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Sodium Hydrogen L-Tartrate

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Abstract

Crystals of sodium hydrogen L-tartrate, $Na^+.C_4H_5O_6^-$, were grown from an aqueous solution at 393 K in a closed vessel. An asymmetric O—H···O hydrogen bond is formed between the hydrogen L-tartrate anions [O···O 2.476 (3) Å]. The H atom involved in the hydrogen bond is disordered. The sodium ion is coordinated by six O atoms, in contrast to the eight atoms about other alkali metal ions in their hydrogen L-tartrates.

Comment

The crystals of alkali metal hydrogen L-tartrates, $M^+.C_4H_5O_6^-$ (M^+ = K^+, Rb^+, Cs^+), belong to space group $P2_12_12_1$ and are isostructural with one another (Buschmann & Luger, 1985; Templeton & Templeton, 1978, 1989). The corresponding sodium salt crystallizes as a monohydrate from an aqueous solution at room temperature (Bott, Sagatys, Lynch, Smith & Kennard, 1993; Kubozono, Hirano, Nagasawa, Maeda & Kashino, 1993). Packing considerations showed that sodium hydrogen L-tartrate, (I), may not crystallize as

an anhydrate which is isostructural with the other alkali metal salts under ambient conditions (Kubozono *et al.*, 1993).

(I)
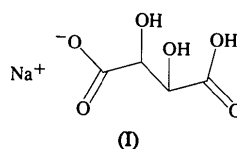
The crystals of sodium hydrogen L-tartrate (Fig. 1) obtained at 393 K belong to space group $P2_1$. The chain of four C atoms in the anion has the usual planar zigzag conformation [C1—C2—C3—C4 177.5 (2)°] and lies nearly on (101). A disordered O—H···O hydrogen bond is formed between the anions related by a c translation. This disordering was confirmed by careful examination of both the $\Delta\rho$ maps and the results of the least-squares refinement. Bond distances around the C1 and C4 atoms indicate that C1 has a larger carboxyl character than C4, as is also suggested by the occupancy factor of the disordered H atom. The O···O distance is shorter than those found in the corresponding hydrogen bonds in the monohydrate of (I) [2.533 (3) Å; Kubozono *et al.*, 1993] and in the acid salts of the other alkali metals [2.531 (1), 2.559 (3) and 2.575 (3) Å for K, Rb and Cs, respectively; Buschmann & Luger, 1985; Templeton & Templeton, 1978, 1989]. The OH groups attached to atoms C2 and C3 donate a hydrogen bond to the O6 atom of the ion
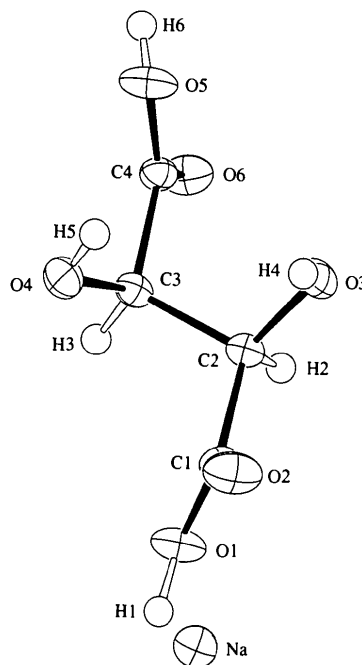

Fig. 1. A displacement ellipsoid plot of (I) with the atomic numbering scheme. Ellipsoids of 50% probability are shown for non-H atoms and H atoms are represented as spheres equivalent to B = 1.0 Å². Atoms H1 and H6 are disordered, their occupancy factors being 0.58 (2) and 0.42 (2), respectively.

related by an **a** translation to form a hydrogen-bond network of the anions on (020) (Table 3).

The Na$^+$ ion is coordinated by four O atoms of the carboxyl and carboxylate groups of the molecules lying on (020), and by two hydroxylic O atoms of the molecules lying on (010) (Fig. 2). The Na$^+$ ion is thus coordinated by six O atoms forming a distorted octahedron. On the other hand, in the acid salts of other alkali metals and the monohydrate of (I), the metal ion is coordinated by eight O atoms forming a distorted square antiprism (Kubozono *et al.*, 1993). This difference in the coordination scheme can be explained in terms of contacts between O atoms in the coordination polyhedra. The shortest contacts are 3.168 (2), 3.344 (3) and 3.379 (3) Å in the K, Rb and Cs salts, respectively. If (I) were isostructural with the other alkali metal salts, the shortest contact between O atoms for (I) should be 2.81 Å, assuming that the average *M*—O distance is shortened by the same amount, as found in the change from the K salt to the monohydrate of (I). The contact distance obtained is much shorter than the sum of the van der Waals radii of the O atoms. In the coordination polyhedron of (I), the shortest contact is found to be 2.982 (3) Å between atoms O3 and O5(2 − *x*, $\frac{1}{2}$ + *y*, 3 − *z*).

There is no significant difference in the C2—O3 and C3—O4 bond lengths in (I), in contrast with the other alkali metal salts in which the differences amount to between 0.012 and 0.028 Å. In (I), both the O3 and O4 atoms are involved in one *M*$^+$···O contact, namely, Na$^+$···O3(1 − *x*, *y* − $\frac{1}{2}$, 2 − *z*) 2.461 (2) and Na$^+$···O4(1 − *x*, $\frac{1}{2}$ + *y*, 2 − *z*) 2.347 (2) Å. In the other salts, however, O3 has two and O4 one *M*$^+$···O contact, where a larger partial electron transfer from O3 to the *M*$^+$ ions rather than from O4 may occur.

Experimental

Crystal data

Na$^+$.C$_4$H$_5$O$_6^-$	Mo *K*α radiation
M_r = 172.07	λ = 0.71073 Å
Monoclinic	Cell parameters from 25
$P2_1$	reflections
a = 5.251 (4) Å	θ = 9.5–11.5°
b = 7.782 (4) Å	μ = 0.237 mm^{-1}
c = 7.592 (5) Å	*T* = 295 K
β = 101.46 (6)°	Prismatic
V = 304.1 (4) Å^3	0.58 × 0.28 × 0.13 mm
Z = 2	Colourless
D_x = 1.880 Mg m^{-3}	
D_m not measured	

Data collection

Rigaku AFC-5*R* diffractometer	721 observed reflections [*I* > 3σ(*I*)]
ω/2θ scans	R_{int} = 0.007
Absorption correction: empirical *via* ψ scan (North, Phillips & Mathews, 1968)	θ_{max} = 27.5°
	h = 0 → 6
	k = 0 → 10
	l = −9 → 9
T_{min} = 0.84, T_{max} = 1.00	3 standard reflections
820 measured reflections	monitored every 97
745 independent reflections	reflections intensity decay: 0.59%

Refinement

|	
Refinement on *F*	Atomic scattering factors
R = 0.026	from *International Tables*
wR = 0.033	*for X-ray Crystallography*
S = 2.18	(1974, Vol. IV, Table
721 reflections	2.3.1)
120 parameters	Absolute configuration: the
w = 1/σ^2(*F*)	absolute configuration was
(Δ/σ)$_{max}$ = 0.51	assigned to agree with
$\Delta\rho_{max}$ = 0.26 e Å^{-3}	the known chirality at C2
$\Delta\rho_{min}$ = −0.18 e Å^{-3}	and C3 arising from the
Extinction correction:	L-tartaric acid precursor
I_{corr} = I_o(1 + *gI*$_c$)	
Extinction coefficient:	
g = 1.65 × 10^{-4}	
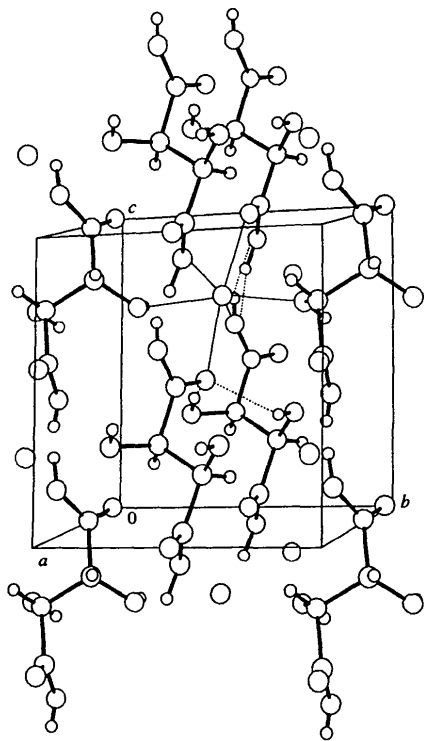
Fig. 2. A perspective view of the molecular packing showing both disordered H atoms. Hydrogen bonds are shown by broken lines and coordination around the Na$^+$ ion is shown by thin solid lines.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($Å^2$)*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Na	0.1407 (2)	0.4212	0.7297 (1)	1.88 (3)
O1	0.5709 (3)	0.3953 (3)	0.8863 (2)	2.15 (6)
O2	0.9773 (3)	0.4782 (3)	0.9961 (2)	2.56 (7)
O3	0.8414 (4)	0.6065 (3)	1.2971 (2)	1.90 (7)
O4	0.8440 (3)	0.2211 (3)	1.2988 (2)	1.87 (7)
O5	0.7160 (3)	0.3503 (3)	1.5988 (2)	2.27 (6)
O6	0.3123 (3)	0.4087 (3)	1.4573 (2)	2.09 (6)
C1	0.7546 (4)	0.4566 (3)	1.0118 (2)	1.51 (7)
C2	0.6628 (4)	0.4976 (4)	1.1857 (3)	1.47 (7)
C3	0.6213 (4)	0.3275 (4)	1.2797 (3)	1.43 (7)
C4	0.5404 (4)	0.3654 (3)	1.4588 (3)	1.43 (7)

Table 2. *Selected geometric parameters ($Å, °$)*

O1—C1	1.304 (3)	O6—C4	1.242 (3)
O2—C1	1.210 (3)	C1—C2	1.526 (3)
O3—C2	1.414 (3)	C2—C3	1.540 (3)
O4—C3	1.417 (3)	C3—C4	1.532 (3)
O5—C4	1.266 (3)		
O1—C1—O2	124.6 (2)	O4—C3—C2	111.1 (2)
O1—C1—C2	113.2 (2)	O4—C3—C4	113.0 (2)
O2—C1—C2	122.2 (2)	C2—C3—C4	109.6 (2)
O3—C2—C1	110.9 (2)	O5—C4—O6	125.0 (2)
O3—C2—C3	112.1 (2)	O5—C4—C3	116.3 (2)
C1—C2—C3	108.6 (2)	O6—C4—C3	118.7 (2)

Table 3. *Hydrogen-bonding geometry ($Å, °$)*

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5^i	0.90 (6)	1.59 (6)	2.476 (3)	169 (9)
O5—H6···O1^{ii}	0.71 (8)	1.81 (8)	2.476 (3)	157 (9)
O3—H4···O6^{iii}	0.75 (4)	2.33 (5)	2.961 (3)	142 (4)
O4—H5···O6^{iv}	0.82 (4)	2.11 (4)	2.907 (3)	161 (3)

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

H atoms were located from a difference Fourier map and the coordinates and displacement parameters were refined, except for the disordered H atoms between the O1···O5 hydrogen bond, H1 and H6, whose B_{iso} values were fixed to the average value of B_{eq} of O1 and O5. The occupancy factors of the H atoms converged to 0.58 (2) and 0.42 (2) in the least-squares refinement. The final $\Delta\rho$ map showed no significant peaks around these H atoms, $\Delta\rho$ being at most $0.10\,e\,Å^{-3}$. All calculations were performed on a VAX3100 computer at the X-ray Laboratory of Okayama University, Japan.

Data collection: *MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1990). Cell refinement: *MSC/AFC Data Collection and Refinement Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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2-Carboxy-9-dihydroxymethyl-1,10-phenanthroline Complexes of Cobalt(II) and Copper(II)

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Abstract

Bis(9-dihydroxymethyl-1,10-phenanthroline-2-carboxylato-N,N',O)cobalt(II) hydrate, $[Co(C_{14}H_9N_2O_4)_2].H_2O$, and bis(9-dihydroxymethyl-1,10-phenanthroline-2-carboxylato-N,N',O)copper(II) hydrate, $[Cu(C_{14}H_9N_2O_4)_2].H_2O$, are isostructural. The stable *gem*-diol ligands are tridentate (two N atoms and one carboxylate O atom) and form a distorted octahedron around the metal center. The total bite angle of the ligand is approximately 150°.

Comment

Tridentate phenanthroline ligands are not common. Lee & Trogler (1990) have reported a monosubstituted phenanthroline ligand which coordinates with Cu^I. The species, however, forms a dimer (M_2L_2) where the phenanthroline N atoms bind one Cu atom and the