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Ammonium Hydrogen 1-Malate

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Abstract. $(NH_4)^+(C_4H_5O_5)^-$. $P2_12_12_1$, $a = 7{\cdot}625$ (3), $b = 8{\cdot}106$ (4), $c = 10{\cdot}607$ (4) Å, $Z = 4$, $D_c = 1{\cdot}703$, $D_m = 1{\cdot}69$ g cm^{-3}, $\mu = 1{\cdot}53$ cm^{-1}. The conformation of the hydrogen malate is different from that found in the corresponding Ni^{2+} salt. This is due to complex formation in the latter compound between the Ni^{2+} ion and the alcoholic OH groups. In the crystal structure of the NH_4 salt all non-aliphatic hydrogen atoms participate in hydrogen bonding.

Introduction. The crystals of ammonium hydrogen malate were grown at room temperature by slow evaporation of a water solution. Reflections were measured on an Enraf–Nonius CAD-4 computer-automated four-circle diffractometer, using Mo $K\alpha$ radiation and a graphite monochromator ($2\theta = 12{\cdot}6°$). From the systematic extinctions the space group $P2_12_12_1$ was inferred. Intensities up to $\theta = 30°$ were measured. For time economy, 978 reflections were retained with $I > 2\sigma(I)$. No absorption or extinction corrections were applied. The structure was solved by direct methods using the *MULTAN* program of Germain, Main & Woolfson (1971). An *E* map with 196 terms showed all non-hydrogen atoms. After refinement of the related atomic parameters, a difference Fourier map revealed the positions of all hydrogen atoms.

The structure was refined by block-diagonal least-squares calculations, in which each reflection was given a weight based on the counting statistics. The final difference Fourier map showed no maxima above the level of 0·15 e Å^{-3}. The positional parameters resulting from this procedure are presented in Table 1.† The positional parameters given correspond to the absolute configuration (*S*) of the malate ion.

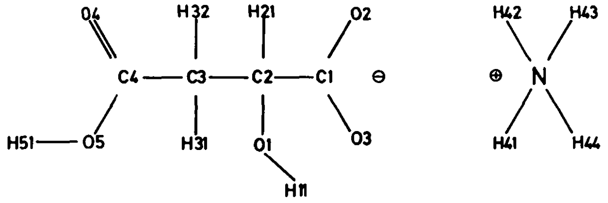

Fig. 1. Numbering of the atoms in ammonium hydrogen malate.

Table 1. *Atomic coordinates of ammonium hydrogen* 1-*malate*

x, *y* and *z* are given as fractional coordinates. The e.s.d. (in parentheses) refers to the last significant digit of the parameter.

	x	*y*	*z*
N	0·9260 (2)	0·8697 (2)	0·0357 (1)
C(1)	0·5446 (2)	0·8585 (3)	0·2587 (2)
C(2)	0·3496 (2)	0·9055 (3)	0·2672 (2)
C(3)	0·2513 (2)	0·7834 (3)	0·3496 (2)
C(4)	0·0708 (2)	0·8479 (3)	0·3786 (2)
O(1)	0·2681 (2)	0·9054 (2)	0·1476 (1)
O(2)	0·6321 (2)	0·8731 (2)	0·3586 (1)
O(3)	0·6032 (2)	0·8108 (2)	0·1564 (1)
O(4)	0·0431 (2)	0·9650 (2)	0·4455 (1)
O(5)	–0·0541 (2)	0·7620 (2)	0·3248 (1)
H(11)	0·334 (3)	0·951 (2)	0·091 (2)
H(21)	0·343 (2)	1·008 (2)	0·301 (1)
H(31)	0·246 (2)	0·676 (2)	0·310 (2)
H(32)	0·311 (2)	0·770 (2)	0·427 (2)
H(41)	0·895 (3)	1·001 (3)	–0·024 (2)
H(42)	0·835 (3)	0·838 (2)	0·075 (2)
H(43)	1·008 (2)	0·896 (3)	0·082 (2)
H(44)	0·960 (3)	0·781 (2)	–0·009 (2)
H(51)	–0·169 (3)	0·817 (2)	0·342 (2)

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33535 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

C(1)—O(2)	1·258 (2)	C(4)—O(4)	1·204 (2)	O(1)—H(11)	0·87 (3)
C(1)—O(3)	1·236 (2)	C(4)—O(5)	1·310 (2)	O(5)—H(51)	1·00 (3)
C(1)—C(2)	1·537 (3)	N—H(41)	1·27 (3)	C(2)—H(21)	0·91 (2)
C(2)—C(3)	1·518 (3)	N—H(42)	0·85 (3)	C(3)—H(31)	0·97 (2)
C(2)—O(1)	1·413 (2)	N—H(43)	0·82 (3)	C(3)—H(32)	0·95 (2)
C(3)—C(4)	1·503 (3)	N—H(44)	0·90 (3)		

O(2)—C(1)—O(3)	125·3 (2)	O(4)—C(4)—O(5)	123·2 (2)	O(1)—C(2)—H(21)	109·0 (1·7)
O(2)—C(1)—C(2)	116·1 (2)	C(1)—C(2)—H(21)	108·0 (1·3)	H(31)—C(3)—H(32)	107·2 (1·9)
O(3)—C(1)—C(2)	118·6 (2)	C(3)—C(2)—H(21)	110·1 (1·3)	H(41)—N—H(42)	110·0 (2·2)
C(1)—C(2)—C(3)	110·5 (2)	C(2)—O(1)—H(11)	111·7 (1·3)	H(41)—N—H(43)	103·3 (2·2)
C(1)—C(2)—O(1)	111·8 (2)	C(2)—C(3)—H(31)	110·8 (1·3)	H(41)—N—H(44)	116·9 (2·2)
C(3)—C(2)—O(1)	107·4 (2)	C(2)—C(3)—H(32)	109·4 (1·3)	H(42)—N—H(44)	104·8 (2·2)
C(2)—C(3)—C(4)	110·1 (2)	C(4)—C(3)—H(31)	111·5 (1·3)	H(42)—N—H(43)	113·7 (2·2)
C(3)—C(4)—O(4)	123·7 (2)	C(4)—C(3)—H(32)	107·7 (1·3)	H(43)—N—H(44)	107·4 (2·2)
C(3)—C(4)—O(5)	113·0 (2)	C(4)—O(5)—H(51)	108·7 (1·7)		

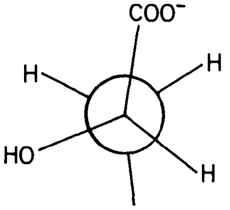

Fig. 2. Newman projection along C(2)—C(3).

The final *R* index thus enumerated is 0·027. The molecular structure is shown in Fig. 1 and Table 2 gives bond lengths and angles.

Discussion. This structure determination was undertaken within the framework of our research on the conformation of malate ions in an attempt to correlate their conformational aspects with *inter alia* the chemical properties of the counter-ion. No information on hydrogen malates is available in the literature. The present compound seemed very suitable to reveal the conformation of the 'normal' hydrogen malate ion, since the NH_4^+ ion does not allow conformational disturbance of complex formation.

In Fig. 2 a Newman projection is given along the central C(2)—C(3) bond of the hydrogen malate ion. The observed conformation agrees well with preliminary CNDO calculations on the isolated ion.

Table 3 gives the relevant torsion angles for the calculated and observed structure as well as for the Ni^{2+} hydrogen malate (Lenstra, unpublished results) in which complexation between the Ni ion and the alcoholic OH groups occurs.

The stoichiometric unit, $NH_4^+C_4H_5O_5^-$, forms a three-dimensional network, in which, apart from Coulomb interactions, hydrogen bonds provide a stabilizing factor. All non-aliphatic hydrogen atoms participate in the hydrogen-bonding scheme. This is illustrated in Fig. 3 and Table 4. In accordance with generally accepted ideas on the strength of hydrogen bonds (*e.g.* Pimentel & McClellan, 1960) the bridge O(5)—H(51)···O(2),

Table 3. *Torsion angles in hydrogen malate ions* (°)

Torsion angle	Ni^{2+} (Lenstra, unpublished results)	NH_4^+ this paper	CNDO calculations on the free ion
⁻OOC—C(2)—C(3)—COOH	179·8	168·7	165
HO—C(2)—C(3)—COOH	60·5	69·0	75

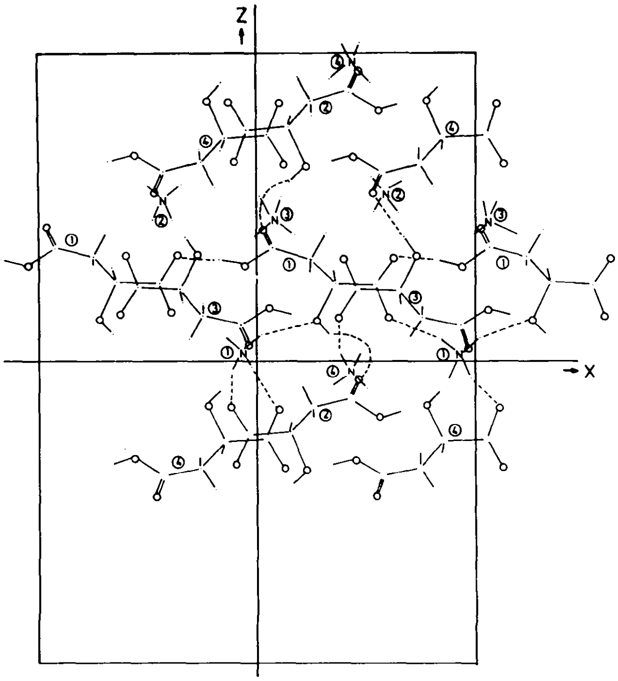

Fig. 3. Packing of ammonium hydrogen malate projected along the *y* axis. Some of the hydrogen bonds are indicated by dashed lines. Molecule (1) is at x,y,z; (2) is at $\frac{1}{2}-x, \bar{y}, \frac{1}{2}+z$; (3) is at $x, \frac{1}{2}+y, \frac{1}{2}-z$; (4) is at $\frac{1}{2}+x, \frac{1}{2}-y, \bar{z}$.

Table 4. *Hydrogen bridges found in ammonium hydrogen malate*

See Fig. 3 for the placing of the molecules.

D–H···*A*	Molecule	*D*···*A*	H···*A*	*D*–H···*A*
O(5)–H(51)···O(2)	(1)	2·58 Å	1·59 Å	169·2°
O(1)–H(11)···O(4)	(2)	2·79	1·93	171·0
N–H(41)···O(2)	(2)	2·84	1·62	160·8
N–H(42)···O(3)	(1)	2·81	1·98	167·3
N–H(43)···O(1)	(1)	2·88	2·10	157·7
N–H(44)···O(3)	(4)	2·85	2·04	148·2

involving the most acidic H atom is 'short', whereas the others are 'long'.

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S-Nitroso-*N*-acetyl-DL-penicillamine

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Abstract. $C_7H_{12}N_2O_4S$, 2-acetamido-3-methyl-3-thionitrosobutanoic acid, space group $P2_1/c$, a = 6·9840 (11), b = 12·698 (2), c = 12·353 (2) Å, β = 107·80 (1)° at 295 K (λ = 1·54051 Å), Z = 4, D_x = 1·403, D_m = 1·398 g cm^{-3}, M_r = 220·25. Thionitrite bond distances [C–S 1·833 (1), S–N 1·763 (2), N–O 1·199 (2) Å] and angles [C–S–N 100·80 (7), S–N–O 113·99 (11)°] are consistent with its description as C–S–N=O. Each thionitrite molecule participates in four intermolecular H bonds which form two-dimensional networks approximately parallel to the *bc* plane.

Introduction. Prismatic crystals formed when water was allowed to diffuse into a saturated solution of *N*-acetyl-DL-penicillamine thionitrite in methanol. The apparent color of the crystals varies with increasing crystal thickness from pale green to reddish in transmitted sunlight. Precession photographs show $2/m$ Laue symmetry with systematic absences for $h0l$ (l odd) and $0k0$ (k odd), establishing the space group as $P2_1/c$. Crystal density was determined by flotation in a mixture of chloroform and *n*-heptane. For data collection a crystal cut perpendicular to [010] (dimensions 0·53 × 0·66 × 0·47 mm, volume 0·168 mm^3) was mounted on a four-circle automated diffractometer with φ approximately parallel to [010], the longest dimension. Resolved Cu $K\alpha_1$ peaks for 28 reflections in the range $83 < 2\theta < 89°$ gave the cell constants (least-squares fit to the 2θ, ω, χ values). Intensity data for 8104 reflections were measured by θ–2θ step scans (0·03° 2θ per step, 1 s counts with 4 s background counts, 0·001 in Nb incident beam filter and Mo $K\alpha$ radiation). The data set included $\pm h,k,\pm l$ for $30 < 2\theta < 60°$ and $\pm h,\pm k,\pm l$ for $0 < 2\theta < 30°$ with Y–Zr balanced filters for $0 < 2\theta < 12°$. As provided in the diffractometer control program (Lenhert, 1975), coincidence losses were corrected, and weak reflections rescanned a maximum of two times with 20 s background counts until $I/\sigma(I) > 15$. Total X-ray exposure (50 kV, 12 mA, take-off angle 2·7°, focal spot 15 × 0·75 mm, 24 cm tube-to-crystal distance) was 424 h during which time three monitor reflections showed an intensity decrease of 1·9%. The computer program *ORABS* (Wehe, Busing & Levy, 1962) was used to calculate absorption corrections (μ = 2·890 cm^{-1}, 5 × 5 × 5 Gaussian grid, maximum, minimum and average transmission factors; 0·878, 0·857, 0·871). Symmetry-related reflections were averaged to give 3551 F_o values (92 with $F_o < 0$ were set to zero). A more complete description of the data collection and reduction procedures is given by Miller, Lenhert & Joesten (1972).