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Reinvestigation of the Structure of Ammonium Hydrogen D-Tartrate

BY A. L. SPEK

Vakgroep Algemene Chemie, Afdeling Kristal- en Structuurchemie, University of Utrecht, Padualaan 8, 3584 *CH Utrecht, The Netherlands*

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Abstract. $NH_4^+.C_4H_5O_6^-$, $M_r = 167{\cdot}1$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}653\ (1)$, $b = 11{\cdot}072\ (1)$, $c = 7{\cdot}849\ (1)$ Å, $V = 665{\cdot}1\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}669$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}7$ cm^{-1}, $F(000) = 352$, $T = 295$ K, $R = 0{\cdot}029$ for 1043 observed reflections with $I > 2{\cdot}5\sigma(I)$. A more accurate and detailed structure of ammonium hydrogen D-tartrate is reported that includes the positions of the H atoms of the NH_4^+ cation that were not revealed in the previous investigation. All H atoms of NH_4^+ are involved in hydrogen bonding to O atoms of the distorted square-antiprism coordination polyhedron. The crystal structure consists of layers of hydrogen-bonded tartrate anions perpendicular to [010], separated by layers of NH_4^+ cations that are hydrogen-bonded to both sides resulting in a three-dimensional network.

Experimental. Diffraction data were collected on an Enraf–Nonius CAD-4*F* diffractometer using Zr-filtered Mo $K\alpha$ radiation for a spherical crystal (radius 0·2 mm; colourless; mounted on top of a glass fibre) that is in use as a CAD-4 test crystal. A redundant set of 7770 reflections (h −10:10; k −14:14; l −10:10; $\theta < 27{\cdot}5°$) was scanned in the ω-scan mode [$\Delta\omega = (0{\cdot}40 + 0{\cdot}35\tan\theta)°$; horizontal and vertical aperture 3 × 2 mm; crystal-to-detector distance 173 mm; max. time per reflection 60 s]. Eight reference reflections (±2, ±2, ±4) remained constant during the 103 h of exposure time. The observed values of $\sigma(I)/I$ were only slightly larger than the preset value 0·006 (maximum value 0·008). Cell dimensions were calculated from the setting angles of 18 reflections ($11 < \theta < 16°$) using an appropriate average of the setting angles for four alternative reflection settings. The reflection data were corrected for Lp effects and averaged into a unique set of 1091 reflections ($R_{\text{int}} = 0{\cdot}026$). Full-matrix least-squares refinement on F with a locally modified implementation of *SHELX*76 (Sheldrick, 1976) on an Eclipse S/230 mini-computer was started from the published coordinates for the non-H atoms. All H atoms were located in a difference Fourier map including those on the N atom that could not be identified in the previous study and their positions refined. Convergence was reached at $R = 0{\cdot}029$ [$wR = 0{\cdot}032$; $S = 0{\cdot}32$; $(\Delta/\sigma)_{\text{av}} = 0{\cdot}05$, $(\Delta/\sigma)_{\max} = 0{\cdot}04$] for 1043 reflections with $I > 2{\cdot}5\sigma(I)$, 130 parameters and weights based on counting statistics. An empirical correction for extinction was applied using the formula $F(\text{corr.}) = F(1 - 0{\cdot}0001g|F|^2/\sin\theta)$ with $g = 0{\cdot}0485\ (7)$. Maximum and minimum residual density in the final electron density difference map were 0·23 and −0·16 e Å^{-3}. All atoms were refined with anisotropic temperature factors except for the H atoms that were refined with one common isotropic temperature factor for those attached to O and one for those on N.

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Table 1. *Final coordinates and isotropic thermal parameters*

	x	y	z	U_{eq}*/U_{iso}(Å^2)
O(1)	0·7946 (2)	0·3836 (1)	0·3709 (2)	0·0297 (3)
O(2)	0·7677 (2)	0·5758 (1)	0·4516 (2)	0·0323 (3)
O(3)	0·4388 (2)	0·5899 (1)	0·3273 (2)	0·0241 (3)
O(4)	0·4328 (2)	0·4174 (1)	0·6117 (1)	0·0268 (3)
O(5)	0·2064 (2)	0·3600 (1)	0·2198 (2)	0·0271 (3)
O(6)	0·1085 (2)	0·3993 (1)	0·4807 (2)	0·0279 (3)
C(1)	0·7076 (2)	0·4848 (2)	0·3900 (2)	0·0205 (4)
C(2)	0·5210 (2)	0·4748 (2)	0·3259 (2)	0·0183 (4)
C(3)	0·4185 (2)	0·3863 (2)	0·4380 (2)	0·0190 (4)
C(4)	0·2292 (2)	0·3809 (1)	0·3741 (2)	0·0190 (4)
H(1)	0·528 (1)	0·4435 (9)	0·210 (1)	0·042 (1)
H(2)	0·467 (1)	0·308 (1)	0·427 (1)	0·042 (1)
H(3)	0·409 (1)	0·6124 (9)	0·214 (1)	0·042 (1)
H(4)	0·381 (1)	0·4930 (9)	0·628 (1)	0·042 (1)
H(5)	0·905 (1)	0·3908 (9)	0·407 (1)	0·042 (1)
N	0·9155 (2)	0·2170 (1)	0·0717 (2)	0·0291 (4)
H(6)	0·834 (1)	0·177 (1)	0·000 (1)	0·140 (1)
H(7)	0·936 (1)	0·281 (1)	0·049 (1)	0·140 (1)
H(8)	0·999 (1)	0·168 (1)	0·084 (1)	0·140 (1)
H(9)	0·871 (1)	0·215 (1)	0·162 (1)	0·140 (1)

$^*U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Relevant data on the geometry of ammonium hydrogen D-tartrate*

(*a*) Bond distances (Å)

O(1)–C(1)	1·312 (2)	C(1)–C(2)	1·518 (2)
O(2)–C(1)	1·209 (2)	C(2)–C(3)	1·533 (2)
O(3)–C(2)	1·421 (2)	C(3)–C(4)	1·534 (2)
O(4)–C(3)	1·410 (2)	C(2)–H(1)	0·976 (8)
O(5)–C(4)	1·246 (2)	C(3)–H(2)	0·951 (10)
O(6)–C(4)	1·263 (2)		

(*b*) Bond angles (°)

O(1)–C(1)–C(2)	112·2 (1)	O(5)–C(4)–C(3)	117·3 (1)
O(2)–C(1)–C(2)	123·5 (1)	C(1)–O(1)–H(5)	111·5 (7)
O(1)–C(1)–O(2)	124·3 (1)	C(2)–O(3)–H(3)	109·4 (6)
C(1)–C(2)–C(3)	109·7 (1)	C(3)–O(4)–H(4)	108·7 (5)
O(3)–C(2)–C(1)	110·4 (1)	H(7)–N(1)–H(9)	109·4 (9)
O(3)–C(2)–C(3)	110·0 (1)	H(6)–N(1)–H(9)	103·6 (8)
O(4)–C(3)–C(2)	111·1 (1)	H(7)–N(1)–H(8)	118·5 (8)
C(2)–C(3)–C(4)	108·7 (1)	H(8)–N(1)–H(9)	101·8 (9)
O(4)–C(3)–C(4)	113·5 (1)	H(6)–N(1)–H(7)	116·1 (9)
O(6)–C(4)–C(3)	117·9 (1)	H(6)–N(1)–H(8)	105·5 (9)
O(5)–C(4)–O(6)	124·9 (1)		

(*c*) Torsion angles (°)

C(1)–C(2)–C(3)–C(4)	178·3 (1)	O(2)–C(1)–C(2)–O(3)	8·0 (2)
O(6)–C(4)–C(3)–O(4)	−2·7 (2)	O(3)–C(2)–C(3)–O(4)	−68·9 (2)

(*d*) Ammonium coordination (Å)

N···O(1)	3·126 (2)	N···O(3^{ii})	3·082 (2)
N···O(3^{iii})	3·156 (2)	N···O(5^{vi})	2·919 (2)
N···O(5^{v})	2·970 (2)	N···O(2^{ii})	2·849 (2)
N···O(2^{i})	2·891 (2)	N···O(4^{iv})	2·900 (2)

(*e*) Hydrogen bonding (Å, °)

D–H···*A*	*D*···*A*	*D*–H	H···*A*	∠*D*–H···*A*
O(3)–H(3)···O(6^{vii})	2·746 (2)	0·951 (8)	1·841 (8)	158·3 (9)
O(4)–H(4)···O(5^{viii})	2·816 (2)	0·93 (1)	1·90 (1)	165·3 (7)
O(1)–H(5)···O(6^{v})	2·558 (2)	0·895 (8)	1·664 (8)	177·3 (9)
N–H(6)···O(5^{vi})	2·919 (2)	0·95 (1)	2·025 (8)	156·4 (8)
N–H(7)···O(2^{ii})	2·849 (2)	0·74 (1)	2·351 (9)	125·3 (7)
N–H(7)···O(3^{ii})	3·082 (2)	0·74 (1)	2·448 (9)	143·7 (8)
N–H(8)···O(2^{i})	2·891 (2)	0·84 (1)	2·076 (9)	162·2 (8)
N–H(9)···O(4^{iv})	2·900 (2)	0·787 (8)	2·351 (9)	127·7 (9)
N–H(9)···O(1)	3·126 (2)	0·787 (8)	2·55 (1)	131 (1)

Symmetry code: (i) $2-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$; (ii) $\frac{3}{2}-x$, $1-y$, $-\frac{1}{2}+z$; (iii) $1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$; (iv) $\frac{1}{2}+x$, $\frac{1}{2}-y$, $1-z$; (v) $1+x$, y, z; (vi) $\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$; (vii) $\frac{1}{2}-x$, $1-y$, $-\frac{1}{2}+z$; (viii) $\frac{1}{2}-x$, $1-y$, $\frac{1}{2}+z$.

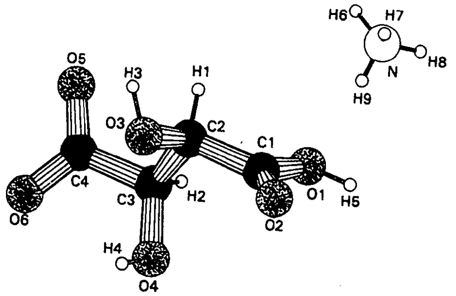

Fig. 1. A perspective view of the molecule with the numbering scheme.

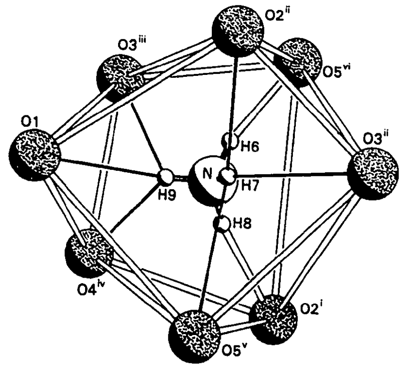

Fig. 2. Pseudo square-antiprism coordination polyhedron of the NH_4^+ cation. O···H contacts <2·75 Å and ∠N–H···O > 110° are drawn. The symmetry code is the same as in Table 2.

Scattering factors were taken from Cromer & Mann (1968). Final parameter values are given in Table 1.* Fig. 1 shows the molecule and Fig. 2 the environment of the NH_4^+ cation. Table 2 lists selected geometrical data.

Related literature. The crystal structure of ammonium hydrogen D-tartrate has been investigated previously in our laboratory (van Bommel & Bijvoet, 1958). The carboxyl geometries reported here are found to be more in agreement with average COO^- and COOH geometries as given by Borthwick (1980).

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* Lists of observed and calculated structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43913 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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