The transition occurs with a gradual decrease of the coordination around V atoms from 6 to 4. This causes notable changes in the structure from columns of quadruple chains of edge-sharing octahedra (I), to columns of double chains of edge-sharing square pyramids (II), to zigzag single chains of corner-sharing tetrahedra (III). Spectroscopic measurements in the IR region (Baiocchi, Bettinelli, Montenero & Di Sipio, 1982; Calestani, Montenero, Pigoli & Bettinelli, 1984) showed analogies in the spectra of PbV_2O_6(III) and of equimolar $PbO.V_2O_5$ glass. The hypothesis of a similitude between their structures can be advanced, the structure of the glass consisting of disordered chains of VO_4 tetrahedra with Pb atoms interspersed.

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Conformational Aspects of *meso*-Tartaric Acid. VIII.* Structure of Sodium *meso*-Tartrate, $2Na^+.C_4H_4O_6^{2-}$

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Abstract. $M_r = 194{\cdot}05$, monoclinic, $P2_1/n$, $a = 4{\cdot}999\ (1)$, $b = 6{\cdot}348\ (2)$, $c = 10{\cdot}135\ (2)$ Å, $\beta = 97{\cdot}49\ (1)°$, $V = 318{\cdot}9\ (1)$ Å^3, $Z = 2$, $D_x = 2{\cdot}021$ Mg m^{-3}, λ(Mo $K\alpha$) = $0{\cdot}71069$ Å, $\mu = 0{\cdot}32$ mm^{-1}, $F(000) = 192$, $T = 295$ K, $R = 0{\cdot}029$ for 2056 observed diffractometer data. The *meso*-tartrate anion adopts an unusual centrosymmetric conformation, imposed by the crystallographic centre of symmetry. This unfavourable conformation accompanies an appreciable distortion from planarity of the glycolic acid parts. The anions are joined together *via* a two-dimensional hydrogen-bonded network. The Na^+ ion is irregularly coordinated to six O atoms.

Introduction. It has been established that in solution *meso*-tartaric acid consists predominantly of enantiomeric rotamers (Kroon, 1982). These are also present in the crystal structures of three modifications of the free acid and of metal complexes studied so far. The first centrosymmetric conformation to be found in a crystal structure was in a complex of *meso*-tartaric acid with an optically active base (Kroon, Duisenberg & Peerdeman, 1984). Therefore it seemed worthwhile to extend our structural knowledge about salts of *meso*-tartaric acid with simple cations. The present crystal-structure determination contributes to that aim.

Experimental. Regularly formed block-shaped crystal, dimensions approximately 0·5 mm, grown from an aqueous solution. Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, cell measurements with setting angles of 12 reflections in θ range from 14 to 17°; $\omega/2\theta$ scan mode, scan width $0{\cdot}40° + 0{\cdot}35°\tan\theta$ and variable speeds; $2\theta_{\max} = 86°$, $h = 0$ to 9, $k = 0$ to 12, $l = -19$ to 19; no systematic fluctuations in standard reflections $11\bar{4}$ and $\bar{1}14$; 2364 independent reflections, 2056 considered observed [$I > 2{\cdot}5\sigma(I)$]. Lp corrections, but no absorption correction. Structure solved by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement on F by full-matrix least squares (*SHELX*76; Sheldrick, 1976). H atoms located from a three-dimensional difference Fourier map and by stereochemical considerations; their positional and isotropic thermal parameters subsequently refined.

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* Part VII: de Vries & Kroon (1984).

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Weights according to $w = [\sigma^2(F) + k|F|^2]^{-1}$, $k = 0{\cdot}001$. Convergence at $R = 0{\cdot}029$, $R_w = 0{\cdot}029$, $S = 1{\cdot}40$, $(\Delta/\sigma)_{\max} = 0{\cdot}02$; max. and min. peaks on final $\Delta\rho$ map 0·15 and −0·06 e Å^{-3} respectively. Calculations carried out on the CDC-Cyber 175 computer of the University of Utrecht with programs *APOLLO* (data reduction and correction by A. L. Spek) and *EUCLID* [calculation of geometrical data and illustrations (Spek, 1982)]. Scattering factors for Na$^+$, O and C from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965).

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

For non-H atoms $U_{\rm eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}/U_{\rm iso}$
Na	0·32855 (8)	0·44874 (6)	0·32694 (4)	0·0195 (1)
O(1)	0·0262 (1)	0·2914 (1)	0·69393 (6)	0·0179 (1)
O(2)	0·3239 (2)	0·3441 (1)	0·55191 (7)	0·0261 (2)
O(3)	0·0001 (1)	0·1682 (1)	0·35234 (6)	0·0176 (1)
C(1)	0·1136 (1)	0·2640 (1)	0·58343 (6)	0·0134 (1)
C(2)	−0·0465 (1)	0·1148 (1)	0·48386 (6)	0·0128 (1)
H(1)	−0·237 (3)	0·104 (2)	0·496 (2)	0·019 (3)
H(2)	−0·151 (4)	0·150 (3)	0·311 (2)	0·040 (5)

Table 2. *Bond distances* (Å), *bond angles* (°) *and* Na$^+$ *coordination*

C(1)–O(1)	1·266 (1)	C(2)–C(2$^{\rm i}$)	1·552 (1)
C(1)–O(2)	1·246 (1)	C(2)–O(3)	1·424 (1)
C(1)–C(2)	1·532 (1)	C(2)–H(1)	0·98 (2)
		O(3)–H(2)	0·82 (2)
O(1)–C(1)–O(2)	124·79 (6)	O(3)–C(2)–C(2$^{\rm i}$)	109·87 (5)
O(1)–C(1)–C(2)	117·19 (5)	C(1)–C(2)–H(1)	113 (1)
O(2)–C(1)–C(2)	117·98 (6)	O(3)–C(2)–H(1)	115 (1)
C(1)–C(2)–C(2$^{\rm i}$)	109·00 (5)	C(2$^{\rm i}$)–C(2)–H(1)	100 (1)
C(1)–C(2)–O(3)	109·54 (5)	C(2)–O(3)–H(2)	101 (2)
Na···O distances (Å)			
Na···O(1$^{\rm ii}$)	2·338 (1)	Na···O(1$^{\rm iv}$)	2·411 (1)
Na···O(2)	2·378 (1)	Na···O(3)	2·458 (1)
Na···O(2$^{\rm iii}$)	2·387 (1)	Na···O(3$^{\rm v}$)	2·526 (1)

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

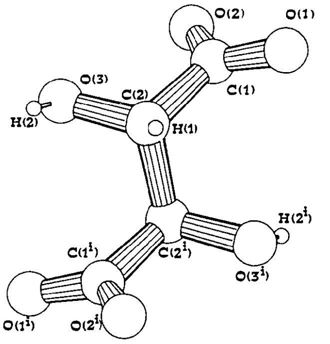

Fig. 1. Perspective view of the *meso*-tartrate dianion with atom-numbering scheme.

Discussion. Positional and isotropic thermal parameters are given in Table 1.* The molecular structure of the anion is shown in Fig. 1, which also gives the atom-numbering scheme. Bond lengths and bond angles are listed in Table 2.

The *meso*-tartrate anion is situated at a crystallographic centre of symmetry. Such a centrosymmetric conformation was also found in the acid and neutral salts of *meso*-tartaric acid with (−)-α-methylbenzylamine (Kroon *et al.*, 1984; Blankensteyn, Moerman, Ouwerkerk & Kroon, 1983). The quite large deviations from coplanarity of the C and O atoms in the two glycolic acid groups are similar to those established in the two centrosymmetric conformations mentioned above; the torsion angle O(2)–C(1)–C(2)–O(3) is −27·2 (1)°.

Preliminary molecular-mechanics calculations show that the centrosymmetric conformation is energetically less favourable than the dissymmetric ones; the calculations support the view (Kroon, Peerdeman & Bijvoet, 1965) that steric repulsion between the carboxyl O atom and the β-hydroxyl group appears to promote the dissymmetric conformations (Kroon-Batenburg, de Vries & Kroon, 1985).

The *meso*-tartrate anions are linked together in a two-dimensional hydrogen-bonded network, consisting of four-membered rings, which are approximately parallel to the (10$\bar{1}$) plane (Fig. 2).

The hydrogen-bond distances O(3)···O(1$^{\rm vi}$) and H(2)···O(1$^{\rm vi}$) are 2·695 (1) and 1·91 (2) Å respectively and the hydrogen-bond angle O(3)–H(2)···O(1$^{\rm vi}$) is 159 (2)°; the superscript (vi) refers to the atom at $-\frac{1}{2}+x$, $\frac{1}{2}-y$, $-\frac{1}{2}+z$. Details about the irregular coordination of the Na$^+$ ion are given in Table 2.

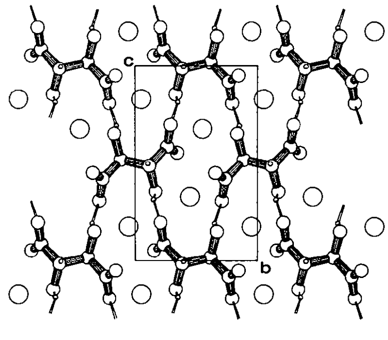

Fig. 2. View of the unit cell along the *a* axis. Na$^+$ ions are represented by the larger spheres, hydrogen bonds are indicated by small open sticks.

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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 39790 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Bis(2,2′-bipyridine)tris(ethyl isocyanide)molybdenum(II) Bis(tetrafluoroborate), $[Mo(C_2H_5NC)_3(C_{10}H_8N_2)_2][BF_4]_2$

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Abstract. $M_r = 747\cdot15$, monoclinic, $P2_1/a$, $a = 14\cdot584\ (5)$, $b = 14\cdot072\ (5)$, $c = 17\cdot281\ (5)$ Å, $\beta = 113\cdot35\ (3)°$, $V = 3256\cdot06$ Å^3, $Z = 4$, $D_x = 1\cdot52$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot41$ mm^{-1}, $F(000) = 1512$, $T = 253 \pm 1$ K, $R = 0\cdot082$ for 3991 observed reflections. The complex is monomeric with an isocyanide-capped distorted octahedral geometry. The influence of donor atoms *trans* to particular metal–ligand bonds is apparent in three types of Mo—N distances [ave. 2·198 Å, *trans* to nitrogen, 2·211 (10) Å *trans* to carbon, 2·257 (10) Å no *trans* donor ligand] and two types of Mo—C distance [2·096 (14) Å, *trans* to nitrogen, ave. 2·028 Å no *trans* donor ligand]. The isocyanide ligands are significantly distorted (ave. ∠CNC 161°).

Introduction. The preparation of seven-coordinate *cis*-$[Mo(CO)_2(bpy)_2(NCMe)][BF_4]_2$ (bpy is 2,2′-bipyridine) by oxidation of *cis*-$[Mo(CO)_2(bpy)_2]$ with silver(I) tetrafluoroborate in methyl cyanide solution has provided a versatile precursor for the chemistry of molybdenum(II). Reaction of precisely three equivalents of an isocyanide, *R*NC, with the molybdenum(II) salt in methyl cyanide solution produces $[Mo(bpy)_2(CNR)_3][BF_4]_2$ (R = Et, *p*-tolyl) (Connor, James, Overton & El Murr, 1984). No other structures of the class $[M(\text{bidentate ligand})_2(CNR)_3]^{2+}$ have been determined. The X-ray structure of $[Mo(bpy)_2(CNEt)_3][BF_4]_2$ is reported.

Experimental. Deep-purple crystals obtained from acetone/diethyl ether were sealed in glass capillaries under nitrogen for X-ray work. Enraf–Nonius CAD-4 diffractometer, crystal size 0·4 × 0·25 × 0·2 mm, cell dimensions from setting angles for 25 widely distributed reflections, graphite-monochromated Mo $K\alpha$ radiation, empirical absorption correction (North, Phillips & Mathews, 1968), 5690 reflections scanned, $\omega/2\theta$ mode, $2\cdot0 \le \theta \le 25\cdot0°$, index range h −17 to 17, k 0 to 16, l 0 to 20, no decay in intensity of three reflections checked every 3600 s exposure time, no slippage in orientation for two reflections checked every 100 measurements, 3991 reflections with $I > 1\cdot5\sigma(I)$ used in analysis. Structure solved by heavy-atom method and refined by full-matrix least squares. Non-hydrogen atoms in the cation were refined anisotropically, atoms in the BF_4^- ions and the ethyl carbons of two EtNC groups which showed very high thermal motion refined isotropically. Only H atoms on the bipyridine groups were included, and these were fixed in idealized positions. To achieve stable refinement, the C_α—C_β and C_α—N bond distances in the EtNC ligands were fixed at 1·50 and 1·45 Å respectively. $\sum w(\Delta F)^2$ minimized, final shifts all $<0\cdot04\sigma$, unit weights, $R = 0\cdot082$, residual electron

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