

Table 2. Comparison of bond distances (Å) and angles (°) in metal complexes of *disn* and *damn*

	Ni( <i>disn</i> ) <sub>2</sub> (square planar)	Pt( <i>disn</i> ) <sub>2</sub> (square planar)	Co( <i>disn</i> ) <sub>2</sub> (CN) (square pyramidal)	<i>damn</i>
M—N(1)	1.827 (4)	1.957 (8)	1.850 (5)	
N(1)—C(1)	1.330 (4)	1.323 (13)	1.325 (8)	1.392 (8)
C(1)—C(2)	1.403 (5)	1.416 (19)	1.428 (8)	1.363 (6)
C(1)—C(5)	1.443 (5)	1.415 (15)	1.454 (9)	1.439 (7)
C(5)—N(5)	1.135 (5)	1.123 (15)	1.122 (10)	1.165 (10)
N(1)—M—N(2)	84.3 (1)	79.1 (5)	82.9 (2)	
M—N(1)—C(1)	114.6 (2)	116.4 (7)	115.5 (4)	
N(1)—C(1)—C(2)	113.2 (3)	114.0 (6)	112.7 (6)	124.1 (4)
N(1)—C(1)—C(5)	124.4 (3)	122.6 (9)	124.3 (6)	117.6 (3)
C(2)—C(1)—C(5)	122.4 (3)	123.3 (7)	122.8 (6)	118.1 (8)
C(1)—C(5)—N(5)	177.8 (4)	179.7 (13)	177.2 (9)	179.8 (9)
Reference	Lauher & Ibers (1975)	Peng <i>et al.</i> (1981)	Penfold & Lipscomb (1961)	

differences between the anion ligand in the complex and the free ligand diaminomaleonitrile (*damn*) (Penfold & Lipscomb, 1961) involve N(1)—C(1) and C(1)—C(2), with N(1)—C(1) much shorter (1.330 Å) and C(1)—C(2) much longer (1.403 Å) in the complex than in the free ligand (1.392, 1.363 Å). This confirms the delocalization of  $\pi$  electrons over the ligand moiety upon complexation.

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## Conformational Aspects of *meso*-Tartaric Acid. VII.\* Structure of Calcium *meso*-Tartrate Trihydrate, Ca<sup>2+</sup>·C<sub>4</sub>H<sub>4</sub>O<sub>6</sub><sup>2-</sup>·3H<sub>2</sub>O

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**Abstract.**  $M_r = 242.20$ , monoclinic,  $P2_1/c$ ,  $a = 8.921$  (1),  $b = 10.300$  (3),  $c = 9.881$  (1) Å,  $\beta = 91.78$  (1)°,  $V = 907.5$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.773$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 6.17$  mm<sup>-1</sup>,  $F(000) = 496$ ,  $T = 295$  K. Final  $R = 0.065$  for 1817 observed unique reflections. The *meso*-tartrate dianion adopts a dissymmetric conformation, staggered about the central C—C bond. The non-H skeleton comprises two nearly planar halves consisting of glycolic acid. The Ca<sup>2+</sup> ion is irregularly surrounded by seven O atoms. Extensive hydrogen bonding contributes to the stabilization of the structure.

**Introduction.** The crystal structure of the title compound has been determined in the course of a

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conformational study of the *meso*-tartaric acid molecule.

**Experimental.** Colourless transparent needle-shaped crystals obtained by single-gel diffusion of CaCl<sub>2</sub> in a gel formed upon mixing sodium silicate and *meso*-tartaric acid; crystal size 0.66 × 0.43 × 0.13 mm. Enraf-Nonius CAD-4 diffractometer, Ni-filtered Cu  $K\alpha$  radiation; lattice dimensions determined using 14 reflections in  $\theta$  range 8 to 21°. Systematic absences  $h0l$  for  $l = 2n+1$  and  $0k0$  for  $k = 2n+1$ ;  $h-10-10$ ,  $k0-12$ ,  $l-12-12$ ; 1858 independent intensities measured up to  $2\theta_{\text{max}} = 75^\circ$ ; 1828 above  $2.5\sigma(I)$  level considered observed. Three periodically measured standard reflections, 102,  $\bar{1}11$ , 02 $\bar{2}$ , showed no significant decay in intensity during data collection; severe absorption (up to 60%) was noticed in the  $\psi$  scan, but initially no absorption correction applied; Lp correction.

\* Part VI: Kroon, Duisenberg & Peerdeman (1984).

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Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}$
Ca	0.18672 (5)	0.06923 (4)	0.21414 (4)	0.0203 (2)
O(1)	0.2654 (3)	0.9515 (2)	0.0253 (2)	0.0324 (6)
O(2)	0.3460 (2)	0.7640 (2)	-0.0595 (2)	0.0298 (5)
O(3)	0.1155 (2)	0.8426 (2)	0.2150 (2)	0.0249 (5)
O(4)	0.0515 (2)	0.5574 (2)	0.1778 (2)	0.0241 (5)
O(5)	-0.1712 (2)	0.6945 (2)	0.0803 (2)	0.0287 (5)
O(6)	-0.0387 (2)	0.7761 (2)	-0.0877 (2)	0.0264 (5)
O(7)	0.3954 (2)	0.9859 (2)	0.3387 (2)	0.0423 (7)
O(8)	0.3649 (2)	0.2200 (2)	0.1316 (2)	0.0289 (5)
O(9)	0.3131 (2)	0.4605 (2)	0.2718 (2)	0.0341 (6)
C(1)	0.2747 (3)	0.8286 (3)	0.0231 (2)	0.0228 (6)
C(2)	0.1955 (3)	0.7540 (2)	0.1339 (2)	0.0196 (6)
C(3)	0.0918 (3)	0.6476 (2)	0.0756 (2)	0.0199 (6)
C(4)	-0.0528 (3)	0.7098 (2)	0.0182 (2)	0.0213 (6)

Table 2. Bond distances ( $\text{\AA}$ ), bond angles and torsion angles ( $^\circ$ ) involving non-H atoms, hydrogen-bonding geometry and Ca coordination

O(1)—C(1)	1.269 (4)	O(2)—C(1)	1.243 (3)	
O(3)—C(2)	1.421 (3)	O(4)—C(3)	1.427 (3)	
O(5)—C(4)	1.248 (3)	O(6)—C(4)	1.259 (3)	
C(1)—C(2)	1.529 (3)	C(2)—C(3)	1.535 (3)	
C(3)—C(4)	1.533 (4)			
O(1)—C(1)—C(2)	117.2 (2)	O(1)—C(1)—O(2)	125.5 (2)	
O(2)—C(1)—C(2)	117.3 (3)	O(3)—C(2)—C(1)	109.4 (2)	
O(3)—C(2)—C(3)	111.3 (2)	C(1)—C(2)—C(3)	112.1 (2)	
O(4)—C(3)—C(2)	111.2 (2)	O(4)—C(3)—C(4)	107.8 (2)	
C(2)—C(3)—C(4)	109.3 (2)	O(5)—C(4)—O(6)	125.8 (2)	
O(5)—C(4)—C(3)	118.7 (2)	O(6)—C(4)—C(3)	115.4 (2)	
O(2)—C(1)—C(2)—O(3)	-177.7 (2)	C(1)—C(2)—C(3)—O(4)	164.1 (2)	
O(1)—C(1)—C(2)—C(3)	128.0 (2)	C(2)—C(3)—C(4)—O(5)	-108.0 (2)	
O(2)—C(1)—C(2)—C(3)	-53.8 (3)	O(4)—C(3)—C(4)—O(6)	-169.0 (2)	
O(3)—C(2)—C(3)—C(4)	-77.0 (2)	C(2)—C(3)—C(4)—O(6)	70.0 (2)	
O(3)—C(2)—C(3)—O(4)	-73.0 (2)			
O—H ( $\text{\AA}$ ) H...O ( $\text{\AA}$ ) O...O ( $\text{\AA}$ ) O—H...O ( $^\circ$ )				
O(3)—H(3)...O(6 <sup>ii</sup> )	1.00 (3)	1.71 (3)	2.712 (3)	177 (3)
O(4)—H(4)...O(9)	1.12 (4)	1.71 (4)	2.678 (3)	142 (3)
O(7)—H(7 <sup>i</sup> )...O(9 <sup>ii</sup> )	0.73 (4)	2.21 (4)	2.864 (3)	149 (4)
O(7)—H(7 <sup>ii</sup> )...O(2)	0.75 (5)	2.20 (4)	2.803 (3)	138 (5)
O(8)—H(8 <sup>i</sup> )...O(5 <sup>iii</sup> )	0.88 (4)	1.97 (4)	2.814 (3)	162 (3)
O(8)—H(8 <sup>ii</sup> )...O(2 <sup>iv</sup> )	0.99 (4)	1.74 (4)	2.702 (3)	165 (3)
O(9)—H(9 <sup>i</sup> )...O(1 <sup>v</sup> )	1.07 (4)	1.68 (4)	2.710 (3)	159 (4)
O(9)—H(9 <sup>ii</sup> )...O(8)	0.84 (5)	2.16 (5)	2.883 (3)	143 (4)
Ca...O ( $\text{\AA}$ )				
Ca...O(1 <sup>v</sup> )	2.350 (2)	Ca...O(4 <sup>vi</sup> )	2.411 (2)	
Ca...O(7 <sup>v</sup> )	2.361 (2)	Ca...O(5 <sup>vi</sup> )	2.214 (2)	
Ca...O(8)	2.384 (2)	Ca...O(3 <sup>v</sup> )	2.419 (2)	
Ca...O(6 <sup>iii</sup> )	2.396 (2)			

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

Ca located by Patterson method; location of remaining non-H atoms by heavy-atom method. After isotropic and anisotropic least-squares refinement no H atoms could be located ( $R = 0.12$ ). Absorption correction following the *DIFABS* procedure (Walker & Stuart, 1983) applied on isotropically refined data. After this absorption correction and subsequent isotropic and

anisotropic full-matrix least-squares refinement on  $F$ , all H atoms could be located on a difference map; they were given the isotropic thermal parameters of the attached O/C atoms. 11 strong extinction-affected reflections with  $(|F_c| - |F_o|)/\sigma(F) > 4$  excluded and, with H-atom positions fixed, final  $R = 0.065$ ,  $R_w = 0.088$ ,  $S = 1.14$ ;  $w = 1/[\sigma^2(F_o) + 0.006537(F_o)^2]$ . Av. and max. shift/error for non-H parameters 0.03 and 0.14 respectively. Max. and min. densities in final difference synthesis 0.51 and  $-0.80 \text{ e \AA}^{-3}$  (around Ca). Scattering factors for  $\text{Ca}^{2+}$ , O and C from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965), dispersion-correction data from Cromer & Liberman (1970). All calculations carried out on the CDC-Cyber 175 of the University of Utrecht with the programs of *SHELX76* (Sheldrick, 1976), *APOLLO* (data reduction and correction by A. L. Spek) and *EUCLID* [calculations of geometrical data and illustrations (Spek, 1982)].

**Discussion.** Final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.\* A perspective view and the numbering scheme of the *meso*-tartrate dianion are shown in Fig. 1. Data on the geometry are given in Table 2.

As in most investigated crystal structures of the *meso*-tartaric acid compounds (Kroon, 1982), this dianion is dissymmetric and adopts a synclinal conformation about the central C—C bond, with a torsion angle C(1)—C(2)—C(3)—C(4) of  $-77.0 (2)^\circ$ . Thus this centrosymmetric crystal structure, containing both conformational antipodes, is of a racemic nature. The non-H skeleton of this dianion is built up by two nearly planar halves consisting of glycolic acid; the torsion

\* Lists of anisotropic thermal parameters, H-atom parameters and structure-factor amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39470 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

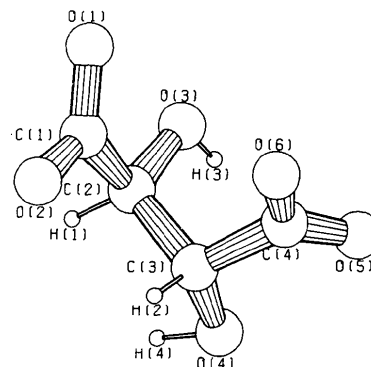


Fig. 1. Perspective view of the *meso*-tartrate dianion with numbering scheme.

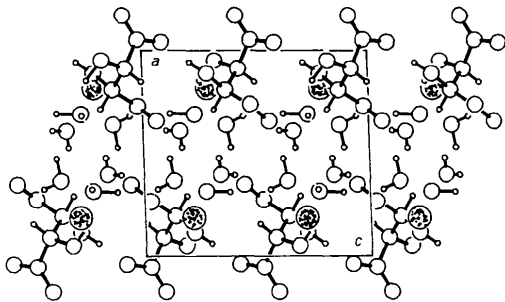


Fig. 2. View of the unit cell along the *b* axis.  $\text{Ca}^{2+}$  ions are indicated by stippled spheres.

angles about the central C—C bonds are  $\text{O}(1)\text{—C}(1)\text{—C}(2)\text{—O}(3) = 4.1(3)$  and  $\text{O}(5)\text{—C}(4)\text{—C}(3)\text{—O}(4) = 13.0(3)^\circ$ . Other torsion angles not involving H atoms are listed in Table 2.

The  $\text{Ca}^{2+}$  ion is irregularly surrounded by seven O atoms, including those of two water molecules. Details on the Ca coordination and the extensive hydrogen

bonding of this structure are given in Table 2. The packing of the structure is shown in Fig. 2.

We thank P. van der Sluis for growing and supplying the crystals.

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### Oxo[3,3'-(1,3-propanediyl-diimino)bis(3-methyl-2-butanone oximato)](3-)- $N,N',N'',N'''$ ]technetium(V), $[\text{TcO}(\text{C}_{13}\text{H}_{25}\text{N}_4\text{O}_2)]$

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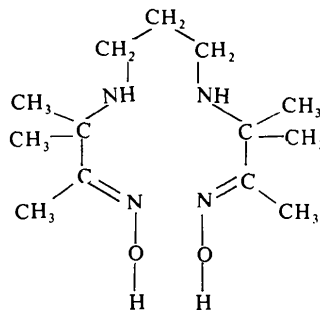
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**Abstract.**  $M_r = 383.4$ , monoclinic, *Pc*,  $a = 6.950(4)$ ,  $b = 11.187(3)$ ,  $c = 11.060(4)$  Å,  $\beta = 104.13(5)^\circ$ ,  $V = 833.8(2)$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.527(1)$  g cm<sup>-3</sup>,  $\text{Mo K}\alpha$ ,  $\lambda = 0.7107$  Å,  $\mu = 8.56$  cm<sup>-1</sup>,  $F(000) = 396$ ,  $T = 295(1)$  K, final  $R_F = 2.4\%$  for 2601 unique reflections. The  $\text{Tc}^{\text{V}}$  ion is surrounded by an approximate square pyramid of one multiple-bonded oxygen atom plus four nitrogen atoms from the tetradentate ligand. The distances between the deprotonated N(imino) atoms and the Tc atom [1.917(3), 1.908(3) Å] are much shorter than the N(oxime)—Tc bond distances [2.086(3) and 2.093(4) Å]. The Tc atom is 0.678(1) Å from the  $\text{N}_4$  plane in the direction of the oxo oxygen.

**Introduction.** The family of  $\alpha$ -amine oxime complexes has been studied extensively (e.g. Schlemper & Siripaisarnpipat, 1984) to obtain information about the

resultant intramolecular hydrogen bonds. The propylene-bridged example is shown below.



Upon complexation with a metal, the molecule usually loses one of the hydrogen atoms to form  $[M(\text{pnao-H})]$  with a hydrogen bond between the oxime oxygen atoms.