recrystallized by lowering the pH of a 0.05 *M* solution of cadmium glutamate to 2.0 (6 *M* HCl). Once the cadmium glutamate was dissolved, the pH was adjusted to 5.88 and the solvent allowed to evaporate.

Crystal data

$[Cd(C_5H_7O_4)(H_2O)].H_2O$	Mo radiation
$M_r = 293.55$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	$\theta = 10.88$–$25.88°$
$a = 11.5747$ (13) Å	$\mu = 2.41$ mm^{-1}
$b = 10.7639$ (7) Å	$T = 296$ K
$c = 7.2556$ (7) Å	Prismatic
$V = 903.97$ (15) Å^3	0.40 × 0.30 × 0.30 mm
$Z = 4$	Colorless
$D_x = 2.157$ Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4 diffractometer	1019 reflections with $I > 3\sigma(I)$
$\theta/2\theta$ scans	$\theta_{max} = 27.96°$
Absorption correction:	$h = 0 \rightarrow 15$
ψ scan (*SDP/PDP*; Enraf–Nonius, 1985)	$k = 0 \rightarrow 14$
$T_{min} = 0.37$, $T_{max} = 0.48$	$l = 0 \rightarrow 9$
1063 measured reflections	3 standard reflections
1063 independent reflections	frequency: 240 min
	intensity decay: <3%

Refinement

Refinement on *F*	$w = 1/\sigma^2$
$R = 0.014$	$(\Delta/\sigma)_{max} < 0.001$
$wR = 0.020$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
$S = 0.91$	$\Delta\rho_{min} = -0.36$ e Å^{-3}
1019 reflections	Extinction correction: none
152 parameters	Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
Only coordinates of H atoms refined	

Table 1. *Selected geometric parameters (Å, °)*

Cd—N	2.290 (2)	Cd—O5	2.255 (2)
Cd—O1	2.282 (3)	C1—O2	1.254 (3)
Cd—O2^{i}	2.254 (2)	C1—O1	1.259 (4)
Cd—O3^{ii}	2.303 (2)	C2—N	1.469 (4)
Cd—O4	2.449 (2)		
O2—C1—O1	124.6 (3)	C4—C3—C2	113.5 (2)
N—C2—C1	112.1 (2)	C3—C4—C5	113.4 (2)
N—C2—C3	111.7 (2)	O4—C5—O3	120.4 (3)
C1—C2—C3	109.4 (2)		

Symmetry codes: (i) $\frac{3}{2} - x, 1 - y, z - \frac{1}{2}$; (ii) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$.

Table 2. *Hydrogen-bonding distances (Å)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*
O5—H8···O6	0.61 (5)	2.12 (5)	2.726 (3)
O5—H9···O6^{i}	0.74 (4)	1.99 (4)	2.717 (3)
O6—H10···O4^{iii}	0.62 (4)	2.14 (5)	2.786 (3)
O6—H11···O3	0.86 (5)	1.85 (5)	2.708 (3)
N—H6···O1^{iii}	0.77 (4)	2.34 (4)	2.957 (3)

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

All non-H atoms were located from electron-density maps and were refined anisotropically by full-matrix least squares. H atoms were located from difference Fourier synthesis and were refined isotropically. Their bonded distances were in the ranges 0.88–1.09 (C—H), 0.77–0.86 (N—H) and 0.62–0.86 Å (O—H).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 SDP* (Frenz, 1978). Program(s) used to solve structure: *CAD-4 SDP*. Program(s) used to refine structure: *CAD-4 SDP*. Molecular graphics: *Xtal3.4 ORTEP* (Hall, Flack & Stewart, 1995). Software used to prepare material for publication: *Xtal3.4 BONDLA CIFIO*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1343). Services for accessing these data are described at the back of the journal.

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Dual Behaviour of Acetylacetonate Anions in the Hydrogen-Bonded Supramolecular Structure (Acetylacetonato-*O*,*O*′)[*trans*-(1*R*,2*R*)-diaminocyclohexane-*N*,*N*′]-platinum(II) Acetylacetonate

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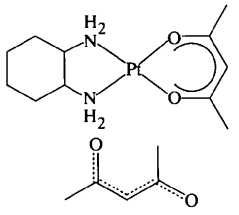
Abstract

The title compound, $[Pt(C_5H_7O_2)(C_6H_{14}N_2)](C_5H_7O_2)$, involves two independent acetylacetonate anions with

different behaviour; one is a planar chelating ligand bound to the Pt atom in the square-planar coordination sphere, and the other is a counteranion with an *E* formation. The latter anion links between its carbonyl O atoms and the amino groups of the cationic complexes to form the hydrogen-bonded network.

Comment

Water-soluble platinum(II) complexes can be prepared from dihydroxoplatinum(II) complexes, *cis*-$[Pt(OH)_2L_2]$ [L = am(m)ines, PMe_3, $AsMe_3$ or $SbMe_3$], and many kinds of inorganic or organic acids through neutralization reactions. We have recently reported the synthesis and structures of the platinum(II) ascorbate complexes, which were obtained from aqueous solutions containing *cis*-$[Pt(OH)_2L_2]$ [L_2 = chelating diamines or $(PMe_3)_2$] and ascorbic acid (H_2asc) in a 1:1 ratio (Yuge & Miyamoto, 1996*a*,*b*). It has already been reported by Hollis *et al.* (1985) that one of the platinum(II) ascorbate complexes, [Pt(*cis*-dach)(asc)] (dach = 1, 2-diaminocyclohexane), prepared in aqueous solution, had a Pt—C bond, although organometallic compounds have been synthesized in organic solvents in general. The ascorbate dianion in the compound can be regarded as an α-hydroxy-β-diketonate bound to the Pt atom by the C atom. Attempts have been made to synthesize such organometallic compounds from aqueous solutions containing the dihydroxoplatinum(II) complexes and some β-diketonates by, for example, investigating the reaction between [Pt(OH)$_2$(*trans*-1*R*,2*R*-dach)] and acetylacetone (Hacac) in a 1:2 ratio to crystallize the prepared compound [Pt(acac-*O*,*O*′)(*trans*-1*R*,2*R*-dach-*N*,*N*′)](acac), (1).



(1)

As shown in Fig. 1, the structure of (1) does not have any Pt—C bonds. Instead it involves two different acac$^-$ anions, as a chelating ligand and a counteranion. The Pt atom in a square-planar coordination sphere is surrounded by two chelating ligands, *trans*-1*R*,2*R*-dach and acac$^-$, each bonded by two N or two O atoms; the planarity of N1, N2, O12 and O14 is within 0.03 Å. The Pt—O distances, 2.004 (4) and 1.995 (4) Å, are comparable with those of [Pt(acac-*O*,*O*′)$_2$].C$_6$H$_6$ (Katoh *et al.*, 1981), 2.008 (15) and 1.979 (14) Å. No remarkable influence is observed on the Pt—N distances by the O donor atoms at the *trans* positions, and *vice versa*. The C—C and C—O bond lengths of the chelate and counteranions are within a reasonable range.

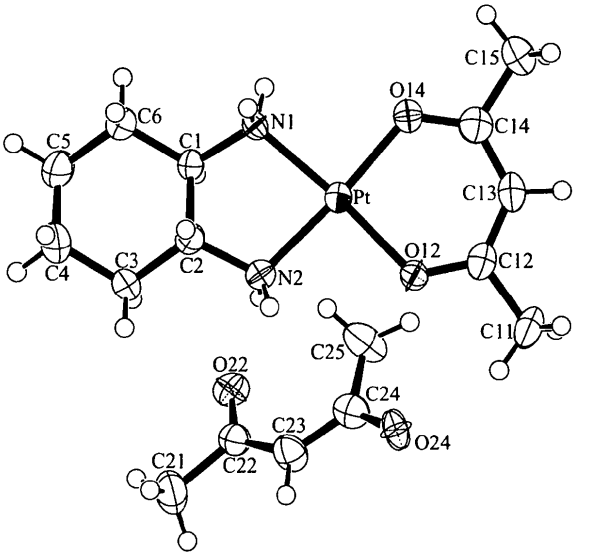


Fig. 1. The asymmetric unit of [Pt(acac-*O*,*O*′)(*trans*-1*R*,2*R*-dach)](acac) showing atomic notations. Displacement ellipsoids are drawn at the 50% probability level.

A Pd^{II} compound, [Pd(acac-*O*,*O*′)(NHEt$_2$)$_2$](acac), (2) (Kotake *et al.*, 1980), has a composition similar to (1). Although both structures involve two kinds of acac$^-$ anions, as chelating and counteranions, the crystal structure of (2) is quite different from that of (1) owing to a pair of the unidentate diethylamine ligands in place of the *trans*-1*R*,2*R*-dach chelate. In (2), the cationic complex [Pd(acac-*O*,*O*′)(NHEt$_2$)$_2$]$^+$ and the counteranion acac$^-$ are arranged in a C_2 symmetry by the hydrogen bonds between the amino groups of

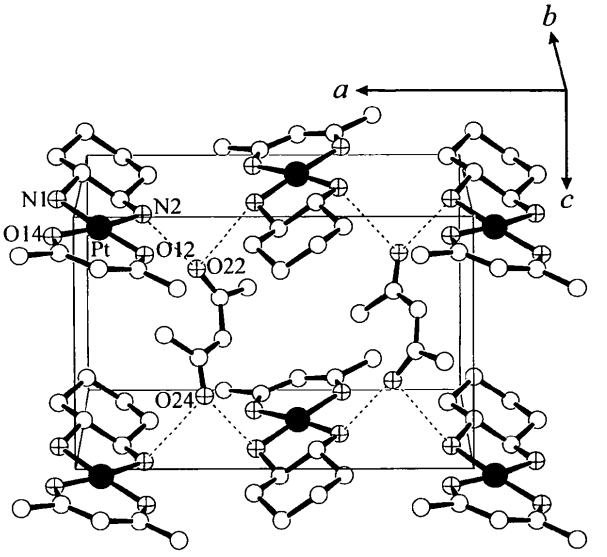


Fig. 2. The two-dimensional hydrogen-bond network of [Pt(acac-*O*,*O*′)(*trans*-1*R*,2*R*-dach)](acac) about the plane at $b = \frac{1}{2}$. Pt atoms are shown by shaded circles and O and N atoms by crossed ones; hydrogen bonds are shown by broken lines. H atoms have been omitted for clarity.

the diethylamine ligands and the carbonyl O atoms of the anion to form an ion pair. The acac$^-$ anion is situated almost perpendicular to the square plane about the Pd atom; the dihedral angle is 80.8°. In contrast, the acac$^-$ anion of (1) is placed between the cationic complexes [Pt(acac-*O*,*O*′)(*trans*-1*R*,2*R*-dach)]$^+$, being rotated around the C23—C24 bond by 177.4 (6)°. The dihedral angle between the acac$^-$ anion and the square plane about the Pt atom is 48.2 (2)°. The free anion of acac$^-$ with an *E* conformation is rather rare, while those as unidentate or bidentate ligands have often been observed in such compounds as [Pd(acac-*O*,*O*′)(acac-C^3)($NHEt_2$)] (Kurokawa *et al.*, 1982) or [Pt_4(μ-CH_3COO-*O*,*O*′)$_4$(μ-acac-*O*,C^3)$_4$] (Yamaguchi *et al.*, 1990). As shown in Fig. 2, one carbonyl O atom of the free acac$^-$ anion links between two amino groups of adjacent Pt complexes by hydrogen bonds along the *a* axis (Table 2), and the anion is arranged in parallel along the *c* axis to form a hydrogen-bonded network.

Experimental

Single crystals were prepared from an aqueous solution containing a 1:2 molar ratio of [$Pt(OH)_2$(*trans*-1*R*,2*R*-dach)] and Hacac, the former of which was synthesized according to the procedure reported by Totani *et al.* (1986).

Crystal data

$[Pt(C_5H_7O_2)(C_6H_{14}N_2)]$-$(C_5H_7O_2)$	Mo $K\alpha$ radiation
M_r = 507.49	λ = 0.71069 Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	θ = 15.045–17.795°
a = 12.4501 (9) Å	μ = 7.580 mm^{-1}
b = 18.6708 (11) Å	T = 293 K
c = 7.9840 (9) Å	Prism
V = 1855.9 (3) Å^3	0.22 × 0.20 × 0.18 mm
Z = 4	Colourless
D_x = 1.816 Mg m^{-3}	
D_m = 1.821 Mg m^{-3}	
D_m measured by flotation in $CHBr_3$–toluene	

Data collection

Rigaku AFC-7*R* diffractometer	2658 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	R_{int} = 0.040
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)	θ_{max} = 29.99°
	$h = -17 \rightarrow 17$
T_{min} = 0.189, T_{max} = 0.256	$k = 0 \rightarrow 26$
6320 measured reflections	$l = 0 \rightarrow 11$
3070 independent reflections (2350 Friedel pairs)	3 standard reflections every 150 reflections
	intensity decay: −2.1%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = 0.002
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$\Delta\rho_{max}$ = 0.576 e Å^{-3}
$wR(F^2)$ = 0.066	$\Delta\rho_{min}$ = −0.850 e Å^{-3}
S = 1.024	Extinction correction: none
5420 reflections (see text)	Scattering factors from *International Tables for Crystallography* (Vol. C)
208 parameters	
H-atom parameters constrained	Absolute configuration: Flack (1983)
$w = 1/[\sigma^2(F_o^2) + (0.0221P)^2 + 0.6053P]$ where $P = (F_o^2 + 2F_c^2)/3$	Flack parameter = −0.028 (12)

Table 1. *Selected geometric parameters (Å, °)*

Pt—N1	2.037 (4)	C22—C23	1.404 (9)
Pt—N2	2.036 (4)	C23—C24	1.398 (8)
Pt—O12	2.004 (4)	C24—C25	1.487 (9)
Pt—O14	1.995 (4)	C22—O22	1.258 (7)
C21—C22	1.525 (8)	C24—O24	1.277 (7)
N1—Pt—N2	84.2 (2)	C22—C23—C24	128.4 (6)
N1—Pt—O12	175.0 (2)	C23—C24—C25	122.1 (6)
N1—Pt—O14	91.1 (2)	C21—C22—O22	116.8 (6)
N2—Pt—O12	90.8 (2)	C23—C22—O22	127.0 (5)
N2—Pt—O14	174.8 (2)	C23—C24—O24	120.3 (6)
O12—Pt—O14	93.9 (2)	C25—C24—O24	117.5 (6)
C21—C22—C23	116.2 (6)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H2···O22^i	0.90	2.088	2.914 (6)	152.1
N2—H4···O22	0.90	2.102	2.908 (6)	148.5
N1—H1···O24^{ii}	0.90	1.983	2.874 (6)	170.5
N2—H3···O24^{iii}	0.90	2.052	2.949 (7)	174.2

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

Each member of the Friedel pairs was treated as an independent observation in the least-squares refinement.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1481). Services for accessing these data are described at the back of the journal.

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Doubly Interpenetrating Three-Dimensional Framework Structure of *trans*-$[Cd(dppn)_2\{Ag(CN)_2\}_2]_n$ [dppn = 1,3-Di(4-pyridyl)propane]

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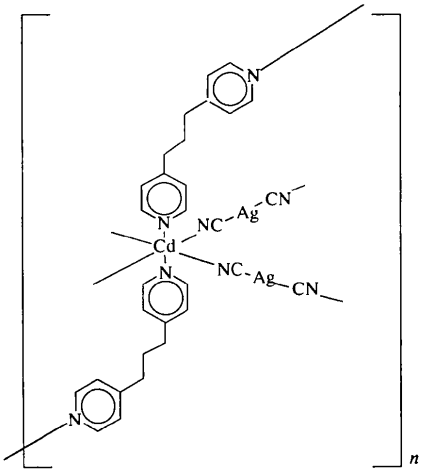
Abstract

In the title compound, poly[bis[μ-1,3-di(4-pyridyl)-propane-*N*:*N*′]cadmium(II)-bis-μ-[dicyanosilver(I)-*N*:*N*′]], $[Cd(C_{13}H_{14}N_2)_2\{Ag(CN)_2\}_2]_n$, the doubly interpenetrating three-dimensional framework structure is built by the stacking of considerably puckered two-dimensional $[Cd\{Ag(CN)_2\}_2]_n$ networks and the 1,3-di(4-pyridyl)-propane bridges from a Cd atom in one network, penetrating through the rhombus meshes of the adjacent networks, to two Ag atoms in the second next networks.

Comment

The two-dimensional (2D) network of $[Cd\{Ag(CN)_2\}_2]_n$ {= $[Cd–(NC–Ag–CN–Cd_{1/4})_4]_n$}, in which the Cd atom is successively linked to four other Cd atoms by the ambidentate $[Ag(CN)_2]^-$ anions, has given a variety of framework structures of $[CdL_2\{Ag(CN)_2\}_2]_n$ by using different *L* ligands, such as pyridine (py), 4-aminopyridine (4-ampy), 4-picoline (4-mepy) and 4,4′-bipyridine (bpy): the three-dimensional (3D) textile interwoven by the 2D networks of *trans*-$[Cd(py)_2\{Ag(CN)_2\}_2]_n$ including benzene or pyrrole guests in the meshes of the network (Soma & Iwamoto, 1996); the doubly interwoven 2D network of *trans*-$[Cd(4\text{-mepy})_2\{Ag(CN)_2\}]_n$ accommodating 4-mepy guests in the meshes of the network (Soma & Iwamoto, 1994); the 2D network of $[Cd(4\text{-ampy})_2\{Ag(CN)_2\}_2]_n$ embracing a pair of the one-dimensional chains of $[Cd\{Ag(CN)_2\}(mea)(4\text{-ampy})\{Ag(CN)_2\}]_n$ in the interlayer space (mea = 2-aminoethanol) (Soma & Iwamoto, 1996); the doubly interpenetrating 3D framework of *trans*-$[Cd(bpy)_2\{Ag(CN)_2\}_2]_n$ (Soma, Yuge & Iwamoto, 1994). The last structure is different from the others in involving the Ag atom in the trigonal three-coordination; the almost flat 2D networks of $[Cd\{Ag(CN)_2\}_2]_n$ stacked along the *a* axis are spanned by the bpy pillars extending from the Cd atom in one network to the Ag atoms in the second next networks to give the doubly interpenetrating 3D framework structure. The present complex, *trans*-$[Cd(dppn)_2\{Ag(CN)_2\}_2]_n$, (1) [dppn = 1,3-di(4-pyridyl)propane], gives the doubly interpenetrating 3D framework structure topologically the same as that observed for *trans*-$[Cd(bpy)_2\{Ag(CN)_2\}_2]_n$, (2), although the bridging ligand dppn in (1) has a flexible and longer skeleton in comparison with the bpy in (2).



(1)

As the *ORTEP*II drawings (Johnson, 1976) in Figs. 2 and 3 show, the 2D network $[Cd\{Ag(CN)_2\}_2]_n$ of (1) has a distorted rhombus mesh cornered by Cd and edged by an –NCAgCN– span similar to those previously observed for the $[CdL_2\{Ag(CN)_2\}_2]_n$ structures (Soma & Iwamoto, 1994, 1996; Soma *et al.*, 1994). The 2D network stacked along the *a* axis is considerably puckered. The dppn spanning the 2D networks from Cd to Ag is twisted at the trimethylene skeleton taking a *trans–gauche* conformation along the linkage of C14—C31—C32—C33—C23 to give a dihedral angle of

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