The structure was solved by the heavy-atom method and refined by full-matrix techniques. The absolute structure was determined [$\eta = +1.05(5)$].

The program system employed was *SHELXTL-Plus* (Sheldrick, 1989).

The crystals were kindly provided by Professor A. Laguna and co-workers, University of Zaragoza, Spain. We thank the Fonds der Chemischen Industrie for financial support.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55893 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1025]

References

Barron, P. F., Dyason, J. C., Healy, P. C., Engelhardt, L. M., Skelton, B. W. & White, A. H. (1986). *J. Chem. Soc. Dalton Trans.* pp. 1965–1970.

Harker, C. S. W. & Tiekink, E. R. T. (1989). *Acta Cryst.* **C45**, 1815–1817.

Meiners, J. H., Clardy, J. C. & Verkade, J. G. (1975). *Inorg. Chem.* **14**, 632–636.

Sheldrick, G. M. (1989). *SHELXTL-Plus*. Version 4.1. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Acta Cryst. (1993). **C49**, 1150–1152

Structure of Sodium Hydrogen (+)-Tartrate Monohydrate

Raymond C. Bott, Dalius S. Sagatys,
Daniel E. Lynch and Graham Smith*

School of Chemistry, Queensland University of Technology, PO Box 2434, Brisbane, 4001, Australia

Colin H. L. Kennard

Department of Chemistry, The University of Queensland, Brisbane, 4072, Australia

(*Received* 18 *May* 1992; *accepted* 30 *November* 1992)

Abstract

The structure of the title compound comprises a network polymer involving all tartrate O atoms (except the protonated carboxyl O atom) in bonding to four separate sodium complex centres. The water molecule completes a distorted bi-face capped trigonal prismatic eight coordination about Na [Na—O range 2.415–2.826 (2), mean 2.57 (14) Å] and is also involved in a number of hydrogen-bonding interactions. In this respect, the structure differs from the isomorphous series of anhydrous univalent cation (K^+, Rb^+, Cs^+, Tl^+, NH_4^+) salts of hydrogen (+)-tartrate.

Comment

The univalent cation hydrogen (+)-tartrates (K^+, Rb^+, Cs^+, Tl^+, NH_4^+) form an isomorphous series (Groth, 1910). Apart from early crystallographic work on the structures including determination of absolute configuration (van Bommel & Bijvoet, 1958), accurate structure determinations have only been completed quite recently [Templeton & Templeton (1978) (Cs); Buschmann & Luger (1985) and Akkurt, Hokelek & Soylu (1987) (K); Templeton & Templeton (1989) (Rb)]. Sodium hydrogen (+)-tartrate is not one of the isomorphous set and although the unit-cell parameters and space group have been reported for the monohydrate† (Zholanov, Umanskii, Barfolomeeva, Eshkova & Zolina, 1956), its crystal structure has not previously been determined. The title compound was isolated during a program of investigation into the stabilizing effect on trivalent Group 15 (As, Sb, Bi) tartrate-anion species using univalent cations (Sagatys, Smith, Lynch & Kennard, 1991) and its crystal structure determined.

The structure of the title compound comprises a three-dimensional network polymer, involving the hydrogen tartrate residues linked by eight-coordinate sodium ion centres (Figs. 1, 2). The NaO_8 stereochemistry is distorted bi-face capped trigonal prismatic, with an Na—O range of 2.415–2.826 (2) Å [mean 2.57 (14) Å]. This compares with a mean value of 2.38 Å over the twelve values for the two coordination centres in the disodium (+)-tartrate dihydrate structure (Ambady & Kartha, 1968) which also forms a network polymer. Also similar to the normal tartrate structure is the incorporation of the water molecule O(1W) in a strong bond to Na [2.420 (2), *cf.* 2.34, 2.42 Å]. The remaining coordination sites in the present structure are occupied by tartrato O atoms, including six from three bidentate chelate hydroxycarboxylate groups, two terminal (α-hydroxy) [$O(2)^a$, $O(12)^a$ ($a = -\frac{1}{2}+x, \frac{1}{2}-y, -z$); $O(3)^c$, $O(41)^c$ ($c = 1-x, -\frac{1}{2}+y, \frac{1}{2}-z$)] and one sub-terminal (β-hydroxycarboxyl) [O(2), O(42)]. In the remaining site, there is a bridging link to another carboxyl O atom [$O(12)^b$ ($b = x-1, y, z$)]. The β-hydroxycarboxyl chelate linkage is unusual in forming a six-membered ring system which expands the

---

† Comparative cell data: $a = 8.663$ (2), $b = 10.580$ (4), $c = 7.230$ (4) Å, space group $P2_12_12_1$.

0108-2701/93/061150-03$06.00

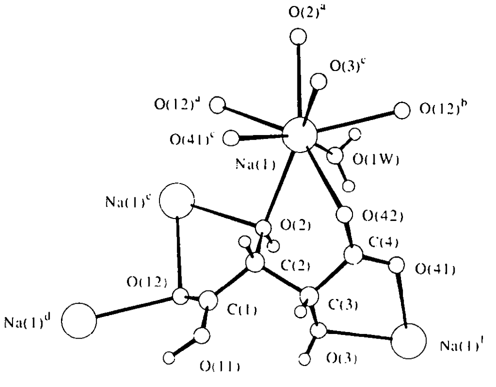

Fig. 1. Molecular conformation and atom-numbering scheme for the tartrate residue and the sodium ion coordination.

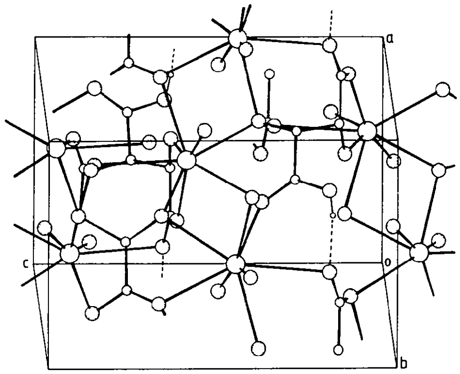

Fig. 2. Packing of the complex shown in a perspective view along the b direction.

bite angle at Na to 71.5 (1)°, compared to 61.3, 65.1 (1)° for the other two five-membered chelate rings.

The overall system of coordination, involving all tartrate O atoms except that of the protonated carboxylic acid group [O(11)], contrasts with those of the other (isomorphous) alkali-metal hydrogen (+)-tartrates. In these, only the α-hydroxycarboxylate groups containing a protonated carboxyl group are involved in chelation (Tapscott, 1982). However, this is not the case with the structure of sodium trihydrogen di-*meso*-tartrate (Blankensteyn & Kroon, 1986). The dihedral angle between the least-squares planes to the two α-hydroxycarboxyl groups [O(11), O(12), C(1), C(2), O(2) and O(41), O(42), C(4), C(3), O(3), 76.5 (2)°] is considerably different from the values for both the parent (+)-tartaric acid 56.5° (Okaya, Stemple & Kay, 1966) and the potassium and disodium salts (59.3 and 69°, respectively). In addition, the tartrate residues are linked by a linear head-to-tail intermolecular hydrogen-bonding association between the carboxylic acid proton H(11) and an adjacent carboxyl O atom [O(42) ($1 + x, y, z$); O···O 2.548 (3) Å, O—H···O 167 (2)°]. The coordinated water O(1W) is also involved in hydrogen-bonding interactions with both carboxylate and hydroxyl oxygens [O(1W)···O(41) 2.765 (3) ($\frac{1}{2} - x, -\frac{1}{2} + y, -\frac{1}{2} + z$); O(1W)···O(3) 2.699 (3) Å ($\frac{3}{2} - x, 1 - y, -\frac{1}{2} + z$)].

Experimental

Crystal data

$Na^+.C_4H_5O_6^-.H_2O$
$M_r = 190.1$
Orthorhombic
$P2_12_12_1$
$a = 7.2425$ (6) Å
$b = 8.676$ (1) Å
$c = 10.592$ (1) Å
$V = 665.6$ (1) Å^3
$Z = 4$
$D_x = 1.896$ Mg m^{-3}
$D_m = 1.90$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 6$-14°
$\mu = 0.22$ mm^{-1}
$T = 297$ K
Prismatic
0.25 × 0.17 × 0.11 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$2\theta/\omega$ scans
Absorption correction: empirical (*PSICALC*; Fair, 1990)
$T_{min} = 0.97$, $T_{max} = 0.98$
763 measured reflections
763 independent reflections

627 observed reflections [$I > 2.5\sigma(I)$]
$\theta_{max} = 25°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 12$
3 standard reflections monitored every 100 reflections
intensity variation: 0.7%

Refinement

Refinement on F
Final $R = 0.021$
$wR = 0.024$
$S = 0.94$
627 reflections
136 parameters
All H-atom parameters refined
$w = [\sigma^2(F_o)+0.00077F_o^2]^{-1}$

$(\Delta/\sigma)_{max} = 0.08$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Na(1)	0.4109 (1)	0.2074 (1)	0.0732 (1)	0.0271 (2)
C(1)	1.0070 (3)	0.2685 (2)	0.2382 (2)	0.0175 (5)
O(11)	1.0846 (2)	0.2175 (2)	0.3413 (2)	0.0240 (4)
O(12)	1.0884 (2)	0.3120 (2)	0.1446 (2)	0.0286 (4)
C(2)	0.7960 (3)	0.2637 (2)	0.2439 (2)	0.0178 (5)
O(2)	0.7189 (2)	0.3310 (2)	0.1341 (2)	0.0275 (5)
C(3)	0.7246 (2)	0.3418 (2)	0.3632 (2)	0.0163 (5)
O(3)	0.7879 (2)	0.4946 (2)	0.3709 (2)	0.0254 (4)
C(4)	0.5116 (3)	0.3428 (2)	0.3643 (2)	0.0176 (5)
O(41)	0.4308 (2)	0.4641 (2)	0.3897 (2)	0.0268 (4)
O(42)	0.4364 (2)	0.2144 (2)	0.3394 (2)	0.0256 (4)
O(1W)	0.4010 (2)	0.4749 (2)	0.0090 (2)	0.0271 (5)

Table 2. *Bond distances* (Å) *and angles* (°)

Na(1)—O(12)^a	2.645 (2)	O(11)—C(1)	1.305 (2)
Na(1)—O(12)^b	2.618 (2)	O(12)—C(1)	1.214 (3)
Na(1)—O(2)	2.558 (2)	C(2)—C(1)	1.530 (3)
Na(1)—O(2)^a	2.620 (2)	O(2)—C(2)	1.415 (3)
Na(1)—O(3)^c	2.414 (2)	C(3)—C(2)	1.524 (3)
Na(1)—O(41)^c	2.434 (2)	O(3)—C(3)	1.405 (2)
Na(1)—O(42)	2.826 (2)	C(4)—C(3)	1.542 (3)
Na(1)—O(1W)	2.420 (2)	O(41)—C(4)	1.234 (2)
		O(42)—C(4)	1.268 (2)
Na(1)^d—O(12)—Na(1)^e	99.2 (1)	O(2)—Na(1)—O(3)^c	141.1 (1)
Na(1)^d—O(2)—Na(1)	101.4 (1)	O(2)—Na(1)—O(41)^c	85.0 (1)
Na(1)^d—O(12)—C(1)	117.2 (1)	O(2)—Na(1)—O(42)	71.5 (1)
Na(1)^e—O(12)—C(1)	124.2 (1)	O(2)—Na(1)—O(1W)	72.2 (1)
Na(1)—O(2)—C(2)	112.2 (1)	O(2)^a—Na(1)—O(3)^c	78.0 (1)
Na(1)^d—O(2)—C(2)	115.2 (1)	O(2)^a—Na(1)—O(41)^c	106.0 (1)
Na(1)^f—O(3)—C(3)	122.8 (1)	O(2)^a—Na(1)—O(42)	150.9 (1)
Na(1)^f—O(41)—C(4)	123.5 (1)	O(2)^a—Na(1)—O(1W)	82.6 (1)
Na(1)—O(42)—C(4)	104.9 (1)	O(3)^c—Na(1)—O(41)^c	65.1 (1)
O(12)^a—Na(1)—O(12)^b	135.0 (1)	O(3)^c—Na(1)—O(42)	79.1 (1)
O(12)^a—Na(1)—O(2)	79.8 (1)	O(3)^c—Na(1)—O(1W)	141.7 (1)
O(12)^a—Na(1)—O(2)^a	61.3 (1)	O(41)^c—Na(1)—O(42)	80.0 (1)
O(12)^a—Na(1)—O(3)^c	117.1 (1)	O(41)^c—Na(1)—O(1W)	153.0 (1)
O(12)^a—Na(1)—O(41)^c	81.8 (1)	O(42)—Na(1)—O(1W)	105.1 (1)
O(12)^a—Na(1)—O(42)	147.1 (1)	O(12)—C(1)—O(11)	125.4 (2)
O(12)^a—Na(1)—O(1W)	80.2 (1)	C(2)—C(1)—O(11)	112.8 (2)
O(12)^b—Na(1)—O(2)	124.0 (1)	C(2)—C(1)—O(12)	121.7 (2)
O(12)^b—Na(1)—O(2)^a	79.2 (1)	O(2)—C(2)—C(1)	110.5 (2)
O(12)^b—Na(1)—O(3)^c	70.2 (1)	C(3)—C(2)—C(1)	111.1 (2)
O(12)^b—Na(1)—O(41)^c	132.4 (1)	C(3)—C(2)—O(2)	111.3 (2)
O(12)^b—Na(1)—O(42)	76.2 (1)	O(3)—C(3)—C(2)	110.9 (2)
O(12)^b—Na(1)—O(1W)	73.9 (1)	C(4)—C(3)—C(2)	110.4 (2)
O(2)—Na(1)—O(2)^a	136.7 (1)	C(4)—C(3)—O(3)	108.7 (1)
		O(41)—C(4)—C(3)	118.7 (2)
		O(42)—C(4)—C(3)	115.0 (2)
		O(42)—C(4)—O(41)	126.2 (2)

Symmetry codes: (*a*) $x-\frac{1}{2}, \frac{1}{2}-y, -z$; (*b*) $x-1, y, z$; (*c*) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (*d*) $\frac{1}{2}+x, \frac{1}{2}-y, -z$; (*e*) $1+x, y, z$; (*f*) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$.

The compound was isolated as a minor crystalline product in the preparation of sodium arsenic(III) (+)-tartrate from the addition of sodium bicarbonate to an aqueous digest of (+)-tartaric acid and arsenic(III) oxide. The same crystalline material was obtained subsequently by half neutralizing (+)-tartaric acid with sodium hydroxide in aqueous solution or by using a previously described procedure (Adin, Klotz & Newman, 1970) in which an equimolar mixture of (+)-tartaric acid and disodium (+)-tartrate is crystallized from water. The density was measured by flotation in $CHCl_3/CHBr_3$.

The data were corrected for Lorentz and polarization effects. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986) and refined by full-matrix least squares (*SHELX*76; Sheldrick, 1976) with anisotropic thermal parameters for all non-H atoms.

Final atomic coordinates are listed in Table 1 and intramolecular bond distances and angles are given in Table 2.

The authors acknowledge financial assistance from the Australian Research Council, The University of Queensland and the Queensland University of Technology. Mr Karl Byriel is thanked for technical assistance.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55894 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1015]

References

Adin, A., Klotz, P. & Newman, L. (1970). *Inorg. Chem.* **9**, 2499–2505.

Akkurt, M., Hokelek, T. & Soylu, H. (1987). *Z. Kristallogr.* **181**, 161–165.

Ambady, G. K. & Kartha, G. (1968). *Acta Cryst.* **B24**, 1540–1547.

Blankensteyn, A. J. A. R. & Kroon, J. (1986). *Acta Cryst.* **C42**, 291–293.

Bommel, A. J. van & Bijvoet, J. M. (1958). *Acta Cryst.* **11**, 61–70.

Buschmann, J. & Luger, P. (1985). *Acta Cryst.* **C41**, 206–208.

Fair, C. K. (1990). *MolEN*. An interactive intelligent system for crystal structure analysis. Enraf–Nonius, Delft, The Netherlands.

Groth, P. (1910). *Chemishe Krystallographie*, Vol. III, pp. 314–322. Leipzig: Englemann.

Okaya, Y., Stemple, N. R. & Kay, M. I. (1966). *Acta Cryst.* **21**, 237–243.

Sagatys, D. S., Smith, G., Lynch, D. E. & Kennard, C. H. L. (1991). *J. Chem. Soc. Dalton Trans.* pp. 361–364.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS*86. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Tapscott, R. E. (1982). *Transition Met. Chem.* **8**, 253–381.

Templeton, L. K. & Templeton, D. H. (1978). *Acta Cryst.* **A34**, 368–371.

Templeton, L. K. & Templeton, D. H. (1989). *Acta Cryst.* **C45**, 675–676.

Zholanov, G. S., Umanskii, M. M., Barfolomeeva, A., Ezhkova, Z. I. & Zolina, Z. K. (1956). *Kristallografiya*, **1**, 271–273.

Acta Cryst. (1993). C**49**, 1152–1154

Structure of Di-2-pyridyl Ketone-Hydrate Platinum(II) Chloride Tetrahydrate

Shaun O. Sommerer*

Division of Science, The Pennsylvania State University at Erie, The Behrend College, Station Road, Erie, Pennsylvania 16563-1200, USA

Khalil A. Abboud

Department of Chemistry, The University of Florida, Gainesville, Florida 32611-2046, USA

(*Received* 4 *August* 1992; *accepted* 25 *November* 1992)

Abstract

An X-ray structural analysis of bis(di-2-pyridylmethanediol)platinum(II) dichloride tetrahydrate revealed a Pt^{II} cation coordinated by four N-atom donors with two additional long-range O-atom interactions resulting in pseudo-octahedral geometry. The formation of a six-membered chelate in boat-like conformation together with the long-range off-axial interactions between Pt and O in the

0108-2701/93/061152-03$06.00