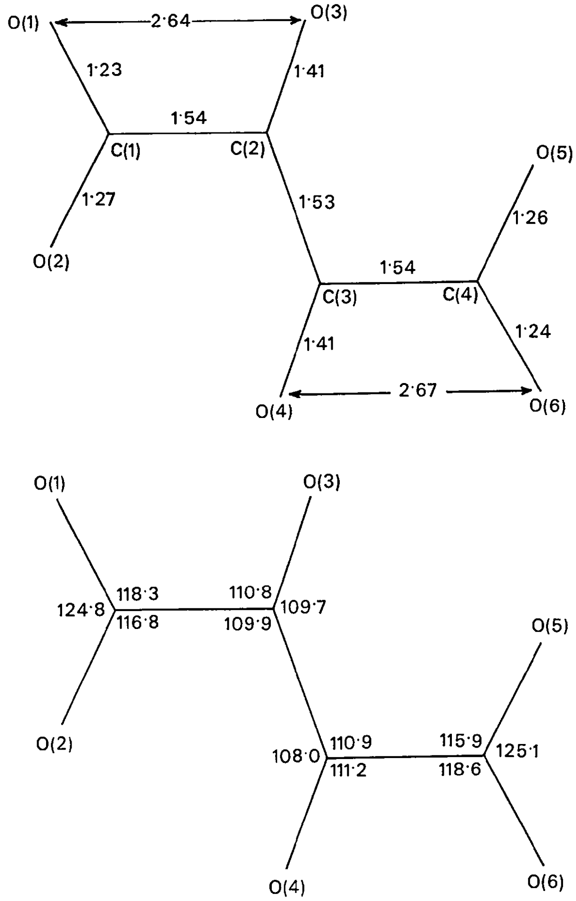


Fig. 1. Features of the tartrate ion.

There were several indications that $b=\pi$, and it was assigned this value. For 'a' and 'c' there were no indications, and as usual 'a' was given the value $\pi/2$, which fixes the enantiomorph. Since $\bar{2}08$ is a centric reflexion for the space group $P2_1$, two sets of phases were obtained one corresponding to $c=0$ and other $c=\pi$. Both these sets were refined by the tangent refinement method using the computer program *TANFMULA* (Sikka, 1970) and phases for 140 reflexions ($E \geq 1 \cdot 2$) were obtained. E maps were drawn for each set and the one corresponding to $c=\pi$ had 10 strong peaks, which were interpretable as atoms of the structure from stereochemical considerations. The remaining two atoms were located from a difference Fourier synthesis.

Refinement

The atomic positions obtained from the E map and Fourier synthesis were refined by the full-matrix least-squares method using the computer program *ORFLS* (Busing, Martin & Levy, 1962). Initially, individual isotropic temperature factors were used for all atoms, unit weights for the observed structure amplitudes and zero weights for the unobserved ones. Individual scale factors were assigned to each layer and were treated as variables during the refinement. The quantity minimized was $\sum w(|F_c| - k|F_o|)^2$. A value of 0·16 was obtained for the conventional R index after a few cycles of refinement.

At this stage, it was noticed that the thermal parameters of some of the atoms had assumed rather low values and also for strong reflexions the discrepancy between $|F_o|$ and $|F_c|$ was rather large. These effects could be due to extinction. Therefore, an approximate correction for isotropic secondary extinction was applied using Zachariasen's (1967) method. $\bar{T}$, the absorption weighted mean path length in the crystal, was calculated for each reflexion from the following relation (Coppens & Hamilton, 1970):

$$\bar{T} \simeq -\log A/\mu\,,$$

A being the absorption correction factor ($\mu r < 0 \cdot 5$). Three cycles of refinement, with the extinction con-

Table 5. *Fractional coordinates and thermal parameters with their standard deviations in parentheses*

The thermal parameters are defined by $T=\exp\{-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\}$.

All values are multiplied by 10^4.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	3022 (12)	2940 (25)	7701 (11)	37 (16)	118 (33)	51 (11)	−41 (25)	−30 (10)	20 (17)
O(1)	3119 (10)	986 (20)	7405 (9)	87 (15)	80 (23)	92 (11)	−27 (19)	20 (9)	7 (13)
O(2)	1495 (9)	4034 (21)	7714 (9)	42 (13)	118 (23)	135 (13)	1 (17)	−17 (9)	−29 (16)
C(2)	4865 (12)	4166 (21)	8134 (10)	41 (17)	56 (27)	65 (12)	−13 (21)	−18 (10)	6 (16)
O(3)	6404 (9)	2708 (20)	8274 (8)	77 (13)	119 (25)	69 (9)	− 9 (18)	−19 (8)	− 4 (13)
C(3)	5250 (12)	5883 (21)	6927 (10)	71 (18)	64 (26)	34 (10)	−24 (23)	6 (11)	− 1 (15)
O(4)	5393 (11)	4802 (17)	5518 (7)	145 (16)	131 (24)	31 (8)	−64 (18)	−23 (8)	−11 (11)
C(4)	7067 (12)	7177 (20)	7349 (9)	38 (14)	64 (26)	43 (11)	18 (20)	10 (10)	17 (14)
O(5)	7127 (11)	8077 (19)	8639 (9)	150 (18)	137 (25)	89 (11)	−47 (21)	22 (11)	−54 (15)
O(6)	8265 (10)	7414 (18)	6371 (9)	83 (15)	133 (25)	99 (11)	4 (18)	1 (10)	11 (14)
N(1)	1051 (11)	8048 (21)	9145 (9)	63 (15)	117 (26)	66 (10)	−32 (21)	− 7 (10)	4 (15)
N(2)	8862 (12)	1947 (22)	5651 (10)	76 (16)	152 (29)	70 (12)	−35 (20)	33 (10)	−23 (15)

stant g' (Coppens & Hamilton, 1970) as a variable parameter, reduced R to 0·11. Another three cycles of refinement with anisotropic temperature factors and a Cruickshank (1961) weighting scheme based on an error analysis plot gave a final R of 0·086. The extinction constant, g', had the value 2·26, corresponding to a value of 2·55 sec for the equivalent mosaic spread parameter, η. For the worst-affected reflexion, 202, F_o^2/F_c^2 was found to be 0·15.

The scattering factors for C, N and O atoms were taken from *International Tables for X-ray Crystallography* (1962). The observed and calculated structure amplitudes are given in Table 4, the fractional coordinates and anisotropic thermal parameters of the atoms in the Table 5. The intramolecular bond lengths and angles in the tartrate ion are given in Table 6.

Table 6. *Intramolecular bond distances and angles with estimated standard deviations*

C(1)–O(1)	1·227 ± 0·013 Å
C(4)–O(6)	1·243
C(1)–O(2)	1·274
C(4)–O(5)	1·262
C(2)–O(3)	1·411
C(3)–O(4)	1·414
C(1)–C(2)	1·540
C(2)–C(3)	1·528
C(3)–C(4)	1·544
O(1)–C(1)–O(2)	124·8 ± 0·9°
O(5)–C(4)–O(6)	125·1
O(1)–C(1)–C(2)	118·3
O(6)–C(4)–C(3)	118·6
O(2)–C(1)–C(2)	116·8
O(5)–C(4)–C(3)	115·9
O(1)–C(2)–C(3)	109·9
C(1)–C(2)–O(3)	110·8
O(3)–C(2)–C(3)	109·7
C(2)–C(3)–O(4)	108·0
O(4)–C(3)–C(4)	111·2
C(2)–C(3)–C(4)	110·9

Distances between non-bonded atoms in the molecule

O(1)···O(3)	2·638 ± 0·013 Å
O(4)···O(6)	2·671
O(1)···O(2)	2·216
O(5)···O(6)	2·223

Discussion

The tartrate ion

The tartrate ion (Fig. 1) consists of two planar halves, O(1) O(2) C(1) C(2) O(3) and C(3) O(4) C(4) O(5) O(6) with an interplanar angle of 62°. The two planes are so oriented that all four carbon atoms lie in a plane. The details of the least-squares planes fitted to the three groups, with deviations from each plane, are given in Table 7. A stereoscopic view of the molecule drawn by the computer program *ORTEP* (Johnson, 1965) is shown in Fig. 2. The chemically equivalent bonds in the tartrate ion are (1) C(1)–C(2) and C(3)–C(4); (2) the four C–O bonds of the carboxyl groups and (3) the two C–O(H) bonds. No significant differences are observed in the lengths of equivalent bonds (Table 6, Fig. 1). The angles O(1)–C(1)–C(2) (118·3°) and O(6)–C(4)–C(3) (118·6°) are larger than the other pair O(2)–C(1)–C(2) and O(5)–C(4)–C(3) (116·8, 115·9°) respectively. Although the accuracy of the determination is not very high in this structure, it appears that basically the two halves of the tartrate ion are identical. The asymmetries between the two halves of the tartrate ion observed in the other tartrates are possibly due to differences in their environment and packing forces (Okaya *et al.*, 1966; Ambady & Kartha, 1968).

Table 7. *Deviations of atoms from least-squares planes* (Å)

Plane 1: C(1) C(2) C(3) C(4)
Plane 2: O(1) O(2) C(1) C(2) O(3)
Plane 3: O(6) O(5) C(4) C(3) O(4)

	Plane 1	Plane 2	Plane 3
O(1)		0·018	
O(2)		−0·043	
O(3)		−0·053	
O(4)			0·047
O(5)			0·025
O(6)			−0·030
C(1)	−0·008	0·016	
C(2)	0·008	0·061	
C(3)	0·009		−0·065
C(4)	−0·008		0·023

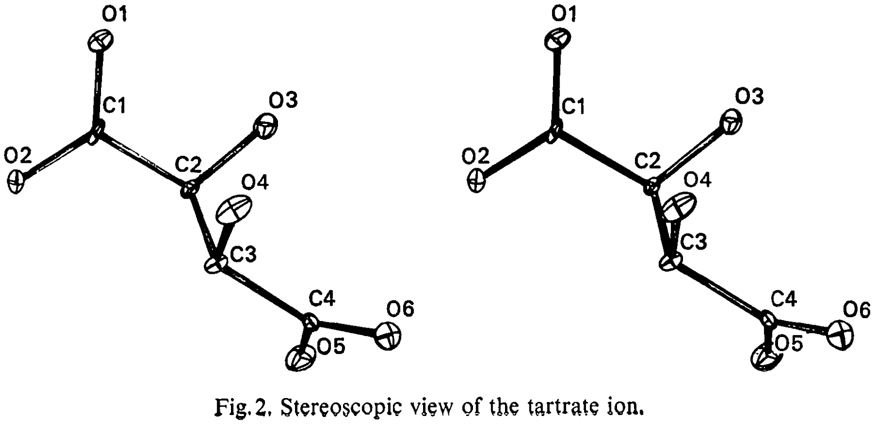


Fig. 2. Stereoscopic view of the tartrate ion.

Molecular packing and hydrogen bonding

The structure projected down the *c* axis is shown in Fig. 3. The tartrate molecules form a zigzag chain along the *b* axis and are held in this position by a network of hydrogen bonds through the ammonium ions. In addition, the hydroxyl oxygen atoms are involved in hydrogen bond formation with carboxyl or hydroxyl oxygen atoms of other tartrate ions. Thus, there are two types of hydrogen bond in the structure: (i) N–H···O, where the nitrogen atom is from an ammonium ion and the acceptor oxygen atoms are either carboxyl or hydroxyl oxygens of different tartrate ions, and (ii) O–H···O, where the donor oxygen atom is a hydroxyl oxygen and the acceptor atom is either a carboxyl or a hydroxyl oxygen of another tartrate ion. The nitrogen atom N(1) of one of the ammonium ions has five oxygen atoms O(2), O(5), O(1), O(3*A*) and O(6) at distances less than 3·4 Å (Table 8). Of these, the three carboxyl oxygens O(2), O(5), O(1) and one hydroxyl oxygen O(3*A*) are at reasonable distances (< 3·0 Å) and in satisfactory directions [angles subtended at N(1) ranging between 93·7 and 135·0°] to form hydrogen bonds in a distorted tetrahedral configuration about the ammonium nitrogen. The nitrogen atom N(2) of the other ammonium ion is surrounded by six oxygen atoms from different tartrate molecules at distances less than 3·4 Å (Table 8). Of these, three carboxyl oxygens O(6), O(2), O(6*A*) and one hydroxyl oxygen, O(4) at distances 2·77 to 3·01 Å are arranged in a distorted tetrahedral configuration (angles 83·4 to 116·1°) and are possibly involved in hydrogen bond formation. The hydroxyl oxygen O(3) is situated at a distance 2·99 Å from nitrogen N(2) and it is possible that one proton of the ammonium ion is involved in the formation of a bifurcated hydrogen bond. For the formation of the second type of hydrogen bond, the hydroxyl oxygen O(4) has four oxygen atoms O(1*A*), O(4*A*), O(4*A*) and O(6*A*) from different tartrate molecules at distances less than 3·4 Å (Table 8). Of these, the carboxyl oxygen O(1*A*) at a distance 2·91 Å and with

Table 8. *Hydrogen bond distances and angles*

The atoms with a superscript are in adjacent unit cells and the superscript gives the change involved.

O(3)···O(5)$^{y-1}$	2·89 Å	C(2)—O(3)-O(5)	139·6°
O(4)···O(1*A*)	2·91	C(3)—O(4)-O(1*A*)	134·5
O(4)···O(4*A*)	3·24	C(3)—O(4)-O(4*A*)	77·7
O(4)···O(4*A*)$^{y-1}$	3·24	C(3)—O(4)-O(4*A*)	132·3
O(4)···O(6*A*)	3·36	C(3)—O(4)-O(6)	123·8
N(1)···O(2)	2·79	O(2)—N(1)-O(5)	93·7
N(1)···O(5)$^{x-1}$	2·80	O(2)—N(1)-O(1)	104·3
N(1)···O(1)$^{y+1}$	2·81	O(2)—N(1)-O(3*A*)	102·5
N(1)···O(3*A*)$^{z+1}$	2·85	O(5)—N(1)-O(1)	116·5
N(1)···O(6)$^{x-1}$	3·10	O(5)—N(1)-O(3*A*)	135·9
		O(1)—N(1)-O(3*A*)	98·9
		O(6)—N(1)-O(2)	67·2
		O(6)—N(1)-O(5)	43·9
		O(6)—N(1)-O(1)	88·8
		O(6)—N(1)-O(3*A*)	168·6
N(2)···O(6*A*)$^{x+1}$	2·77	O(6*A*)-N(2)-O(2)	83·4
N(2)···O(2)$^{x+1}$	2·85	O(6*A*)-N(2)-O(6)	111·0
N(2)···O(6)$^{y-1}$	2·88	O(6*A*)-N(2)-O(4)	122·4
N(2)···O(4)	3·01	O(2)—N(2)-O(6)	112·9
N(2)···O(3)	2·99	O(2)—N(2)-O(4)	105·9
N(2)···O(1)	3·38	O(6)—N(2)-O(4)	116·1
		O(3)—N(2)-O(6*A*)	61·3
		O(3)—N(2)-O(2)	79·9
		O(3)—N(2)-O(6)	83·4
		O(3)—N(2)-O(4)	55·8

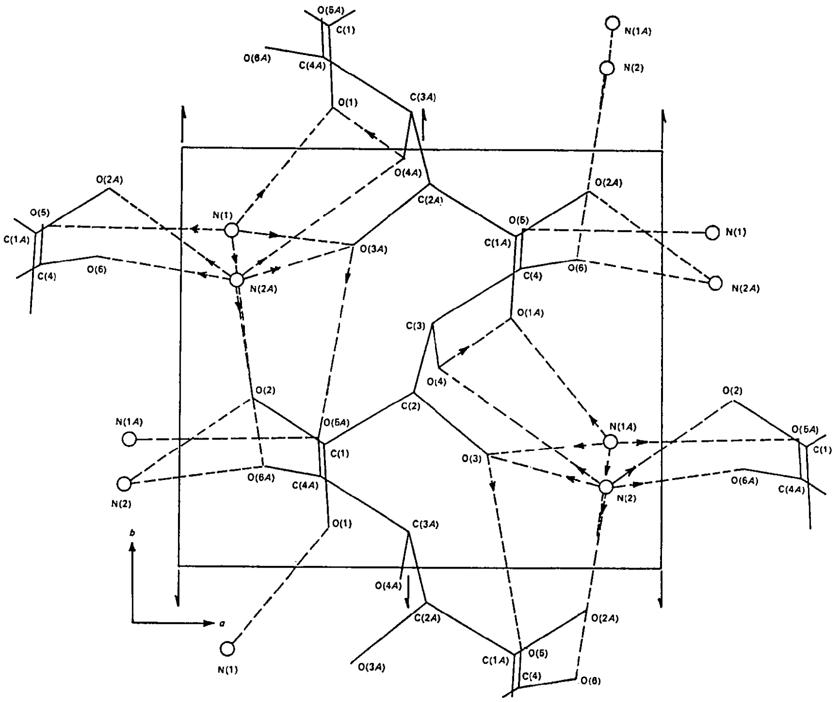


Fig. 3. The crystal structure of ammonium tartrate projected down the *c* axis.

the angle C(2)–O(3)–O(5) as 139·6° is favourable for hydrogen bond formation. The other hydroxyl oxygen O(3) has only one intermolecular oxygen O(5) at a distance less than 3·4 Å (Table 8) and a hydrogen bond is possible between these two. These possible hydrogen bonds have been indicated by dashed lines in Fig. 3.

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The Crystal Structure of Thallous Fluoride Carbonate

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The crystal structure of the title compound has been determined by single-crystal X-ray diffraction with 611 observed reflexions refined to an R value of 0·072. The crystals are monoclinic, space group $P2_1/m$ with $a=7{\cdot}510$, $b=7{\cdot}407$, $c=6{\cdot}069$ Å, $\gamma=120{\cdot}00°$, $Z=2$, $D_c=7{\cdot}86$. The structure is ionic and the three independent Tl^+ ions have almost identical environments, each with 7 neighbours in capped octahedral geometry. The Tl–O and Tl–F distances show the effect of a sterically active electron pair on Tl^+. There are three long Tl–O (3·09 and 3·18 Å twice, mean values) to oxygen atoms in one face of the octahedron. The opposite face has intermediate distances Tl–F 2·751 Å (twice) and Tl–O 2·85 Å, while the capping oxygen atom in the centre of this face has Tl–O 2·61 Å. The packing is related to that of NiAs.

While attempting to grow crystals of thallous fluoride (Alcock, 1969 and in preparation) by slow evaporation in air of an ethanol/water (5:1) solution, a small number of chunky hexagonal-prismatic crystals were observed, very different from the thin flakes normal for thallous fluoride. The crystals were examined in the hope that they might be a less intractable modification of thallous fluoride, but they were eventually found to be a mixed fluoride–carbonate. However, as such their structure is of interest, both as an apparently novel structure type, with affinities with the NiAs structure, and as an instance of a stereochemically active 'inert' pair of electrons on the Tl^+ ion.

Experimental

Preparation

The characterization of this compound rests on the results of the structure analysis and is discussed below. The original sample of thallous fluoride was prepared by heating thallous carbonate with excess hydrofluoric acid, reducing to dryness and then heating to above the melting point; it had lattice constants identical with those reported for thallous fluoride. The mixed fluoride carbonate was then obtained as above. From the method of preparation it should have contained no carbonate and the only likely source for this is carbon