by comparison with a *bona fide* hindered triarylphosphine, *i.e.* tris(2,6-dimethylphenyl)phosphine, (3) (Sobolev, Chetkina, Romm & Gur'yanova, 1976). In this case, although the average C—P bond length (1·842 Å) is similar to that of (1), the average C—P—C bond angle (109·5°) is substantially enlarged.

In (1) the average P—C—C angle *syn* to the phosphorus lone pair [*e.g.* P(1)—C(1)—C(6)] is 116·3° and the average P—C—C angle *anti* to the phosphorus lone pair [*e.g.* P(1)—C(1)—C(2)] is 126·8°. While these are appreciably different from the idealized 120°, they are essentially the same as the corresponding averages in (2) (*i.e.* 116·1 and 124·0°) and (3) (*i.e.* 113·5 and 127·3°).

Phosphine (1) differs from (3) in one curious respect: the *ortho*-methyl groups of (3) are bent slightly *away* from the C—P bonds while the *ortho*-methoxy groups of (1) are bent slightly *toward* the C—P bonds. In (1) the average of all O—C—C angles analogous to O(6)—C(22)—C(17) is 116·6° while the average of O—C—C angles analogous to O(6)—C(22)—C(21) is 121·9°. For (3) the corresponding averages are 123·2 and 117·0°, respectively.

The angles that the planes of the aryl rings make with the plane defined by C(1), C(9) and C(17) are 95·2 [C(1)–C(6)], 43·7 [C(9)–C(14)] and 33·6° [C(17)–C(22)]. Thus two phenyls are tilted and one [C(1)–C(6)] is approximately parallel to the phosphorus lone pair. Five of the six methoxy groups lie approximately in the plane of the attached aromatic ring. Absolute values of the pertinent dihedral angles are: C(7)—O(1)—C(2)—C(3), 4·0; C(15)—O(3)—C(10)—C(11), 23·1; C(16)—O(4)—C(14)—C(13), 1·3; C(23)—O(5)—C(18)—C(19), 17·3; C(24)—O(6)—C(22)—C(21), 4·9°. One methoxy group lies approximately perpendicular to the plane of the attached aromatic ring, *e.g.* the dihedral angle C(8)—O(2)—C(6)—C(5) is 93·3°.

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Acta Cryst. (1991). C**47**, 1005–1009

Structural Studies of Systems with 'Very Short' Hydrogen Bonds. Structures of Methylammonium Hydrogen Succinate Monohydrate and Dimethylammonium Hydrogen Succinate at 110 K

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(*Received* 12 *July* 1990; *accepted* 31 *August* 1990)

Abstract. Methylammonium hydrogen succinate monohydrate, $CH_6N^+.C_4H_5O_4^-.H_2O$, $M_r = 167·16$, monoclinic, $P2_1/m$, $a = 4·9788$ (5), $b = 13·077$ (1), $c = 6·133$ (2) Å, $\beta = 92·34$ (1)°, $V = 399·0$ (2) Å^3, $Z = 2$, $D_x = 1·391$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 1·164$ cm^{-1}, $F(000) = 180$, $T = 110$ K, $R = 0·035$ for 2627 observed reflections. Dimethylammonium hydrogen succinate, $C_2H_8N^+.C_4H_5O_4^-$, $M_r = 163·17$, orthorhombic, $Cmcm$, $a = 6·7863$ (9), $b = 8·069$ (2), $c = 14·284$ (3) Å, $V = 782·2$ (4) Å^3, $Z = 4$, $D_x = 1·386$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 1·086$ cm^{-1}, $F(000) = 352$, $T = 110$ K, $R = 0·028$ for 1142 observed reflections. In both compounds the hydrogen succinate ions are linked by 'very short' hydrogen bonds resulting in infinite chains. The O atoms involved in the short hydrogen bond are related by crystallographic symmetry in both salts, making the short hydrogen bond symmetric in a crystallographic sense. The H atom in the short hydrogen bond seems to be best described by two equally populated sites on each side of the centre of the hydrogen bond.

Introduction. Acid salts of carboxylic acids have earlier been thoroughly investigated by Speakman (1972). He classified these compounds in two groups.

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In type *A* salts the two COO groups are related by crystallographic symmetry elements. The other acid salts where the COO and COOH groups are crystallographically non-equivalent are referred to as type *B* salts.

An interesting aspect of the carboxylic acid salts is the presence of very short hydrogen bonds in their crystal structures. The shortest hydrogen bonds were found in type *A* salts. Hadži (1965) has performed careful spectroscopic investigations of systems with short hydrogen bonds and was able to make a classification according to their IR spectra. These short hydrogen bonds are also found in acid salts of dicarboxylic acids. It is a general trend in many of these compounds that the short hydrogen bonds link the anions into infinite chains.

The present structure determinations for methylammonium hydrogen succinate monohydrate (MAHS) and dimethylammonium hydrogen succinate (DIMAHS) are part of our investigations of acid salts of malonic and succinic acids. These salts contain the same structural element of infinite chains of anions. The structural features of the short hydrogen bonds are elucidated by use of accurate low-temperature X-ray diffraction data and the influence of different cations on the crystal packing is examined.

Table 1. *Crystal data and a summary of results from data reduction and structure refinements*

	MAHS	DIMAHS
Formula	$C_5H_{13}NO_5$	$C_6H_{13}NO_4$
M_r	167·16	163·17
Space group	$P2_1/m$	*Cmcm*
Temperature (K)	110	110
Crystal size (mm)	0·20 × 0·30 × 0·50	0·17 × 0·30 × 0·35
Unit-cell dimensions		
a (Å)	4·9788 (5)	6·7863 (9)
b (Å)	13·077 (1)	8·069 (2)
c (Å)	6·133 (2)	14·284 (3)
β (°)	92·34 (1)	
V (Å^3)	399·0 (2)	782·2 (4)
Z	2	4
D_x (g cm^{-3})	1·391	1·386
Intensity data collection		
Max. $\sin\theta/\lambda$ (Å^{-1})	1·0770	1·1079
Range of *h*	−8–10	0–17
Range of *k*	−12–28	−15–15
Range of *l*	−13–13	−31–31
Standard reflections	101, 040, 121	202, 002, 022
Type of scan	ω–2θ	ω
Number of measured reflections	7207	8725
Structure refinement		
Number of unique reflections	4282	2391
Number of reflections used in refinement [$I/\sigma(I) > 2$]	2627	1142
Number of variables	85	53
Weights w^{-1}	$\sigma_{cs}^2(F) + 0·0004F^2$	$\sigma_{cs}^2(F) + 0·0004F^2$
R	0·035	0·028
wR	0·046	0·036
S	1·444	0·990
Max. shift/e.s.d.	0·01	0·02
Max. and min. $\Delta\rho$ (e Å^{-3})	0·745, −0·420	0·551, −0·284

Experimental. Suitable crystals for the diffraction study of MAHS and DIMAHS were obtained by slow evaporation from aqueous solutions which contained equivalent molar amounts of succinic acid and the respective amine (methylamine and dimethylamine). Both compounds form colourless crystals. The crystals of MAHS are elongated along the *c* axis. The data collection was performed at 110 K using an Enraf–Nonius CAD-4 diffractometer with graphite-monochromatized Mo $K\bar{\alpha}$ radiation. Unit-cell dimensions were determined from a least-squares refinement of 22 reflections with θ in the range 18·9–22·8° (MAHS) and 18·3–21·8° (DIMAHS). The different experimental conditions and results from the refinements are summarized in Table 1. Data reduction included Lp correction, and for DIMAHS also a correction for decay, but no absorption correction in either case. The remeasurement of three standard reflections every 10 000 s showed a linear decrease in intensity with exposure time up to 4·6% for this compound. The maximum correction factor is 1·024. The symmetry-equivalent reflections were averaged giving $R_{\text{int}} = 0·021$ (MAHS) and 0·032 (DIMAHS). Both structures were solved by direct methods with the program *SHELXS*86 (Sheldrick, 1990) and refined using the full-matrix least-squares method minimizing $\sum w(|F_o| - |F_c|)^2$. The positions of the H atoms were shown clearly in a difference Fourier map and their positional parameters and isotropic displacement parameters were included in the refinement. Two refinement models were examined for the position of the H atom in the short hydrogen bond. One with the H atom in a special position yielded physically unreasonable, very large displacement parameters $B = 5·1$ (5) Å^2 for MAHS and $B = 9·1$ (9) Å^2 for DIMAHS. When the H atom was placed in a general position corresponding to two half-populated sites on each side of the centre of the hydrogen bond, physically reasonable displacement parameters were obtained, $B = 2·8$ (4) Å^2 for MAHS and $B = 1·1$ (6) Å^2 for DIMAHS. These values are comparable to those of the other H atoms in the structures. The separation between the two half-populated H atoms is 0·60 (4) Å in MAHS and 1·03 (3) Å in DIMAHS. The fact that the displacement parameter for the H atom in DIMAHS, when placed in the special position, is much larger than in MAHS fits well with the larger distance between the two hydrogen sites in this compound. All calculations except the structure determinations were performed using the Enraf–Nonius *SDP* program system (Enraf–Nonius, 1985). Scattering-factor values for all atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and used as contained in the program system. The final

Table 2. *Fractional coordinates and displacement parameters* (Å^2) *for* MAHS *and* DIMAHS

	Wyckoff	Site symmetry	*x*	*y*	*z*	U_{11}/U_{iso}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
MAHS											
O(1)	*f*	1	0·17475 (8)	0·53918 (3)	0·39328 (6)	0·01946 (11)	0·01499 (9)	0·01324 (10)	−0·00532 (10)	0·01068 (8)	−0·00544 (10)
O(2)	*f*	1	0·12537 (7)	0·40465 (3)	0·17205 (6)	0·01543 (11)	0·01066 (9)	0·01126 (10)	−0·00293 (10)	0·00533 (8)	−0·00136 (8)
C(1)	*f*	1	0·23789 (8)	0·48631 (3)	0·22601 (7)	0·01069 (11)	0·01005 (9)	0·00799 (10)	−0·00033 (10)	0·00389 (8)	−0·00002 (10)
C(2)	*f*	1	0·46505 (8)	0·53077 (4)	0·09967 (7)	0·01200 (11)	0·01308 (17)	0·00944 (11)	−0·00259 (12)	0·00549 (9)	−0·00160 (10)
N	*e*	*m*	−0·11604 (12)	0·25000	−0·56863 (9)	0·01345 (18)	0·01048 (17)	0·01082 (15)	0·00000	0·00209 (14)	0·00000
C(3)	*e*	*m*	−0·41425 (15)	0·25000	−0·58190 (14)	0·01274 (21)	0·01542 (26)	0·02420 (30)	0·00000	−0·00138 (22)	0·00000
O(*W*)	*e*	*m*	0·09454 (18)	0·25000	0·85626 (10)	0·05198 (39)	0·01308 (17)	0·01254 (19)	0·00000	−0·00756 (23)	0·00000
H(1)	*f*	1	0·0465 (48)	0·5016 (28)	0·4705 (44)	0·035 (5)					
H(2)	*f*	1	0·6183 (19)	0·5361 (7)	0·2051 (17)	0·018 (3)					
H(3)	*f*	1	0·4163 (20)	0·6035 (8)	0·0593 (15)	0·020 (3)					
H(4)	*f*	1	−0·0543 (18)	0·1918 (8)	−0·6353 (14)	0·018 (3)					
H(5)	*e*	*m*	−0·0610 (28)	0·2500	−0·4341 (22)	0·018 (3)					
H(6)	*f*	1	−0·4832 (20)	0·3089 (8)	−0·5079 (15)	0·023 (3)					
H(7)	*e*	*m*	−0·4697 (29)	0·2500	−0·7300 (23)	0·023 (4)					
H(*W*)	*f*	1	0·1257 (22)	0·1960 (10)	0·9361 (19)	0·038 (3)					
DIMAHS											
O(1)	*f*	*m*	0·00000	0·13466 (6)	0·53880 (3)	0·03024 (28)	0·00584 (13)	0·00858 (10)	0·00000	0·00000	0·00026 (15)
O(2)	*f*	*m*	0·00000	0·25033 (6)	0·39650 (3)	0·02258 (23)	0·00838 (13)	0·00672 (10)	0·00000	0·00000	−0·00108 (15)
C(1)	*f*	*m*	0·00000	0·26020 (7)	0·48306 (4)	0·01132 (21)	0·00597 (16)	0·00806 (21)	0·00000	0·00000	−0·00055 (15)
C(2)	*f*	*m*	0·00000	0·42606 (7)	0·53257 (4)	0·01815 (26)	0·00574 (13)	0·00786 (21)	0·00000	0·00000	−0·00102 (15)
N	*c*	*m2m*	0·00000	0·02452 (10)	0·25000	0·01482 (35)	0·00947 (23)	0·00744 (21)	0·00000	0·00000	0·00000
C(3)	*g*	*m*	0·18234 (14)	−0·07702 (10)	0·25000	0·01715 (28)	0·02137 (26)	0·01137 (21)	0·00599 (29)	0·00000	0·00000
H(1)	*h*	1	0·0408 (40)	0·0516 (25)	0·5083 (20)	0·014 (8)					
H(2)	*h*	1	−0·1197 (12)	0·4254 (8)	0·5740 (6)	0·016 (3)					
H(3)	*f*	*m*	0·0000	0·0867 (15)	0·2998 (8)	0·025 (4)					
H(4)	*h*	1	0·1830 (16)	−0·1464 (9)	0·1938 (5)	0·023 (3)					
H(5)	*g*	*m*	0·2965 (20)	−0·0038 (14)	0·2500	0·025 (3)					

Table 3. *Geometry of some hydrogen succinate salts*

Cation	Space group	*Z*	Site symmetry of anion	O(1)—C(1)—C(2)—C(2′) (°)	O(1)—O(1′) (Å)	Reference
$CH_3NH_3^+$	*P*2$_1$/*m*	2	$\bar{1}$	179	2·4448 (5)	
$(CH_3)_2NH_2^+$	*Cmcm*	4	*2/m*	180	2·4395 (7)	
Cs^+	*P*2$_1$/*m*	2	$\bar{1}$	177	2·41 (4)	McAdam & Speakman (1971)
Li^+	*C*2/*c*	4	$\bar{1}$	178	2·478 (1)	Küppers (1982)
K^+	*C*2/*c*	4	$\bar{1}$	180	2·446 (4)	McAdam, Currie & Speakman (1971)

positional parameters, displacement parameters, Wyckoff letters and site symmetries are listed in Table 2.*

Discussion. In the two structures both cation and anion are found on crystallographic symmetry elements. Methylammonium hydrogen succinate monohydrate has the O atom of the water molecule and the nitrogen and carbon from the methylammonium cation in the crystallographic mirror plane $y = \frac{1}{4}$; in this structure the hydrogen succinate ion contains a centre of inversion $(\frac{1}{2},\frac{1}{2},0)$. A comparison with other hydrogen succinate salts revealed that MAHS is isostructural with the caesium salt (McAdam & Speakman, 1971). In the structure of dimethylammonium hydrogen succinate the site symmetry of the N atom is *m2m*, which implies that the attached methyl groups are related by crystallographic mirror planes. In DIMAHS the hydrogen succinate ion is bound by higher crystallographic symmetry than in the MAHS structure. It has *2/m* symmetry (Wyckoff site *b*) which causes all the non-H atoms to be in a crystallographic mirror plane $x = 0$.

It appears to be a general trend that salts of the hydrogen succinate ion form crystals where the molecular symmetry of the anion is bound by crystallographic symmetry. Table 3 contains structural information about hydrogen succinate salts of small monovalent cations. In all these structures the crystallographic site symmetry of the anion is either $\bar{1}$ or *2/m*.

In the DIMAHS structure the site symmetry of the anion constrains the relative orientation of the two planar C—COO groups. The conformation of the hydrogen succinate ion can be illustrated by the torsion angle O(1)—C(1)—C(2)—C(2′) (the labelling is according to Fig. 1). The hydrogen succinate ion is found in a *trans* conformation in all the salts listed in Table 3, the torsion angle being virtually 180° also in structures where it is not implied by the crystallographic symmetry. The cations are shown in Fig. 2.

The hydrogen succinate ions are linked by short hydrogen bonds which form infinite chains in the structures. In contrast to the equivalent hydrogen

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* Lists of observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53531 (46 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

tartrate salts (Larsen, Andersen & Dam, 1990), the chains of hydrogen succinate ions are not parallel to any crystallographic unit-cell axis. The short hydrogen bonds that connect the hydrogen succinate ions are of the same magnitude (~2·44 Å) in the investigated salts. Owing to crystallographic symmetry they are symmetric. The lengths of the short hydrogen bonds in the hydrogen tartrate structures are in the same range but clearly asymmetric (Larsen *et al.*, 1990). The present structure determinations show that the symmetric hydrogen bond is best described by two half-populated H atoms placed on each side of the inversion centre. A similar observation was made by Küppers (1982) in the structure of the equivalent lithium salt. McAdam, Currie & Speakman (1971) investigated the potassium salt by both X-ray and neutron diffraction methods; however, their results were not of an accuracy that makes it possible to discriminate between the two situations where the H atom is placed on the crystallographic inversion centre or distributed between two sites as indicated in the present structures.

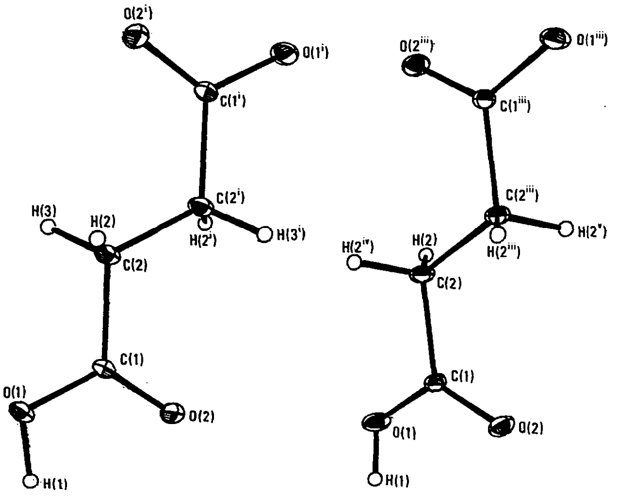


Fig. 1. *ORTEP* drawings of the hydrogen succinate ions found in MAHS (left) and DIMAHS (right) illustrating the atom-numbering scheme. The thermal ellipsoids enclose 50% probability, and the H atoms are drawn as spheres: (i) $1-x$, $1-y$, $-z$; (ii) x, $-y-\frac{1}{2}$, z; (iii) $-x$, $1-y$, $1-z$; (iv) $-x$, y, z; (v) x, $1-y$, $1-z$; (vi) x, y, $\frac{1}{2}-z$; (vii) $-x$, y, $\frac{1}{2}-z$.

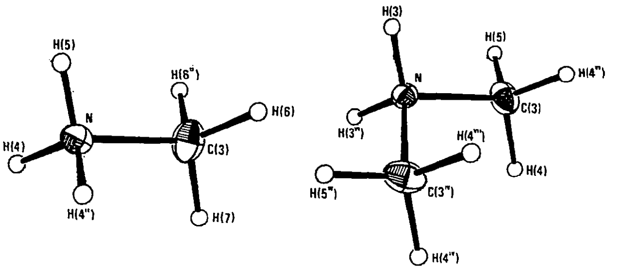
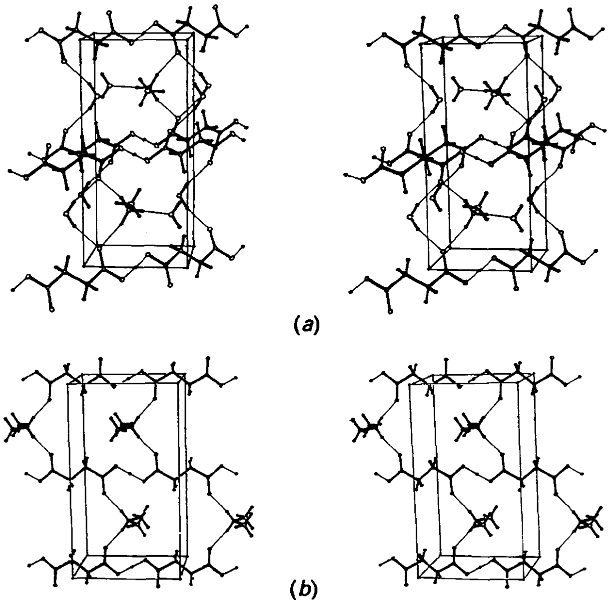


Fig. 2. *ORTEP* drawings of the two cations in MAHS (left) and DIMAHS (right) made as described in Fig. 1.

IR spectra for both compounds show the type (ii) spectrum of Hadži (1965) characteristic for type *A* and type A_2 salts, and this is consistent with the fact that the hydrogen bonds are crystallographically symmetric.

The packings in the MAHS and DIMAHS structures are illustrated by the stereo pairs shown in Fig. 3. The short O(1)—O(1′) hydrogen bonds are in the *ac* plane in the MAHS structure and almost parallel to the *b* axis in the DIMAHS structure. As shown in Table 4 there are hydrogen bonds between the cations and O(2), the oxygen atom which is not involved in the short hydrogen bond. This interaction is slightly stronger in DIMAHS where the N—O(2) distance is 2·7747 (7) compared to 2·8670 (5) Å in MAHS. The difference in N—O distance is probably related to the difference in character: N with three and two H atoms in MAHS and DIMAHS, respectively. Fig. 3(*b*) shows how these hydrogen bonds connect the chains of hydrogen succinate ions in DIMAHS. In MAHS the chains are linked by methylammonium ions and water molecules, and these two entities are also connected by a hydrogen bond. The difference in hydrogen bonding is reflected in the geometry of the hydrogen succinate ion. As observed in Table 5 the only significant difference between the two structures is found in the C(1)—O(2) distance, which is slightly longer in MAHS than in DIMAHS [1·2443 (6)


(*a*)

(*b*)

Fig. 3. Stereo drawings of (*a*) MAHS and (*b*) DIMAHS seen along the *a* axis. In MAHS the *b* axis is vertical and the *c* axis horizontal, while in DIMAHS the *b* axis is horizontal and the *c* axis vertical.

Table 4. *Hydrogen-bond geometry* (Å, °)

A = Donor atom, *B* = acceptor atom.

A—H···B	A···B	A—H	H···B	A—H···B
MAHS				
O(1)—H(1)···O(1^{i})	2·4448 (5)	0·95 (3)	1·507 (27)	169·5 (34)
N—H(4)···O(2^{ii})	2·8670 (5)	0·92 (1)	1·968 (9)	164·2 (8)
N—H(5)···O(W^{iii})	2·7688 (9)	0·86 (1)	1·913 (13)	174·7 (14)
O(W)—H(W)···O(2)	2·8000 (5)	0·88 (1)	1·956 (12)	163·2 (11)
DIMAHS				
O(1)—H(1)···O(1^{iv})	2·4395 (7)	0·85 (2)	1·670 (22)	150·0 (26)
N—H(3)···O(2)	2·7747 (7)	0·88 (1)	1·911 (12)	171·5 (11)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $x, y, 1+z$; (iii) $x, y, z-1$; (iv) $-x, -y, 1-z$.

Table 5. *Bond lengths* (Å) *and angles* (°)

	MAHS	DIMAHS
Anion		
C(1)—O(1)	1·2866 (6)	1·2884 (8)
C(1)—O(2)	1·2443 (6)	1·2391 (8)
C(1)—C(2)	1·5130 (6)	1·5137 (8)
C(2)—C(2^{i})	1·5159 (6)	1·5132 (8)
O(1)—C(1)—O(2)	123·45 (4)	124·47 (6)
O(1)—C(1)—C(2)	114·45 (4)	113·98 (5)
O(2)—C(1)—C(2)	122·11 (4)	121·54 (5)
C(1)—C(2)—C(2^{i})	114·20 (4)	114·20 (5)
Cation		
N—C(3)	1·4837 (10)	1·4841 (10)
C(3)—N—C(3^{ii})		112·99 (7)

Symmetry codes: (i) $1-x, 1-y, -z$ (MAHS); (i) $-x, 1-y, 1-z$ (DIMAHS); (ii) $-x, y, z$.

compared to 1·2391 (8) Å]. It is likely that this difference is due to the fact that O(2) is involved in two hydrogen bonds in MAHS and only one in DIMAHS. As commonly observed the average C—O distance of each carboxyl group is nearly the same in MAHS and DIMAHS: 1·2655 and 1·2638 Å, respectively, from Table 5.

Both cations have also been investigated structurally as part of the charge-density studies of hydrogen oxalate ions performed by Thomas (1975) and Thomas & Pramatus (1975). The N—C bond lengths found in the present structure determinations are slightly larger, 1·484 (1) Å, than in the room-temperature studies of the hydrogen oxalate salts, 1·463 (3) Å (methylammonium) and 1·471 (2) and 1·476 (2) Å (dimethylammonium). As these bond lengths have not been corrected for thermal vibration, it is possible that the apparent discrepancy may be due to the effect of thermal vibration.

We thank Flemming Hansen who assisted in the experimental crystallographic work. The Danish Natural Science Research Council supported this research by financing the X-ray diffractometer (J.nr. 11-1837 to SL), and the Carlsberg Foundation provided financial support to NK.

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Acta Cryst. (1991). C**47**, 1009–1012

Structure of 1-{5-Fluoro-4-[(5-oxo-1,3-pentadienyl)amino]-2-pyrimidinyl}pyridinium Chloride Trihydrate

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(*Received* 20 *March* 1990; *accepted* 28 *August* 1990)

Abstract. $C_{14}H_{12}FN_4O^+.Cl^-.3H_2O$, $M_r = 360·8$, monoclinic, $P2_1/c$, $a = 7·2493$ (6), $b = 18·611$ (1), $c = 13·0710$ (6) Å, $\beta = 96·973$ (6)°, $V = 1750·5$ (2) Å^3, $Z = 4$, $D_x = 1·369$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 21·5$ cm^{-1}, $F(000) = 752$, $T = 290$ K, $R = 0·041$ for 2134 observed reflections. The aliphatic chain at N(4)

0108-2701/91/051009-04$03.00