Table 2. Selected geometric parameters (Å, °)

C(11)—N(1)	1.423 (4)	C(27)—O(22)	1.225 (5)
C(14)—C(17)	1.474 (4)	Cd—Cl(1^i)	2.5673 (7)
C(17)—O(11)	1.282 (4)	Cd—Cl(1^ii)	2.7287 (8)
C(17)—O(12)	1.226 (5)	Cd—Cl(2)	2.6000 (7)
C(21)—N(2)	1.419 (4)	Cd—Cl(2^iii)	2.6126 (8)
C(24)—C(27)	1.466 (5)	Cd—N(1^iv)	2.357 (3)
C(27)—O(21)	1.286 (5)	Cd—N(2^v)	2.344 (3)
N(1)—C(11)—C(12)	118.8 (3)	Cl(1^i)—Cd—Cl(2)	174.71 (3)
N(1)—C(11)—C(16)	121.0 (3)	Cl(1^i)—Cd—Cl(2^iii)	91.79 (2)
C(13)—C(14)—C(17)	118.7 (3)	Cl(1^i)—Cd—N(1^iv)	91.23 (6)
C(15)—C(14)—C(17)	121.9 (3)	Cl(1^i)—Cd—N(2^v)	94.12 (7)
O(11)—C(17)—O(12)	124.9 (3)	Cl(1^ii)—Cd—Cl(2)	87.58 (2)
O(11)—C(17)—C(14)	114.9 (3)	Cl(1^ii)—Cd—Cl(2^iii)	86.11 (3)
O(12)—C(17)—C(14)	120.1 (3)	Cl(1^ii)—Cd—N(1^iv)	169.65 (6)
N(2)—C(21)—C(22)	121.0 (3)	Cl(1^ii)—Cd—N(2^v)	81.37 (7)
N(2)—C(21)—C(26)	119.2 (3)	Cl(2)—Cd—Cl(2^iii)	89.66 (2)
C(23)—C(24)—C(27)	122.6 (3)	Cl(2)—Cd—N(1^iv)	93.99 (6)
C(25)—C(24)—C(27)	118.1 (3)	Cl(2)—Cd—N(2^v)	83.36 (6)
O(21)—C(27)—O(22)	123.4 (3)	Cl(2^iii)—Cd—N(1^iv)	83.67 (7)
O(21)—C(27)—C(24)	116.3 (3)	Cl(2^iii)—Cd—N(2^v)	165.89 (7)
O(22)—C(27)—C(24)	120.5 (3)	N(1^iv)—Cd—N(2^v)	108.96 (9)
Cl(1^i)—Cd—Cl(1^ii)	87.44 (2)		

Symmetry codes: (i) $x-1, y-1, z$; (ii) $-x, 1-y, -z$; (iii) $-x, -y, -z$; (iv) $-x, -y, 1-z$; (v) $x-1, y, z$.

Table 3. Hydrogen-bonding geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O(11)—H(O11)···O(22^i)	0.83 (6)	1.79 (5)	2.608 (4)	169 (6)
O(21)—H(O21)···O(12^ii)	0.92 (6)	1.72 (6)	2.630 (4)	169 (6)
N(1)—H(1N1)···Cl(1^iii)	0.87 (4)	2.63 (4)	3.456 (3)	158 (3)
N(1)—H(2N1)···Cl(1^iv)	0.82 (3)	2.74 (4)	3.448 (2)	145 (4)
N(2)—H(1N2)···Cl(2^v)	0.80 (4)	2.75 (4)	3.293 (3)	126 (4)
N(2)—H(2N2)···Cl(2)	0.85 (4)	2.88 (4)	3.674 (3)	156 (3)
C(12)—H(C12)···Cl(1^vi)	0.89 (4)	2.85 (4)	3.619 (3)	145 (3)

Symmetry codes: (i) $x, 1+y, z$; (ii) $x, y-1, z$; (iii) $-x, 1-y, 1-z$; (iv) $x, y, 1+z$; (v) $1+x, y, z$; (vi) $1-x, 1-y, 1-z$.

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977a). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *SDP* (Enraf–Nonius, 1977b). Program(s) used to solve structure: *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). Program(s) used to refine structure: *SDP*. Molecular graphics: *MOLVIEW* (Cense, 1989) and *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SDP*.

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Caesium 2-(2-Hydroxy-5,5-dimethyl-1,3-dioxo-2-cyclohexyl)-5,5-dimethyl-1,3-cyclohexanedionato(1−) Monohydrate

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Abstract

One of the 5,5-dimethyl-1,3-cyclohexanedione residues of the title compound, $Cs^+.C_{16}H_{21}O_5^-.H_2O$, appears as the anion whereas the other is in the keto form; in the parent molecule the enol predominates. The Cs atom is seven coordinate, with Cs—O distances between 3.008 (3) and 3.396 (4) Å.

Comment

There has been interest recently in the roles of cyclic β-diketones in above-molecular host–guest structures and as potential ligands to metal ions (Braga & Grepioni, 1996). The structures of cyclohexane-1,3-dione (CHD) (Etter, Urbanczyck-Lipkowska, Jahn & Frey, 1986), 4,4-dimethyl-CHD (Barnes, 1996b) and 5,5-dimethyl-CHD [(Ia), dimedone; Singh & Calvo, 1975] show that the CHD derivatives exist in the enol form in the crystal as well as in solution. Hydrogen-bonded inclusion compounds (CHD)₆–benzene (Etter *et al.*, 1986) and 2,5,5-dimethyl-CHD monohydrate [(Ib); Barnes, 1996b] have been reported, and Braga, Grepioni, Bryne & Wolf
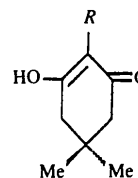
(Ia) $R = H$
(Ib) $R = CH_3$
(Ic) $R = SO_3^-$

(1995) have described a salt in which $Cr(C_6H_6)_2^+$ is encircled by hydrogen-bonded CHD_4^- anions.

Pentane-2,4-dione exists as the chelating acetyl-acetonate anion (II) in a host of metal complexes. Although the cyclic analogues such as (I) are more acidic, the exposed bridgehead C2 position makes chelation sterically impossible, whether C2 is substituted or not. Metal complexes in which the cyclic anions must be either monodentate or bridging are known to exist in solution, but none have been crystallized as yet (Barnes, 1996a). In sodium 5,5-dimethyl-CHD-2-sulphonate, (Ic), CHD exists as the protonated enol, coordinated to the Na atom only *via* the keto O atom. Each Na atom is also bonded to one terminal and four bridging water molecules (Apinitis, 1983).
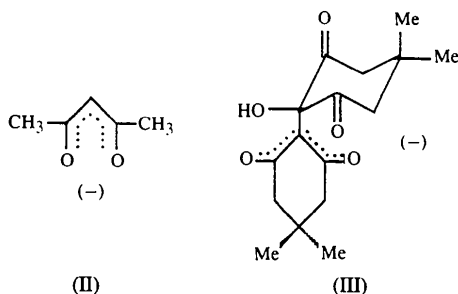
(II)   (III)

An attempt to prepare $NH_4^+.5,5\text{-}CHD^-$ gave an ammonia-free trimer containing a nine-membered ring and an O-protonated furan (Barnes, 1996a). Reaction of 5,5-CHD with Cs_2CO_3 yielded the title compound in which the anion, (III), is derived from two 5,5-CHD molecules fused at the 2,2′ positions (Fig. 1). One 5,5-CHD unit, (IIIa), appears as the enolate anion with the charge delocalized over the O7 and O8 atoms. All the atoms, except C5 and the C9 and C10 methyl groups, are coplanar and the C2 atom is sp^2 hybridized. The other 5,5-CHD entity, (IIIb), has become a chair-shaped α-hydroxy-β-diketone. Anion (III) has a non-
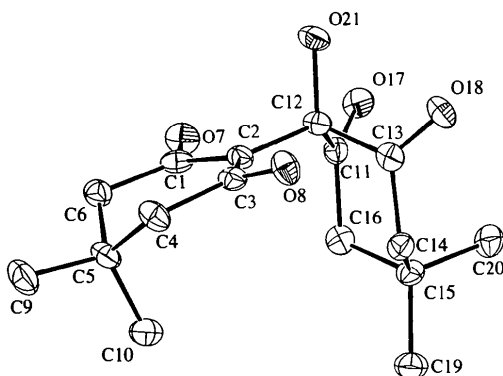

Fig. 1. The 2-(2-hydroxy-5,5-dimethyl-1,3-dioxo-2-cyclohexyl)-5,5-dimethyl-1,3-cyclohexanedionate anion. Ellipsoids are drawn at the 50% probability level.

crystallographic mirror plane through atoms C9, C10, C5, C2, C12, O21, C15, C19 and C20, with an r.m.s. deviation of 0.056 Å. The torsion angles C1—C2—C12—O21 and C1—C2—C12—C11 are 84.0(5) and −39.0(6)°, respectively. The angle between the planes C2, C3, C5, C6 and C12, C13, C15, C16 is 85.8(2)°. Atoms O7, O8, O17 and O18 are coplanar, with an r.m.s. deviation of 0.063 Å. Atoms O7, O17 and O21 are attached to one Cs^+ ion, with atoms O17 and O21 linked to neighbouring Cs^+ ions (Fig. 2).
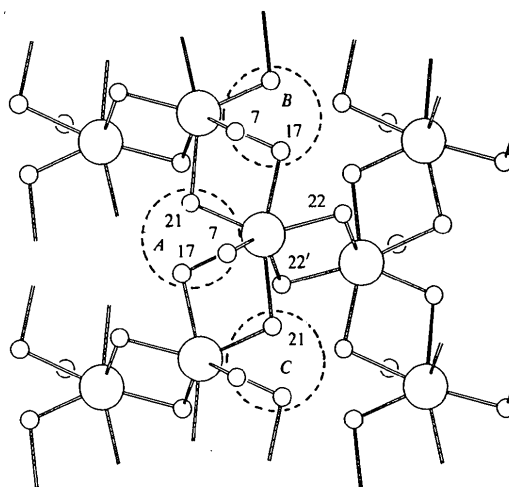

Fig. 2. A partial packing diagram viewed down c showing the coordination of the Cs^+ ion. The C atoms and O8 and O18 atoms have been omitted for clarity. Regions A, B and C are individual anions.

Table 3 shows that the C—C and C—O distances in (IIIa) are very similar to those of (I) in sodium acetylacetonate (Hoser, Kaluski, Januszczyk, Pietrzak & Glowiak, 1983) and that (IIIb) has keto groups separated by C—C single bonds, quite different from the enols 5,5-CHD, which is not substituted in the 2-position, and 5,5-CHD-2-sulphonate.

The Cs^+ ion is coordinated by seven O atoms (3.00 < Cs—O < 3.35 Å) belonging to three anions and two water molecules (Fig. 2). The Cs atom is connected to three adjacent Cs atoms by two O bridges, either by two O22 water molecules or by the O17 and O21′ atoms of different anions. The coordination geometry approximates to a flattened tris-bidentate octahedral propeller capped by the O7 atom. The anions form chains in the c direction cross-linked at unit-cell intervals by water molecules to give layers which are one unit cell thick in the a direction (Fig. 2).

In the presence of the Cs atom, not all the H atoms which might be involved in hydrogen bonding could be observed. There are several O⋯O contacts of about 2.8 Å, of which the most plausible hydrogen bond is O8⋯O22.

C15	0.2696 (3)	1.0972 (4)	0.6915 (5)	0.0214 (10)
C16	0.2567 (3)	0.9721 (4)	0.6857 (5)	0.0236 (11)
O17	0.3876 (2)	0.8748 (3)	0.6887 (4)	0.0254 (8)
O18	0.4251 (2)	1.1203 (3)	0.4600 (4)	0.0268 (8)
C19	0.1905 (3)	1.1507 (4)	0.7456 (7)	0.0288 (13)
C20	0.3448 (3)	1.1255 (5)	0.7987 (6)	0.0278 (11)
O21	0.4137 (3)	0.9040 (3)	0.4022 (4)	0.0268 (9)
O22	0.4477 (2)	0.4038 (3)	0.6437 (4)	0.0298 (8)

Experimental

The reaction of 5,5-CHD (0.02 mol) with Cs_2CO_3 (0.01 mol) in ethanol gave a syrup from which $CsHCO_3$ needles separated. After a year, the title compound grew from the syrup as rhomboidal crystals (yield 12%).

Crystal data

$Cs^+.C_{16}H_{21}O_5^-.H_2O$
$M_r = 443.25$
Monoclinic
$P2_1/c$
$a = 15.890 (2)$ Å
$b = 12.264 (2)$ Å
$c = 9.147 (2)$ Å
$\beta = 93.90 (2)°$
$V = 1778.5 (5)$ Å^3
$Z = 4$
$D_x = 1.655$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters: see below
θ: see below
$\mu = 2.108$ mm^{-1}
$T = 150 (2)$ K
Rhomb
$0.28 \times 0.28 \times 0.26$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 FAST system diffractometer
Area-detector scans
Absorption correction: none
7365 measured reflections
2691 independent reflections

2487 observed reflections
$[I > 2\sigma(I)]$
$R_{int} = 0.0826$
$\theta_{max} = 25.03°$
$h = -14 \rightarrow 17$
$k = -13 \rightarrow 13$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R(F) = 0.0505$
$wR(F^2) = 0.1303$
$S = 1.071$
2691 reflections
216 parameters
H atoms riding, except for H21 and H22 which were refined
$w = 1/[\sigma^2(F_o^2) + (0.0924P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 2.772$ e Å^{-3}
$\Delta\rho_{min} = -1.381$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from International Tables for Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cs1	0.41657 (2)	0.65236 (2)	0.55738 (3)	0.0275 (2)
C1	0.2246 (3)	0.8285 (4)	0.3821 (5)	0.0223 (11)
C2	0.2656 (3)	0.9280 (4)	0.3563 (5)	0.0196 (10)
C3	0.2404 (3)	0.9980 (4)	0.2380 (5)	0.0205 (10)
C4	0.1602 (3)	0.9690 (4)	0.1440 (5)	0.0265 (11)
C5	0.0984 (3)	0.8960 (4)	0.2187 (5)	0.0247 (11)
C6	0.1488 (3)	0.7970 (4)	0.2806 (5)	0.0236 (11)
O7	0.2495 (2)	0.7637 (3)	0.4822 (4)	0.0279 (8)
O8	0.2806 (2)	1.0812 (3)	0.2042 (4)	0.0276 (8)
C9	0.0288 (3)	0.8550 (5)	0.1084 (7)	0.0350 (14)
C10	0.0573 (3)	0.9563 (5)	0.3415 (6)	0.0327 (12)
C11	0.3313 (3)	0.9217 (4)	0.6178 (5)	0.0200 (10)
C12	0.3424 (3)	0.9555 (4)	0.4557 (5)	0.0200 (10)
C13	0.3559 (3)	1.0798 (4)	0.4734 (5)	0.0211 (10)
C14	0.2844 (3)	1.1415 (4)	0.5360 (6)	0.0216 (11)

Table 2. *Selected geometric parameters (Å, °)*

Cs1—O22^i	3.008 (3)	C3—C4	1.529 (6)
Cs1—O7	3.022 (3)	C4—C5	1.524 (7)
Cs1—O17	3.028 (4)	C5—C6	1.540 (7)
Cs1—O22	3.179 (4)	C11—O17	1.213 (6)
Cs1—O21^{ii}	3.233 (4)	C11—C16	1.508 (6)
Cs1—O17^{iii}	3.389 (3)	C11—C12	1.561 (6)
C1—O7	1.256 (6)	C12—O21	1.412 (5)
C1—C2	1.411 (7)	C12—C13	1.547 (7)
C1—C6	1.519 (7)	C13—O18	1.220 (6)
C2—C3	1.417 (7)	C13—C14	1.510 (6)
C2—C12	1.511 (6)	C14—C15	1.555 (7)
C3—O8	1.254 (6)	C15—C16	1.549 (7)
O7—C1—C2	122.9 (5)	C16—C11—C12	115.2 (4)
O7—C1—C6	118.9 (5)	O21—C12—C2	109.1 (4)
C2—C1—C6	118.2 (4)	O21—C12—C13	111.6 (4)
C1—C2—C3	122.8 (4)	C2—C12—C13	112.6 (4)
C1—C2—C12	117.1 (4)	O21—C12—C11	110.8 (4)
C3—C2—C12	119.9 (4)	C2—C12—C11	111.9 (4)
O8—C3—C2	124.0 (4)	C13—C12—C11	100.7 (4)
O8—C3—C4	118.0 (4)	O18—C13—C14	122.8 (5)
C2—C3—C4	118.0 (4)	O18—C13—C12	120.6 (4)
C5—C4—C3	115.0 (4)	C14—C13—C12	115.5 (4)
C4—C5—C6	107.1 (4)	C13—C14—C15	109.7 (4)
C1—C6—C5	113.3 (4)	C16—C15—C14	110.0 (4)
O17—C11—C16	123.1 (4)	C11—C16—C15	108.3 (4)
O17—C11—C12	120.5 (4)		

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

Table 3. *Comparison of bond distances (Å) in anion (III) with the pentane-2,4-dionate anion and 5,5-CHD enols*

	(IIIa) (anion)	(II) (anion)*	(IIIb) (keto)	(Ia) (enol)†	(Ic) (enol)‡
C1—C2	1.411 (7)	1.407	1.561 (6)	1.351 (3)	1.295
C2—C3	1.417 (7)	1.410	1.547 (7)	1.418 (3)	1.472
C1—O7	1.256 (6)	1.266	1.213 (6)	1.326 (3)	1.356
C3—O8	1.254 (6)	1.265	1.220 (6)	1.246 (3)	1.226

* Hoser *et al.* (1983). † Singh & Calvo (1975). ‡ Apinitis (1983).

An area-detector system was used. Cell dimensions were refined from 250 reflections selected from two regions 90° apart and 5° wide at $\kappa = 90°$. No conventional diffraction standards were used and intensities were monitored between detector frames. No sample deterioration was observed. The final difference map has a peak of 2.8 e Å^{-3} very close to the Cs atom and the next highest peak of 1.2 e Å^{-3} is in a chemically impossible position near the inversion centre at 0.5,0,0.5 and close to the O18, C12 and C13 atoms.

Data collection: *MADNES* (Enraf–Nonius, 1990). Cell refinement: *MADNES*. Data reduction: *MADNES*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLATON92* (Spek, 1992a) and *PLUTON92* (Spek, 1992b). Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: LI1149). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis[bis(2-pyridyl-N) sulfide]palladium(II) Bis(tetrafluoroborate)

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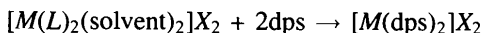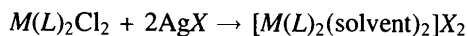
(Received 7 March 1996; accepted 9 May 1996)

Abstract

The title compound, [Pd(dps)₂](BF₄)₂ (dps = di-2-pyridyl sulfide, $C_{10}H_8N_2S$), has been synthesized by the reaction of dps with the solvated species [Pd(L_2)(acetone)₂](BF₄)₂ (L = 1,5-cyclooctadiene) at low temperature. Its structure has been determined by X-ray diffraction. In the [Pd(dps)₂]²⁺ cation, the Pd atom lies on a crystallographic inversion centre and exhibits a square-planar geometry involving four pyridine N atoms of both dps molecules acting as chelating ligands [Pd—N1 2.032 (2) and Pd—N2 2.026 (3) Å]. The two equal six-membered chelate rings are in boat conformations.

Comment

As a continuation of our investigation on the coordination chemistry of flexible bipyridine-like ligands (Tresoldi, Piraino, Rotondo & Faraone, 1991; Tresoldi, Rotondo, Piraino, Lanfranchi & Tiripicchio, 1992; De Munno *et al.*, 1993; Bruno, Nicolò, Lo Schiavo, Sinicropi & Tresoldi, 1995), we have examined the reactions of di-2-pyridyl sulfide (dps) with solvated species formed *in situ* by the reaction of the palladium(II)–diene or platinum(II)–diene complexes. Recently, some crystal structure determinations (Tresoldi *et al.*, 1992; De Munno *et al.*, 1993; Bruno *et al.*, 1995) have shown that the chelate ring of coordinated flexible bipyridine-like ligands adopts a boat conformation in the solid state, while NMR studies have shown that these flexible ligands favour dynamic processes in solution (Tresoldi *et al.*, 1991, 1992; De Munno *et al.*, 1993). Concerning dps and its derivatives, it is known that the free dps ligand adopts three planar (*N,N*-inside, *N,N*-outside, *N*-inside/*N*-outside) and several twisted conformations in the three states of matter (Chachaty, Pappalardo & Scarlata, 1976; Amato, Bandoli, Grassi, Pappalardo & Scarlata, 1989; Bombieri, Forsellini & Graziani, 1978; Colonna, Distefano, Galasso, Pappalardo & Scarlata, 1977) which may be present when dps is bonded to the metal as a chelate or bridging ligand. The route followed for the synthesis of the dicationic complexes [M^{II}(dps)₂](BF₄)₂ [M = Pd, (I), and Pt, (II)] is

$$M(L)_2Cl_2 + 2AgX \rightarrow [M(L)_2(solvent)_2]X_2$$

$$[M(L)_2(solvent)_2]X_2 + 2dps \rightarrow [M(dps)_2]X_2$$

where M is Pd or Pt, L is 1,5-cyclooctodiene or dicyclopentadiene, and X is BF_4^- or PF_6^-. The procedure involves the *in situ* formation of solvated species followed by the displacement of the diene and the coordinated solvent by dps. The final reactions proceed slowly at 255 K and complexes (I) and (II) are obtained in high yields, whereas at higher temperature, large decomposition occurs.

(I)
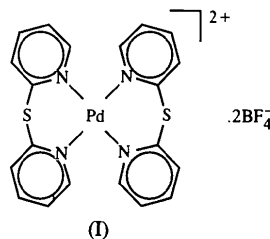
Dissolving (I) in boiling methanol and filtering to eliminate black powder allowed a highly crystalline product to be obtained. The compounds are white, stable for several months in the solid state and slightly soluble in methanol at 323 K and stable for several days. The IR spectra of (I) and (II) show a very strong band in the range 1590–1594 cm⁻¹ and two in the range