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.

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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]

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

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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

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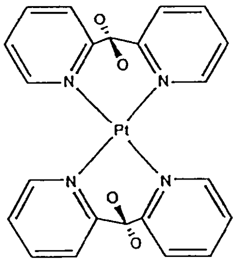
apical positions of the complex appear to contribute to the stability of the hydrated species.

Comment

The ligand di-2-pyridyl ketone (DPK) has been confirmed by X-ray diffraction studies to undergo hydration at the carbonyl forming a geminal diol when in aqueous media and in the presence of a transition-metal cation (Wang, Richardson, Briggs, Jacobson & Jensen, 1986). The geometry around the central metal is observed to be pseudo-octahedral for all 1:2 (metal:ligand) complexes reported to date (Sommerer, Jensen & Jacobson, 1990). This metal-promoted hydration is unusual since ketones are not typically hydrated to any appreciable extent in aqueous media unless flanked by very strong electron-withdrawing atoms such as Cl or F.

A structural analysis of the title complex (I) was motivated by our interest in observing the behaviour of DPK in aqueous media combined with a metal that is not customarily stabilized by an octahedral coordination sphere. Thus, platinum(II) was chosen because square-planar geometry is usually observed for this species.



(I)

Potassium tetrachloroplatinate(II) was combined on a 1:2 mol basis with DPK in 70 mL of H_2O and refluxed for 6 h. The resulting mixture was filtered and slow evaporation of the clear filtrate gave clear crystals suitable for X-ray diffraction studies. Notably, the IR spectrum of these crystals revealed an absence of an absorption band at 1690 cm^{-1} indicating that hydration had occurred at the ketone.

The crystals were found to consist of $[Pt(DPK\text{-}hydrate)_2]^{2+}$ species in which Pt^{2+} attains the usual square-planar coordination environment. The Pt cation is involved in two long-range interactions, at the approximate octahedral sites of the metal, with the O(1) atom of one hydroxyl group [Pt—O(1) = 2.940 (6) Å]. These distances are much longer than those usually considered as bonding interactions (*ca* 2.05 Å) though they are shorter than normal non-bonded distances (*ca* 3.6 Å). The angle formed between the Pt—O(1) vector and the normal to the coordination plane is 29.5°. The ligand forms a six-membered chelate displaying a boat-like conformation, resulting in a dihedral angle of 118.5° being formed between the two pyridine planes.

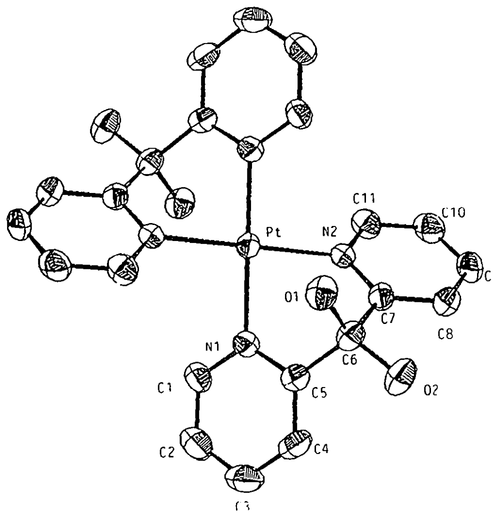


Fig. 1. Representation of $[Pt(DPK\text{-}hydrate)_2]^{2+}$ with thermal ellipsoids drawn at the 50% probability level.

Experimental

Crystal data

$[Pt(C_{11}H_{10}N_2O_2)_2]Cl_2.4H_2O$
$M_r = 742.47$
Monoclinic
$C2/c$
$a = 14.953$ (1) Å
$b = 12.428$ (1) Å
$c = 14.228$ (1) Å
$\beta = 91.778$ (7)°
$V = 2643.02$ Å^3
$Z = 4$
$D_x = 1.866$ Mg m^{-3}
$D_m = 1.86$ Mg m^{-3}
Density measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 10$–$11°$
$\mu = 5.617$ mm^{-1}
$T = 298$ K
Cubic block
0.28 × 0.28 × 0.27 mm
Colorless
Crystal source: slow evaporation of aqueous solution of DPK/K_2PtCl_4

Data collection

Siemens *P*3*m*/*V* diffractometer
ω scans
Absorption correction: analytical, based on measured crystal faces
$T_{min} = 0.232$, $T_{max} = 0.353$
6365 measured reflections
3070 independent reflections
2175 observed reflections
$[I_{net} > 2.5\sigma(I_{net})]$

$R_{int} = 0.013$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 19$
$k = -16 \rightarrow 16$
$l = -18 \rightarrow 18$
4 standard reflections ($\bar{2}02$, $0\bar{2}2$, $71\bar{2}$ and $13\bar{2}$)
monitored every 96 reflections
intensity variation: $<0.01\%$

Refinement

Refinement on F
Final $R = 0.035$
$wR = 0.033$
$S = 1.37$
2175 reflections
205 parameters
Only coordinates of H atoms refined, located *via* difference map; U_{iso} set at 0.049 Å^2
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.318$
$\Delta\rho_{max} = 0.98$ e Å^{-3}
$\Delta\rho_{min} = -0.78$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: *SHELXTL-Plus* (Sheldrick, 1990). Cell refinement: *SHELXTL-Plus*. Data reduction: *NRCVAX* (*DATRD*2) (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX* (*SOLVER*). Program(s) used to refine structure: *NRCVAX* (*LSTSQ*). Molecular graphics: *NRCVAX* (*ORTEPII*). Software used to prepare material for publication: *NRCVAX* (*TABLES*/*UTILITY*).

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}
Pt	0.0	0.0	0.5	0.029 (1)
Cl(1)	0.3086 (2)	−0.0481 (3)	0.5105 (2)	0.095 (2)
O(1)	0.1383 (3)	−0.1683 (5)	0.5073 (3)	0.043 (2)
O(2)	0.1620 (3)	−0.2587 (4)	0.3681 (3)	0.047 (2)
N(1)	−0.0457 (3)	−0.1412 (4)	0.4464 (4)	0.033 (2)
N(2)	0.0765 (3)	0.0118 (5)	0.3857 (3)	0.030 (2)
C(1)	−0.1346 (5)	−0.1632 (7)	0.4434 (5)	0.038 (3)
C(2)	−0.1669 (5)	−0.2580 (7)	0.4056 (5)	0.048 (3)
C(3)	−0.1089 (6)	−0.3315 (7)	0.3699 (6)	0.053 (4)
C(4)	−0.0171 (6)	−0.3085 (6)	0.3729 (5)	0.046 (4)
C(5)	0.0129 (4)	−0.2139 (5)	0.4103 (4)	0.033 (3)
C(6)	0.1120 (4)	−0.1816 (6)	0.4126 (5)	0.035 (3)
C(7)	0.1215 (4)	−0.0759 (6)	0.3565 (4)	0.033 (3)
C(8)	0.1769 (5)	−0.0680 (7)	0.2816 (5)	0.039 (3)
C(9)	0.1834 (5)	0.0276 (6)	0.2336 (5)	0.043 (3)
C(10)	0.1356 (5)	0.1156 (7)	0.2623 (5)	0.046 (3)
C(11)	0.0818 (5)	0.1045 (6)	0.3384 (5)	0.039 (3)
O(*W*1)	0.4124 (5)	−0.1157 (9)	0.3436 (7)	0.116 (6)
O(*W*2)	0.3761 (3)	0.1125 (4)	0.6347 (3)	0.043 (2)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Pt—N(1)	2.023 (5)	C(2)—C(3)	1.370 (13)
Pt—N(2)	2.026 (5)	C(3)—C(4)	1.401 (13)
O(1)—C(6)	1.400 (8)	C(4)—C(5)	1.360 (10)
O(2)—C(6)	1.382 (8)	C(5)—C(6)	1.534 (9)
N(1)—C(1)	1.356 (9)	C(6)—C(7)	1.546 (10)
N(1)—C(5)	1.372 (8)	C(7)—C(8)	1.375 (10)
N(2)—C(7)	1.354 (9)	C(8)—C(9)	1.376 (11)
N(2)—C(11)	1.338 (10)	C(9)—C(10)	1.376 (12)
C(1)—C(2)	1.375 (11)	C(10)—C(11)	1.377 (10)
N(1)—Pt—N(1*a*)	179.9	N(1)—C(5)—C(4)	120.6 (6)
N(1)—Pt—N(2)	87.30 (22)	N(1)—C(5)—C(6)	116.7 (6)
N(1)—Pt—N(2*a*)	92.70 (22)	C(4)—C(5)—C(6)	122.7 (6)
N(1*a*)—Pt—N(2)	92.70 (22)	O(1)—C(6)—O(2)	112.7 (5)
N(1*a*)—Pt—N(2*a*)	87.30 (22)	O(1)—C(6)—C(5)	106.9 (5)
N(2)—Pt—N(2*a*)	180.0	O(1)—C(6)—C(7)	111.6 (5)
Pt—N(1)—C(1)	120.4 (5)	O(2)—C(6)—C(5)	110.2 (5)
Pt—N(1)—C(5)	120.0 (4)	O(2)—C(6)—C(7)	106.9 (5)
C(1)—N(1)—C(5)	119.6 (6)	C(5)—C(6)—C(7)	108.5 (5)
Pt—N(2)—C(7)	119.2 (5)	N(2)—C(7)—C(6)	118.0 (6)
Pt—N(2)—C(11)	120.7 (5)	N(2)—C(7)—C(8)	119.9 (7)
C(7)—N(2)—C(11)	120.0 (5)	C(6)—C(7)—C(8)	122.0 (6)
N(1)—C(1)—C(2)	121.1 (7)	C(7)—C(8)—C(9)	120.1 (7)
C(1)—C(2)—C(3)	119.9 (7)	C(8)—C(9)—C(10)	119.5 (6)
C(2)—C(3)—C(4)	119.0 (7)	C(9)—C(10)—C(11)	118.5 (7)
C(3)—C(4)—C(5)	119.8 (7)	N(2)—C(11)—C(10)	121.9 (7)

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 71131 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1033]

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Redetermination of the Structure of Polymeric Carboxyethylgermanium Sesquioxide

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Abstract

Polymeric carboxylethylgermanium sesquioxide has an infinite sheet structure in which the basic unit of the network is a 12-membered ring made up of six Ge tetrahedra bridged by O atoms. The carboxylate chains are arranged alternately above and below the Ge—O network around the ring. Sheets are vertically linked by hydrogen-bond pairs between the carboxylate groups.

Comment

During our investigations on antitumour structure–activity relationships of the main-group metallic compounds, the structure of carboxyethylgermanium sesquioxide, $O_3(GeCH_2CH_2COOH)_2$, was brought to our attention owing to its extremely low toxicity and

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