Tableau 3. *Géométrie de l'eau et liaisons hydrogènes*

O	H	X	O–H(Å)	∠HOH(°)	O···X(Å)	H···X(Å)	∠OHX(°)
O(5)	H(1) O(4ⁱ)		0,90 (5)	105 (5)	2,781 (4)	1,88 (6)	169 (5)
	H(2) O(6)		0,93 (5)		2,934 (4)	2,01 (5)	169 (5)
O(6)	H(3) O(7ⁱ)		0,77 (4)	78 (6)	2,783 (6)	2,32 (5)	119 (3)
	H(4) O(2ⁱⁱ)		0,78 (5)		2,848 (4)	2,10 (5)	161 (6)
O(7)	H(5) O(2)		0,81 (6)	104 (5)	2,750 (5)	1,96 (7)	162 (5)
	H(6) O(4)		0,91 (5)		2,775 (4)	1,87 (6)	168 (11)

Code de symétrie: (i) $x, y - 1, z$; (ii) $-1 - x, -y, 1 - z$.

sont conformes aux liaisons d'un cycle conjugué: $1,322 < d < 1,380$ Å.

Les longueurs des liaisons C–N du cycle varient en fonction des autres atomes liés au carbone. Elles sont comprises entre 1,322 et 1,338 Å quand les substituants sur le carbone sont un atome d'hydrogène et un autre atome d'azote et varient de 1,373 à 1,380 Å lorsque l'atome de carbone se trouve relié à un hydrogène et à un autre atome de carbone.

Les différentes longueurs des liaisons C–O reflètent les interactions auxquelles sont soumis les atomes d'oxygène des ions acétates. Cette longueur décroît selon que l'oxygène est lié aux deux cuivres, à un seul d'entre eux ou bien encore à une molécule d'eau par l'intermédiaire d'un pont hydrogène (Tableau 2). Les liaisons C–H dans les cycles imidazole valent en moyenne 0,95 Å. Dans les groupements méthyles des cycles, elles sont toujours de l'ordre de 0,98 Å en moyenne, alors que dans les ions acétates, elles ont pour valeur moyenne 1,03 Å.

Les angles moyens des groupements méthyles des cycles imidazole sont de l'ordre de 109° mais ne sont que de 106° pour les méthyles des groupements acétates.

La géométrie des molécules d'eau est classique. Les distances O–H sont comprises entre 0,77 et 0,93 Å

avec une moyenne de 0,85 Å. Le rôle des molécules d'eau est d'assurer la cohésion de l'édifice cristallin dans ce composé (Tableau 3).

Références

BAIRD, M. C. (1968). *Prog. Inorg. Chem.* 9, 1–159.
BERNARD, M. A., BOREL, M. M., BUSNOT, A., BUSNOT, F. & LECLAIRE, A. (1979). *Rev. Chim. Minér.* 16, 124–133.
BOREL, M. M., BUSNOT, A., BUSNOT, F. & LECLAIRE, A. (1980). *Rev. Chim. Minér.* 17, 202–208.
BOREL, M. M., BUSNOT, A., BUSNOT, F., LECLAIRE, A. & BERNARD, M. A. (1981a). *Rev. Chim. Minér.* 18, 74–82.
BOREL, M. M., BUSNOT, A., BUSNOT, F., LECLAIRE, A. & BERNARD, M. A. (1981b). *Rev. Chim. Minér.* 18, 370–375.
CATTERICK, J. & THORNTON, P. (1977). *Adv. Inorg. Chem. Radiochem.* 20, 291–354.
CHAUDHURY, G. R. & DASH, K. C. (1981). *J. Inorg. Nucl. Chem.* 43, 2189–2190.
CROMER, D. T. (1965). *Acta Cryst.* 18, 17–23.
CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* 18, 104–109.
DASH, K. C. & PUJARI, P. (1977). *J. Inorg. Nucl. Chem.* 39, 2167–2171.
HENRIKSON, H. A. (1977). *Acta Cryst.* B33, 1947–1950.
International Tables for X-ray Crystallography (1962). Tome III. Birmingham: Kynoch Press.
KATO, M., JONASSEN, H. B. & FANNING, J. C. (1964). *Chem. Rev.* 64, 99–128.
LUNDBERG, B. K. S. (1966). *Acta Cryst.* 21, 901–909.
OLDHAM, C. (1968). *Prog. Inorg. Chem.* 10, 223–258.
PROUT, C. K., ALLISON, G. B. & ROSSOTI, F. J. C. (1971). *J. Chem. Soc. A*, pp. 3331–3335.
REEDIJK, J. (1971). *J. Inorg. Nucl. Chem.* 33, 179–188.
SUNDBERG, R. J. & MARTIN, R. B. (1974). *Chem. Rev.* 4, 471–486.

Structure of Calcium Tartrate Tetrahydrate

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Abstract. Calcium tartrate tetrahydrate, $Ca^{2+}.C_4H_4O_6^{2-}.4H_2O$, orthorhombic, $P2_12_12_1$, $a = 9.631$ (2), $b = 10.573$ (3), $c = 9.215$ (2) Å, $V = 938.4$ Å³, $Z = 4$, $D_c = 1.84$ Mg m⁻³. The structure was solved by Patterson and Fourier methods, and refined by full-matrix least squares to an R of 0.037 for 1504 observed reflexions using Mo $K\alpha$ ($\lambda = 0.71069$ Å) X-radiation. Calcium links the tartrate molecules into infinite chains along **c**, with the chains cross-linked by a network of hydrogen bonds.

Introduction. A gel was prepared by acidification of a sodium silicate solution with tartaric acid in a constant-temperature bath, and a solution of 0.5 mol dm⁻³

calcium chloride was gently poured on top of the gel (Henisch, Dennis & Hanoka, 1965). Within a few days, clear crystals formed both within the gel and at the gel/solution interface. The powder pattern of these crystals was identical to that of calcium tartrate tetrahydrate [JCPDS Powder Diffraction File (1976), card No. 26-330], and single-crystal precession photographs confirmed the space group $P2_12_12_1$ assigned by Evans (1935). Cell dimensions were also obtained by least-squares refinement of 15 reflexions automatically aligned on a four-circle diffractometer. Intensity data were collected from an equidimensional crystal ~0·10 mm diameter. 1876 independent intensities with $2\theta <$ 60° were measured with graphite-monochromatized Mo $K\alpha$ radiation on a Syntex $P2_1$ diffractometer operating in the θ–2θ scan mode with variable scan rates between 4 and 20° min^{-1}. The intensities of two standard reflexions were measured every 50 reflexions throughout the data collection to monitor instrument stability and crystal alignment; no significant variations in intensity were observed. No absorption corrections were made as absorption effects were negligible ($\mu =$ 6·9 cm^{-1}). Standard data-reduction procedures resulted in 1504 observed reflexions, where a reflexion is considered as observed if its magnitude exceeds that of three standard deviations based on counting statistics.

Scattering factors for neutral atoms and anomalous dispersion corrections were taken from Cromer &

Mann (1968) and Cromer & Liberman (1970). The structure was solved by 3D Patterson and Fourier methods, and refined by full-matrix least squares on $\sum w(F_o - F_c)^2$ to a final conventional R of 0·037 for an anisotropic thermal model, except for the H atoms which were assigned constant isotropic temperature factors of 1·0 Å^2; unit weights were used throughout the refinement. Final parameters are given in Table 1, and selected interatomic distances and angles are given in Table 2.*

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* Anisotropic temperature factors, the magnitudes and orientations of the axes of the thermal ellipsoids, and structure factor tables have been deposited as Supplementary Publication No. SUP 36835 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
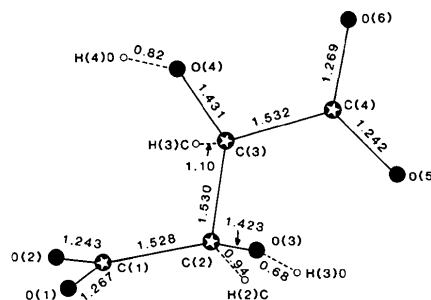
Table 2. *Interatomic distances* (Å) *and angles* (°) *in calcium tartrate tetrahydrate*

Ca polyhedron		Tartrate bond angles	
Ca–O(1)	2·377 (3)	O(1)–C(1)–O(2)	126·1 (3)
Ca–O(2)	2·425 (3)	O(1)–C(1)–C(2)	117·5 (3)
Ca–O(3)	2·474 (3)	O(2)–C(1)–C(2)	116·4 (3)
Ca–O(4)	2·518 (3)	C(1)–C(2)–O(3)	110·1 (3)
Ca–O(5)	2·506 (2)	C(1)–C(2)–C(3)	109·3 (3)
Ca–O(6)	2·416 (3)	O(3)–C(2)–C(3)	111·5 (3)
Ca–O(7)	2·494 (3)	C(2)–C(3)–O(4)	111·2 (3)
Ca–O(8)	2·432 (3)	C(2)–C(3)–C(4)	113·8 (3)
Mean	2·455 (3)	O(4)–C(3)–C(4)	109·0 (3)
		C(3)–C(4)–O(5)	119·9 (3)
		C(3)–C(4)–O(6)	114·7 (3)
		O(5)–C(4)–O(6)	125·3 (3)

Hydrogen bonds

A–H$\cdots B$	A–H	H$\cdots B$	$A\cdots B$	$\angle A$–H$\cdots B$
O(7)–H(7)$A\cdots$O(2)	0·93 (5)	2·26 (5)	2·871 (4)	123 (3)
O(7)–H(7)$B\cdots$O(4)	1·09 (4)	1·89 (4)	2·981 (5)	176 (4)
O(8)–H(8)$A\cdots$O(9)	0·78 (5)	2·02 (5)	2·781 (5)	162 (5)
O(8)–H(8)$B\cdots$O(1)	0·93 (5)	2·11 (5)	2·869 (5)	138 (4)
O(9)–H(9)$A\cdots$O(1)	0·91 (5)	2·03 (5)	2·919 (5)	165 (4)
O(9)–H(9)$B\cdots$O(5)	0·80 (5)	2·47 (5)	3·101 (5)	138 (4)
O(10)–H(10)$A\cdots$O(8)	0·82 (5)	2·23 (5)	3·036 (5)	167 (5)
O(10)–H(10)$B\cdots$O(7)	0·89 (5)	2·30 (5)	3·353 (5)	157 (4)

Table 1. *Atomic parameters for calcium tartrate tetrahydrate*

	x	y	z	B (Å^2)*
Ca	0·31773 (8)	0·67721 (7)	0·18649 (8)	1·18 (1)
C(1)	0·6495 (4)	0·7027 (3)	0·1085 (4)	1·28 (5)
C(2)	0·6596 (3)	0·7273 (3)	0·2716 (3)	1·04 (4)
C(3)	0·7219 (4)	0·8584 (2)	0·2971 (4)	1·16 (4)
C(4)	0·7385 (4)	0·8928 (3)	0·4577 (4)	1·22 (4)
O(1)	0·5316 (3)	0·6730 (3)	0·0578 (3)	1·91 (4)
O(2)	0·7584 (3)	0·7154 (3)	0·0377 (3)	1·75 (4)
O(3)	0·5263 (3)	0·7157 (3)	0·3369 (3)	1·65 (4)
O(4)	0·6416 (3)	0·9545 (2)	0·2268 (3)	1·47 (4)
O(5)	0·7628 (3)	0·8086 (3)	0·5481 (3)	1·66 (4)
O(6)	0·7319 (4)	0·0103 (3)	0·4850 (3)	2·25 (5)
O(7)	0·8360 (4)	0·5901 (3)	0·7735 (3)	2·47 (5)
O(8)	0·4345 (3)	0·3304 (5)	0·6933 (4)	3·63 (7)
O(9)	0·5855 (4)	0·4270 (4)	0·9254 (4)	3·22 (6)
O(10)	0·4242 (5)	0·0635 (4)	0·9299 (5)	4·23 (8)
H(7)A	0·800 (5)	0·569 (5)	0·864 (5)	1·0
H(7)B	0·948 (5)	0·574 (5)	0·769 (5)	1·0
H(8)A	0·473 (5)	0·343 (5)	0·767 (5)	1·0
H(8)B	0·486 (5)	0·272 (5)	0·640 (5)	1·0
H(9)A	0·552 (5)	0·500 (5)	0·965 (5)	1·0
H(9)B	0·593 (6)	0·378 (5)	0·990 (6)	1·0
H(10)A	0·449 (5)	0·996 (5)	0·895 (5)	1·0
H(10)B	0·359 (5)	0·094 (5)	0·870 (5)	1·0
H(2)C	0·726 (4)	0·676 (5)	0·317 (5)	1·0
H(3)C	0·828 (5)	0·856 (4)	0·254 (5)	1·0
H(3)O	0·534 (5)	0·683 (5)	0·400 (5)	1·0
H(4)O	0·680 (5)	0·970 (5)	0·150 (5)	1·0

* $B = \frac{4}{3} \sum_i \sum_j g_{ij}\beta_{ij}$; g_{ij} = real-space metric tensor.

Fig. 1. Bond lengths (Å) in the tartrate molecule. E.s.d.'s are 0·004 Å for C–C and C–O, 0·05 Å for C–H and O–H.

Discussion. Details of the tartrate molecule are shown in Fig. 1. The bond lengths and angles observed are conformable with those reported for (+)-tartaric acid (Okaya, Stemple & Kay, 1966). Calcium is surrounded by eight oxygens at distances between 2·377 (3) and
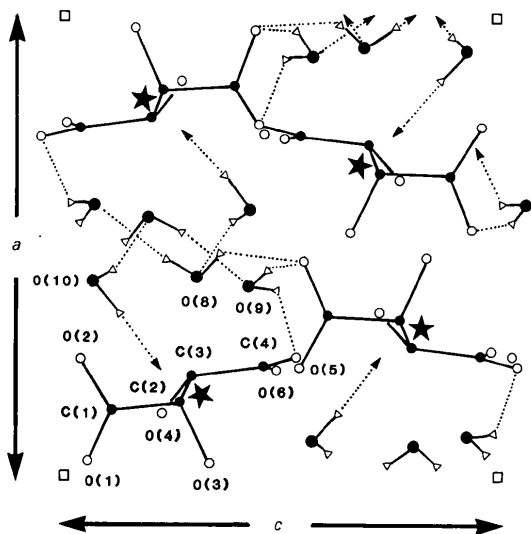

Fig. 2. A projection of the calcium tartrate tetrahydrate structure down the b axis. Ca—O and intramolecular hydrogen bonds have been omitted for clarity. The O(7) water molecule is almost occluded by Ca in this view and is also omitted: hydrogen bonds to O(7) are shown by arrowheads. Ca is represented by stars, C by small filled circles, O by hollow circles, H by hollow triangles and water oxygen atoms by large filled circles.

2·518 (3) Å, arranged in a distorted Siamese dodecahedron (Johnson, 1966).

The intermolecular linkages are shown in Fig. 2. Calcium links the tartrate molecules into infinite chains parallel to the c axis, with one Ca—O bond per tartrate molecule linking adjacent chains in the a direction. Most interchain linkage is provided by the network of hydrogen bonds formed by the additional water molecules. Calcium bonds directly to O(7) and O(8), each of which hydrogen bonds to an adjacent tartrate group. The water molecules O(9) and O(10) only participate in intermolecular linkage through hydrogen bonding.

References

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
EVANS, H. (1935). *Z. Kristallogr. Teil A*, **92**, 154–155.
HENISCH, H. K., DENNIS, J. & HANOKA, J. I. (1965). *J. Phys. Chem. Solids*, **26**, 493–500.
JCPDS Powder Diffraction File (1976). Card No. 26-330. Swarthmore, PA: JCPDS.
JOHNSON, N. W. (1966). *Can. J. Math.* **18**, 169–200.
OKAYA, Y., STEMPLE, N. R. & KAY, M. I. (1966). *Acta Cryst.* **21**, 237–242.

cis-Dichlorobis[dibenzyl(phenyl)phosphine]platinum(II)

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Abstract. [Pt(C$_{20}$H$_{19}$P)$_2$Cl$_2$], C$_{40}$H$_{38}$Cl$_2$P$_2$Pt, monoclinic; at 289 K: $C2/c$, a = 20·017 (5), b = 13·772 (5), c = 15·306 (4) Å, β = 114·47 (2)°, V = 2840 (2) Å^3, Z = 4, D_c = 1·51, D_x = 1·47 g cm^{-3}, μ(Mo $K\alpha$) = 39·37 cm^{-1}. R = 0·038 for 3170 observed reflections. Distances around Pt are normal [Pt—Cl 2·355 (4), Pt—P 2·246 (4) Å] but some angles indicate a significant degree of overcrowding [P—Pt—P 103·0 (1)°, one P—C(CH$_2$)—C(aromatic) 120·2 (4)° |.

Introduction. The title compound was prepared in the course of investigations into Pt—SnCl$_3$ compounds (Nelson, MacDougall, Holt, Alcock & Mathey, 1981). Its structure is of interest in relation to other *cis*-square-planar complexes for the degree of overcrowding around the heavy-metal atom (Alcock, Kemp & Wimmer, 1981). Chunky crystals were isolated by slow evaporation of a CH$_2$Cl$_2$ solution of *trans*-(Bz$_2$PPh)$_2$Pt(SnCl$_3$)Cl (Bz = benzyl). Although ^{31}P