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

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

	x	y	z	$U_{\rm eq}$
Cr	0.66754 (3)	0.28763 (2)	0.29828 (4)	0.0175
O1	0.4895 (1)	0.2735 (1)	0.0732 (2)	0.0228
O2	0.5132 (1)	0.2146 (1)	0.3704 (2)	0.0233
O3	0.8149 (1)	0.3432 (1)	0.1985 (2)	0.0235
O4	0.7253 (1)	0.1328 (1)	0.1982 (2)	0.0232
O5	0.6434 (1)	0.4476 (1)	0.4198 (2)	0.0241
O6	0.8333 (1)	0.3051 (1)	0.5372 (2)	0.0260
O7	0.2402 (1)	0.2042 (1)	−0.0532 (2)	0.0280
O8	0.2656 (2)	0.1401 (1)	0.2568 (2)	0.0277
O9	0.9820 (1)	0.2742 (1)	0.0651 (2)	0.0263
O10	0.9001 (2)	0.0586 (1)	0.0808 (2)	0.0285
O11	0.7295 (2)	0.5788 (1)	0.6969 (2)	0.0326
O12	0.9318 (2)	0.4314 (1)	0.8170 (2)	0.0321
O*W*1	0.0947 (2)	−0.0831 (1)	0.2890 (2)	0.0376
O*W*2	0.3591 (2)	0.0348 (2)	0.6283 (3)	0.0501
O*W*3	1.2602 (2)	0.4579 (1)	−0.0621 (2)	0.0389
O*W*4	0.6322 (2)	0.1801 (2)	0.7288 (2)	0.0475
O*W*5	0.4983 (2)	0.3923 (2)	0.7759 (3)	0.0508
O*W*6	0.1135 (2)	0.1890 (2)	0.5735 (2)	0.0476
C1	0.3649 (2)	0.2252 (1)	0.0726 (2)	0.0191
C2	0.3784 (2)	0.1894 (1)	0.2477 (2)	0.0196
C3	0.8838 (2)	0.2628 (2)	0.1304 (2)	0.0197
C4	0.8334 (2)	0.1391 (1)	0.1351 (2)	0.0199
C5	0.7289 (2)	0.4858 (2)	0.5899 (2)	0.0216
C6	0.8424 (2)	0.4013 (2)	0.6580 (2)	0.0222
Li1	1.0705 (4)	0.1122 (3)	−0.0148 (5)	0.0298
Li2	1.1061 (4)	0.4032 (3)	0.0376 (5)	0.0322
Li3	0.2077 (5)	0.0767 (3)	0.4282 (5)	0.0375

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

Cr—O1	1.975 (1)	O6—C6	1.282 (2)
Cr—O2	1.977 (2)	O7—C1	1.229 (2)
Cr—O3	1.982 (2)	O8—C2	1.222 (2)
Cr—O4	1.987 (1)	O9—C3	1.218 (3)
Cr—O5	1.969 (1)	O10—C4	1.227 (2)
Cr—O6	1.984 (1)	O11—C5	1.218 (2)
O1—C1	1.278 (2)	O12—C6	1.225 (2)
O2—C2	1.282 (2)	C1—C2	1.545 (3)
O3—C3	1.280 (2)	C3—C4	1.550 (3)
O4—C4	1.279 (3)	C5—C6	1.551 (3)
O5—C5	1.286 (2)		
O1—Cr—O2	82.68 (6)	O5—Cr—O6	82.77 (5)
O3—Cr—O4	82.74 (6)		

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989*a*). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989*b*). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1109). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Mononuclear Tetraphenylphosphonium Oxochromium Complexes: (PPh$_4$)[Cr$^{\rm V}$O$_3$(H$_2$O)] and (PPh$_4$)[Cr$^{\rm VI}$O$_3$(OH)]

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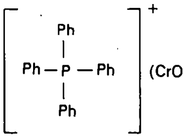
Abstract

The crystal structures of two isostructural compounds, tetraphenylphosphonium aquatrioxochromium(V), (1), [P(C$_6$H$_5$)$_4$][CrO$_3$(H$_2$O)], and tetraphenylphosphonium hydroxytrioxochromium(VI), (2), [P(C$_6$H$_5$)$_4$][CrO$_3$(OH)], have been determined by single-crystal X-ray diffraction. (1) is paramagnetic and (2) is diamagnetic. The P atoms in the cations of both structures lie on the crystallographic $\bar{4}$ symmetry axis at the unit-cell origin. The anions are found near the $\bar{4}$ sites at 0, $\frac{1}{2}$, $\frac{1}{4}$ and are fourfold disordered. The Cr atoms in the anions have a deformed tetrahedral coordination geometry with three normal Cr═O bonds [average 1.56 (3) Å] and a fourth Cr—O(aqua) or Cr—O(hydroxy) bond [2.07 (3) and 2.02 (3) Å in compounds (1) and (2), respectively].

Comment

High-valent chromium complexes featuring oxo ligands are of use in catalytic oxidation reactions. The title chromium compounds were obtained during our studies of oxo- and sulfido-bridged metal complexes (Bhattacharya *et al.*, 1991). Preliminary spectroscopic data and their resemblance to oxochromium salts (Elisabeth, 1975) indicated the presence of mononuclear oxyanions of chromium in high formal oxidation states [(V) and (VI)]. Though the existence of $[Cr^{VI}O_3(OH)]^-$ has been reported (Rosenblum & Holt, 1972), no X-ray structure analysis of any complex containing this anion has been published so far. Here we report the determination of the structures of the title Cr^V and Cr^{VI} species containing the $[CrO_3X]^-$ anion, where $X = H_2O$ or OH^-.


$$\left[\begin{array}{c} Ph \\ | \\ Ph-P-Ph \\ | \\ Ph \end{array}\right]^+ (CrO_3X)^-$$

(1) $X = H_2O$
(2) $X = OH$

In view of the high degree of disorder affecting the anions in $(PPh_4)[Cr^VO_3(H_2O)]$, (1), and $(PPh_4)[Cr^{VI}O_3(OH)]$, (2), the oxidation state of chromium in each compound was confirmed by room-temperature magnetic susceptibility and/or EPR measurments carried out on powder samples. (1) is paramagnetic with an observed magnetic moment of 1.76 μ_B at 295 K, consistent with the d^1 configuration of pentavalent chromium. Room-temperature EPR measurements for (1) gave $\bar{g} = 1.975$, corroborating this conclusion (Mitewa & Bontchev, 1985). On the other hand, (2) shows a feeble diamagnetic susceptibility, as expected in case of hexavalent chromium.

The title compounds are isostructural and consist of discrete $(PPh_4)^+$ cations and $[CrO_3X]^-$ anions [$X = H_2O$ in (1) and OH in (2)] held together by electrostatic forces. In both compounds, the P atoms of the cations lie on the crystallographic $\bar{4}$ symmetry axis at the origin of the unit cell and the cations display almost ideal tetrahedral geometry. The anions are located near the $\bar{4}$ site at 0, $\frac{1}{2}$, $\frac{1}{4}$ and are fourfold disordered. The coordination polyhedra around the metal atoms in the anions are distorted tetrahedra resulting from the asymmetric nature of the ligands [three oxo groups and one aqua group in (1), and three oxo groups and a hydroxy group in (2)]. The distortion of the tetrahedra is reflected by the bond angles at the metal centres. The Cr=O distances, which range from 1.54 (2) to 1.59 (3) Å in (1) and from 1.54 (2) to 1.58 (2) Å in (2), are similar to those found in analogous structures (Noh, Heintz, Haggerty, Rheingold & Theopold, 1992; Nishino & Kochi, 1990; Krumpolc, Deboer & Rocek, 1978). The observed Cr^V—O(aqua) distance of 2.07 (3) Å in (1) is slightly longer than the Cr^{VI}—O(hydroxy) distance of 2.02 (3) Å in (2). Cr—O(aqua) bonds have also been reported to be longer than Cr—O(hydroxy) bonds for Cr^{III} complexes (Larsen, Nielsen & Trabjerg, 1983). The phenyl rings, in which the C—C bond distances range from 1.36 (2) to 1.44 (2) Å, are essentially planar.

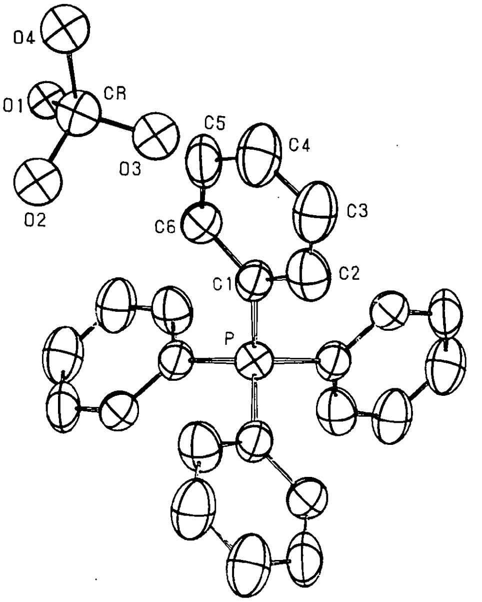


Fig. 1. *ORTEP*II (Johnson, 1976) view of (1) showing the atom-labelling scheme. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Compound (1)

Crystal data

$[(C_6H_5)_4P][CrO_3(H_2O)]$
$M_r = 457.41$
Tetragonal
$I\bar{4}$
$a = 12.250$ (3) Å
$c = 7.309$ (2) Å
$V = 1096.8$ (5) Å^3
$Z = 2$
$D_x = 1.385$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 20 reflections
$\theta = 15–30°$
$\mu = 5.281$ mm^{-1}
$T = 293$ (2) K
Parallelepiped
0.4 × 0.25 × 0.15 mm
Deep green

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical
$T_{min} = 0.685$, $T_{max} = 0.856$
304 measured reflections
304 independent reflections
296 observed reflections
[$I > 2\sigma(I)$]

$\theta_{max} = 68.90°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 8$
3 standard reflections
monitored every 50 reflections
intensity variation: <2%

Refinement

Refinement on F^2
$R(F) = 0.0765$
$wR(F^2) = 0.1841$
$S = 1.239$

$(\Delta/\sigma)_{max} = 0.443$
$\Delta\rho_{max} = 0.293$ e Å^{-3}
$\Delta\rho_{min} = -0.246$ e Å^{-3}
Extinction correction: none

296 reflections
83 parameters
H-atom parameters not refined
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.1311P)^2 + 0.5169P]$
where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)

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

$U_{\rm iso}$ for O atoms, $U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for all others.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Cr1†	0.0186 (15)	0.465 (3)	0.285 (3)	0.137 (14)
O1†	0.0362 (13)	0.5102 (18)	0.479 (3)	0.045 (6)
O2†	0.124 (2)	0.403 (4)	0.231 (8)	0.108 (19)
O3†	−0.069 (3)	0.371 (4)	0.312 (8)	0.131 (25)
O4†	−0.051 (3)	0.570 (3)	0.097 (6)	0.105 (12)
P1	0	0	0	0.061 (2)
C1	0.0966 (8)	0.0651 (8)	0.144 (2)	0.069 (3)
C2	0.0571 (11)	0.1317 (12)	0.278 (2)	0.091 (4)
C3	0.1321 (17)	0.1841 (12)	0.403 (3)	0.116 (5)
C4	0.2412 (12)	0.1654 (13)	0.383 (3)	0.108 (5)
C5	0.2818 (11)	0.0996 (12)	0.246 (2)	0.093 (4)
C6	0.2093 (9)	0.0497 (8)	0.124 (2)	0.081 (4)

† Site occupancy = 0.25.

Table 2. *Selected geometric parameters* (Å, °) *for* (1)

Cr1—O1	1.54 (2)	C1—C6	1.401 (15)
Cr1—O2	1.56 (3)	C2—C3	1.44 (2)
Cr1—O3	1.59 (3)	C3—C4	1.36 (2)
Cr1—O4	2.07 (3)	C4—C5	1.38 (2)
P1—C1	1.773 (12)	C5—C6	1.40 (2)
C1—C2	1.37 (2)		
O1—Cr1—O2	107 (2)	C2—C1—P1	117.3 (9)
O1—Cr1—O3	104 (2)	C6—C1—P1	122.3 (10)
O1—Cr1—O4	117 (3)	C1—C2—C3	119.6 (13)
O2—Cr1—O3	104 (2)	C4—C3—C2	118.8 (16)
O2—Cr1—O4	119 (3)	C5—C4—C3	122.0 (14)
O3—Cr1—O4	105 (2)	C4—C5—C6	119.3 (13)
C2—C1—C6	120.3 (11)	C5—C6—C1	119.9 (12)

Compound (2)

Crystal data

$[(C_6H_5)_4P][CrO_3(OH)]$
$M_r = 456.40$
Tetragonal
$I\bar{4}$
$a = 12.306$ (3) Å
$c = 7.336$ (2) Å
$V = 1110.9$ (5) Å^3
$Z = 2$
$D_x = 1.364$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 15 reflections
$\theta = 10$–$18°$
$\mu = 0.614$ mm^{-1}
$T = 293$ (2) K
Parallelepiped
0.45 × 0.30 × 0.15 mm
Deep yellow

Data collection

Hilger & Watts diffractometer

ω–2θ scans
$\theta_{\rm max} = 23.25°$

Absorption correction: empirical
$T_{\rm min} = 0.784$, $T_{\rm max} = 0.882$
431 measured reflections
431 independent reflections
372 observed reflections $[I > 2\sigma(I)]$

$h = 0 \rightarrow 13$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 7$
3 standard reflections monitored every 50 reflections
intensity variation: <3%

Refinement

Refinement on F^2
$R(F) = 0.0732$
$wR(F^2) = 0.1810$
$S = 1.192$
401 reflections
82 parameters
H-atom parameters not refined
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.1423P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.115$
$\Delta\rho_{\rm max} = 0.257$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.156$ 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)
Absolute configuration: Flack (1983)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (2)

$U_{\rm iso}$ for O atoms, $U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for all others.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Cr1†	0.0103 (10)	0.4599 (5)	0.2951 (7)	0.086 (2)
O1†	0.0372 (12)	0.5117 (15)	0.481 (3)	0.074 (5)
O2†	0.1198 (17)	0.407 (2)	0.227 (5)	0.106 (10)
O3†	−0.068 (3)	0.364 (3)	0.329 (6)	0.154 (18)
O4†	−0.047 (3)	0.567 (3)	0.110 (6)	0.136 (11)
P1	0	0	0	0.0608 (11)
C1	0.0963 (5)	0.0648 (5)	0.1456 (11)	0.065 (2)
C2	0.0608 (7)	0.1305 (8)	0.2828 (15)	0.092 (3)
C3	0.1289 (10)	0.1870 (8)	0.3999 (17)	0.107 (3)
C4	0.2384 (8)	0.1679 (9)	0.3835 (18)	0.107 (3)
C5	0.2779 (7)	0.0995 (7)	0.2491 (18)	0.095 (3)
C6	0.2089 (6)	0.0503 (6)	0.1297 (13)	0.077 (2)

† Site occupancy = 0.25.

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

Cr1—O1	1.54 (2)	C1—C6	1.402 (9)
Cr1—O2	1.58 (2)	C2—C3	1.387 (13)
Cr1—O3	1.55 (3)	C3—C4	1.37 (2)
Cr1—O4	2.02 (3)	C4—C5	1.38 (2)
P1—C1	1.784 (7)	C5—C6	1.363 (12)
C1—C2	1.362 (12)		
O1—Cr1—O2	106 (1)	C2—C1—P1	119.7 (5)
O1—Cr1—O3	108 (2)	C6—C1—P1	123.3 (6)
O1—Cr1—O4	114 (2)	C1—C2—C3	124.2 (8)
O2—Cr1—O3	106 (2)	C2—C3—C4	116.9 (10)
O2—Cr1—O4	111 (2)	C5—C4—C3	120.8 (9)
O3—Cr1—O4	113 (2)	C6—C5—C4	120.6 (7)
C2—C1—C6	117.0 (7)	C5—C6—C1	120.4 (8)

Axial photographs and the intensity distribution indicated that both crystals belonged to the tetragonal system (Laue symmetry $4/m$). The lattice centring, I, was established by the systematic absences in hkl for $h + k + l = 2n + 1$. Data collection, cell refinement and data reduction were carried out using *CAD*-4 *Software* (Enraf–Nonius, 1989) and Hilger & Watts software for compounds (1) and (2), respectively. The data were corrected for Lorentz, polarization and absorption effects. Attempts to solve the structures using routine Patterson (*SHELX76*; Sheldrick, 1976) and direct methods (*MULTAN88*; Debaerdemaeker *et al.*, 1988) failed because of an inability to determine the Cr-atom positions uniquely, while the P atoms in the cations could be located easily. The structures could only be solved by a painstakingly meticulous interpretation of the Patterson maps coupled with judicious symmetry considerations. The P atoms in the cations were placed at the origin while the two anions in the unit cell of each compound were statistically distributed among the eight possible sites leading to fourfold disordering of the Cr-atom environments. Full-matrix least-squares refinements on F^2 using *SHELXL93* (Sheldrick, 1994) with anisotropy for Cr, P and phenyl C atoms and isotropic displacement parameters for O atoms with fractional occupancies were carried out. H atoms were placed in geometrically calculated positions and were not refined. Program used for geometric calculations: *PARST* (Nardelli, 1983). Software used to prepare material for publication: *SHELXL93*. The calculations were performed using a VAX3400 computer at the Computer Centre, Indian Association for the Cultivation of Science.

The authors wish to thank Professor R. G. Bhattacharya, Jadavpur University, for providing the crystals and many helpful discussions. They are also indebted to Professors R. Bhattacharya, D. Ghosh and A. Pal, IACS, Calcutta, for magnetic susceptibility and EPR studies.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HE1008). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 1404–1406

Bis[tris(tetrahydrofuran)lithium(1+)] Bis(μ-diphenylphosphanido)bis(tetracarbonylmolybdate)(2−)

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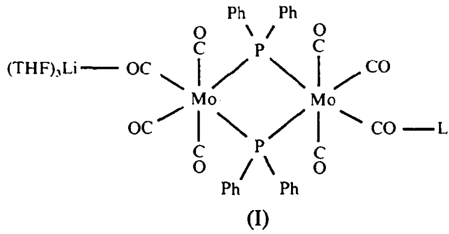
Abstract

In the dianion $[Mo_2(\mu\text{-}PPh_2)_2(CO)_8]^{2-}$, two Mo centres are 4.100 (1) Å apart and are bridged asymmetrically by two diphenylphosphanido ligands. Each metal atom is also coordinated by four terminal CO ligands. There are strong interactions between the dianion and the two $[Li(thf)_3]^+$ cations (thf = C_4H_8O). The Li ions are ligated by one equatorial carbonyl O atom from each Mo centre.

Comment

Binuclear transition metal complexes $[M_2(\mu\text{-}PR_2)_2(CO)_8]$ (M = Cr, Mo, W) have been investigated extensively with respect to their synthesis, bonding, chemical and electrochemical properties (Linck & Nassimbeni, 1973; Treichel, Dean & Douglas, 1972; Keiter *et al.*, 1992). It has been demonstrated that the complex $[W_2(\mu\text{-}PPh_2)_2(CO)_8]$ undergoes two-electron reduction with cleavage of the M—M bond to give the dianion $[W_2(\mu\text{-}PPh_2)_2(CO)_8]^{2-}$ (Shyu, Calligaris, Nardin & Wojcicki, 1987). We have prepared the lithium salt of $[Mo_2(\mu\text{-}PPh_2)_2(CO)_8]^{2-}$ by reaction of $[Mo(CO)_4(Ph_2PLi)_2]$ with $SmCl_3$ in thf. Yellow crystals of $[Li(thf)_3]_2[Mo_2(\mu\text{-}PPh_2)_2(CO)_8]$, (I), suitable for X-ray analysis were obtained by cooling a saturated solution of thf. The present structure is isomorphous with its tungsten analogue



(I)

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