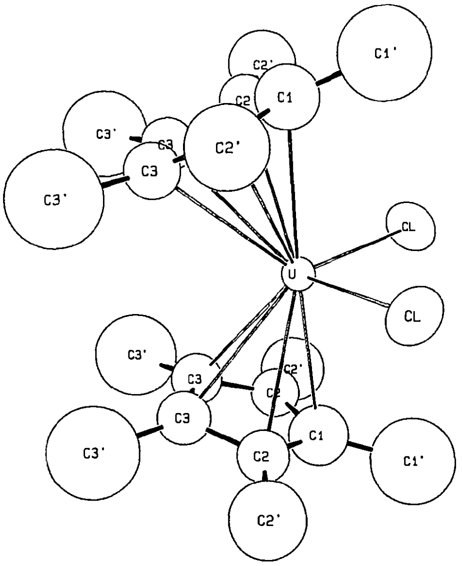

Fig. 1. The molecular structure of the U complex. The Th complex is isostructural. Thermal ellipsoids are drawn at the 50% probability level (Johnson, 1976).

1984). The An—C distances (Table 2) are comparable to the average value of 2.74 (2) Å in [U{$C_5(CH_3)_5$}$Cl_2$($C_3H_4N_2$)] (Eigenbrot & Raymond, 1982), and 2.82 Å in [Th{$C_5(CH_3)_5$}Cl{$COCH_2C(CH_3)_3$}] (Fagan *et al.*, 1980). The angles Cp—An—Cp (where Cp = ring centroid) are in both cases much larger than the angle Cl—An—Cl, presumably as a result of intramolecular contact of methyl groups.

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# Structures of Two Molybdenum Oxo Complexes [$MoOCl_3(OPPh_3)_2$] and [$MoOI(dmpe)_2$]I

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**Abstract.** Trichlorooxobis(triphenylphosphine oxide-*O*)molybdenum(V), [$MoOCl_3(C_{18}H_{15}OP)_2$], $M_r$ = 774.88, monoclinic, $C2/c$, $a$ = 14.041 (2), $b$ = 13.126 (1), $c$ = 19.210 (4) Å, $\beta$ = 96.08 (1)°, $V$ = 3521 (2) Å$^3$, $Z$ = 4, $D_x$ = 1.46 g cm$^{-3}$, $\lambda$(Cu $K\alpha$) = 1.54178 Å, $\mu$ = 64.06 cm$^{-1}$, $F$(000) = 1572, $T$ = 293 K, $R$ = 0.064 for 2054 observed reflections. The compound was obtained by refluxing [$Cp_2Mo_2Cl_5$]$^-$ and $POPh_3$ in THF. Electron paramagnetic resonance (EPR) spectral parameters (X-band, room temperature, $CH_2Cl_2$): $g$ = 1.938, $a_{Mo}$ = 54.7 G. The compound adopts an octahedral geometry with *cis* phosphine oxide ligands and a meridional arrangement of the three Cl atoms. There is disorder

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between the O and Cl atoms which are related by a crystallographic twofold axis. Bis[1,2-bis(dimethylphosphino)ethane-*P*,*P*′]iodooxomolybdenum(IV) iodide, [MoOI(C$_6$H$_{16}$P$_2$)$_2$]I, $M_r$ = 666.03, monoclinic, $P2_1/n$, $a$ = 11.310 (2), $b$ = 15.580 (6), $c$ = 13.741 (3) Å, $\beta$ = 96.58 (2)°, $V$ = 2405 (2) Å$^3$, $Z$ = 4, $D_x$ = 1.84 g cm$^{-3}$, $\lambda$(Cu $K\alpha$) = 1.54178 Å, $\mu$ = 276.67 cm$^{-1}$, $F$(000) = 1280, $T$ = 296 K, $R$ = 0.056 for 2343 observed reflections. The cation has *trans* geometry and exhibits a fairly long Mo—I distance of 2.964 (2) Å.

**Introduction.** Cyclopentadienyl-containing Mo$^{III}$ complexes of general formula [CpMo$X_2L_2$] ($X$ = halide, $L$ = neutral 2e-donor ligand) have been obtained by two main synthetic procedures: (i) addition of neutral ligands (typically phosphines) to the precursor [Cp$_2$Mo$_2$Cl$_5$]$^-$ (Krueger, Owens & Poli, 1990), or (ii) addition of Cp-delivering agents to solutions containing [Mo$X_3$(thf)$_3$] and phosphines (Krueger, Poli, Rheingold & Staley, 1989). In both instances, exposure of the solutions to air results in the formation of oxo compounds with loss of the cyclopentadienyl ring. We report here the structures of two such compounds.

[MoOCl$_2$(OPPh$_3$)$_2$] was obtained from [Cp$_2$Mo$_2$Cl$_5$]$^-$ and POPh$_3$ under an inert atmosphere. The purpose of the reaction was the preparation of [CpMoCl$_2L_2$] derivatives with $L$ = phosphine oxide. Presumably the phosphine oxide O atom serves as the oxidizing agent in this case.

[MoOI(dmpe)$_2$]I was obtained during an attempt to synthesize [CpMoI$_2$(dmpe)] by the second procedure as described in more detail elsewhere (Owens & Poli, 1991). Although the synthetic strategy failed to afford any crystalline material, exposure of the solution to air afforded X-ray-quality crystals of the Mo$^{IV}$–oxo compound.

**Experimental.** Crystals of both [MoOCl$_3$(OPPh$_3$)$_2$] and [MoOI(dmpe)$_2$]I were obtained by diffusion of *n*-heptane into CH$_2$Cl$_2$ solutions. In both cases, a single crystal was glued onto the tip of a thin glass fiber with epoxy cement and examined on an Enraf–Nonius CAD-4 four-circle automated diffractometer equipped with graphite-monochromated Cu radiation. Crystal parameters and basic information on data collection and refinement are presented in Table 1. Both structures were solved by direct methods and refined by alternating full-matrix least-squares cycles (on $F$) and difference Fourier maps.

Table 1. *Crystallographic data*

| | [MoOCl$_3$(OPPh$_3$)$_2$] | [MoOI(dmpe)$_2$]I |
|---|---|---|
| Systematic absences | *hkl*: $h + l \neq 2n$; *h*0*l*: $l \neq 2n$ | 0*k*0: $k \neq 2n$; *h*0*l*: $h + l \neq 2n$ |
| Crystal size (mm) | 0.5 × 0.4 × 0.4 | 0.2 × 0.2 × 0.4 |
| Orientation reflections: No., $2\theta$ range (°) | 25, 30–50 | 25, 40–60 |
| Absorption correction | $\psi$ scans | $\psi$ scans |
| Transmission factors: maximum, minimum | 1.000, 0.808 | 1.000, 0.380 |
| Scan method | $\omega$–$2\theta$ | $\omega$–$2\theta$ |
| Data collection: $2\theta$ range (°) | 4–110 | 5–110 |
| Range of $h$, $k$, $l$ | +14, +13, ±20 | +12, −16, ±14 |
| Standard reflections: No., variation (%) | 3, +1.6 | 3, −0.4 |
| No. of reflections measured | 2449 | 3350 |
| No. of unique reflections | 2331 | 3163 |
| $R_{\rm merge}$ (%) | 0.7 | 3.9 |
| No. of unobserved reflections [$F_o < 6\sigma(F_o)$] | 395 | 820 |
| No. of parameters refined | 213 | 181 |
| $R$ | 0.064 | 0.056 |
| $wR$ [$w = 1/\sigma^2(\lvert F_o\rvert)$] | 0.118 | 0.085 |
| $S$ | 2.65 | 1.59 |
| Largest shift/e.s.d. in final cycle | 0.39 | 0.36 |
| Largest peak in final difference Fourier map (e Å$^{-3}$) | 0.45 | 1.16 |

For the [MoOCl$_3$(OPPh$_3$)$_2$] compound, the centrosymmetric space group was selected on the basis of intensity statistics. The atom Cl(2), originally treated at full occupancy, refined with a large thermal parameter. Furthermore, a strong peak along the Mo—Cl(2) vector (*ca* 1.85 Å away from Mo) was found in the difference Fourier map. Chemical considerations (EPR spectroscopy) indicated that the metal is pentavalent, therefore needing three valencies in the two coordination positions related by the twofold axis. The obvious choice was disorder between an oxo and a chloro ligand which are related by the twofold axis, thus both atoms were assigned occupancy of 0.5. The model refined well, with no significant correlation between parameters of the anisotropically refined Cl(2) and O atoms. The symmetry-related disorder could in principle be eliminated by moving to the *Cc* space group. A refinement in this space group was attempted, but high correlation was present between most pairs of variables that would be related by symmetry in *C*2/*c*, and convergence could not be achieved. The molecule probably crystallizes in the centrosymmetric space group because packing is dominated by the van der Waals interactions between the phenyl rings in the phosphine oxide ligands.

For both structures, all non-H atoms were refined anisotropically, and H atoms were included as isotropic contributions at calculated positions and used for structure factor calculations, but not refined. All software and the sources of the scattering factors are contained in the *TEXSAN* program library (Molecular Structure Corporation, 1985).

**Discussion.** Given the well known oxophilicity of Mo, it is not surprising that O$_2$ or other oxygen-containing reagents decompose lower valent Mo complexes to form products that contain the Mo═O functionality. It is more surprising that, during this process, the cyclopentadienyl ligand is lost, since high-oxidation-state Mo complexes that contain both Cp and O ligands are known (Cousins & Green, 1969).

Table 2. *Positional parameters and equivalent isotropic thermal parameters* (Å$^2$) *for* [$MoOCl_3(OPPh_3)_2$]

$B_{eq}$ is defined as one third of the trace of the orthogonalized $B_{ij}$ tensor.

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| Mo | 1.0000 | 0.37391 (6) | $\frac{1}{4}$ | 4.91 (6) |
| Cl(1) | 1.1610 (1) | 0.3558 (2) | 0.3000 (1) | 5.5 (1) |
| Cl(2)* | 1.0461 (4) | 0.5009 (4) | 0.1859 (2) | 4.9 (2) |
| P(1) | 1.0207 (1) | 0.1797 (1) | 0.12445 (8) | 4.14 (9) |
| O(1) | 1.0322 (3) | 0.2559 (4) | 0.1834 (2) | 4.7 (2) |
| O(2)* | 0.977 (1) | 0.452 (1) | 0.3282 (8) | 6.4 (7) |
| C(10) | 1.1378 (5) | 0.1493 (6) | 0.1010 (3) | 4.1 (3) |
| C(11) | 1.2128 (6) | 0.1454 (7) | 0.1537 (5) | 6.2 (4) |
| C(12) | 1.3048 (7) | 0.1164 (8) | 0.1354 (7) | 7.6 (6) |
| C(13) | 1.3175 (7) | 0.0915 (9) | 0.0675 (6) | 7.4 (5) |
| C(14) | 1.2428 (8) | 0.094 (1) | 0.0183 (7) | 9.7 (7) |
| C(15) | 1.1518 (7) | 0.1226 (8) | 0.0332 (5) | 6.7 (5) |
| C(20) | 0.9624 (5) | 0.0647 (6) | 0.1489 (3) | 4.4 (3) |
| C(21) | 1.0030 (6) | −0.0300 (7) | 0.1423 (5) | 6.1 (4) |
| C(22) | 0.951 (1) | −0.1135 (7) | 0.1591 (6) | 8.0 (6) |
| C(23) | 0.8625 (9) | −0.1067 (9) | 0.1841 (6) | 7.7 (6) |
| C(24) | 0.8252 (6) | −0.0136 (8) | 0.1928 (5) | 6.8 (5) |
| C(25) | 0.8741 (6) | 0.0741 (7) | 0.1745 (4) | 5.8 (4) |
| C(30) | 0.9534 (4) | 0.2285 (6) | 0.0485 (3) | 4.5 (3) |
| C(31) | 0.8873 (6) | 0.1699 (7) | 0.0071 (4) | 6.3 (4) |
| C(32) | 0.8406 (7) | 0.206 (1) | −0.0547 (5) | 7.7 (6) |
| C(33) | 0.8580 (7) | 0.306 (1) | −0.0725 (5) | 8.0 (6) |
| C(34) | 0.916 (1) | 0.3654 (9) | −0.0366 (5) | 8.2 (6) |
| C(35) | 0.9674 (7) | 0.3290 (7) | 0.0269 (4) | 6.4 (4) |

*Site occupancy factor = 0.5.

Table 3. *Intramolecular distances* (Å) *and angles* (°) *for* [$MoOCl_3(OPPh_3)_2$]

| | | | |
|---|---|---|---|
| Mo—Cl(1) | 2.373 (2) | P(1)—O(1) | 1.506 (5) |
| Mo—Cl(2) | 2.210 (4) | P(1)—C(10) | 1.795 (7) |
| Mo—O(1) | 2.090 (5) | P(1)—C(20) | 1.803 (8) |
| Mo—O(2) | 1.87 (2) | P(1)—C(30) | 1.772 (7) |
| Cl(1)—Mo—Cl(1′) | 168.5 (1) | O(1)—Mo—O(1′) | 84.3 (3) |
| Cl(1)—Mo—Cl(2) | 88.8 (1) | O(1)—Mo—O(2) | 164.3 (5) |
| Cl(1)—Mo—Cl(2′) | 99.9 (1) | O(1)—Mo—O(2′) | 81.1 (5) |
| Cl(1)—Mo—O(1) | 85.1 (1) | O(1)—P(1)—C(10) | 107.9 (3) |
| Cl(1)—Mo—O(1′) | 86.3 (1) | O(1)—P(1)—C(20) | 112.1 (3) |
| Cl(1)—Mo—O(2) | 88.3 (5) | O(1)—P(1)—C(30) | 112.6 (3) |
| Cl(1)—Mo—O(2′) | 98.0 (5) | C(10)—P(1)—C(20) | 109.4 (3) |
| Cl(2)—Mo—O(1) | 97.1 (2) | C(10)—P(1)—C(30) | 107.3 (3) |
| Cl(2)—Mo—O(1′) | 174.8 (2) | C(20)—P(1)—C(30) | 107.4 (3) |
| Cl(2)—Mo—O(2) | 97.0 (5) | Mo—O(1)—P(1) | 160.0 (3) |

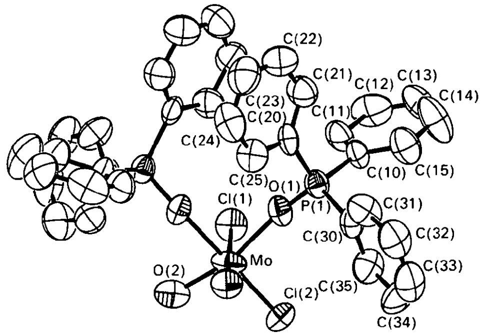

Fig. 1. Molecular configuration and atomic labelling scheme for [$MoOCl_3(OPPh_3)_2$]. Only one configuration for the disorder of the O(2) and Cl(2) atoms is shown for clarity.

For the [$MoOCl_3(OPPh_3)_2$] compound (Fig. 1), the positional parameters and equivalent isotropic thermal parameters are given in Table 2, and selected bond distances and angles are in Table 3.* The structure of this compound has been determined earlier in a different form ($P2_1/c$) with two independent molecules in the asymmetric unit (Garner, Howlader, Mabbs, McPhail & Onan, 1978), of which one is ordered and the other has a similar disorder to that observed for the form reported here, although the atoms are not related by a crystallographic twofold axis. On the other hand, the structure reported here is isomorphous with that of the corresponding W compound, [$WOCl_3(OPPh_3)_2$] (Hill, Howlader, Mabbs, Hursthouse & Malik, 1980). In analogy to this W structure, the data allowed the distinction of the two disordered atoms as separate electron densities, and these could be independently refined to convergence. This is not always the case for O/Cl disorder on Mo complexes (Yoon, Parkin & Rheingold, 1991) and on those of other metals (Yoon & Parkin, 1991). However, the resulting distances are anomalous, indicating that problems remain with the correct modelling of two disordered atoms at such close distances from each other. For instance, the Mo—O(2) distance of 1.87 (2) Å is much longer than the corresponding distance in the other (ordered) form of the same molecule: 1.662 (13) Å (Garner, Howlader, Mabbs, McPhail & Onan, 1978). Correspondingly, the Mo—Cl(2) distance of 2.210 (4) Å is significantly shorter than that of the equivalent bond in the ordered form [2.348 (3) Å]. These two distances should therefore not be given chemical meaning. The distance to the other two mutually *trans* Cl ligands is statistically equivalent to the corresponding distances in the $P2_1/c$ structure, whereas the two Mo—$OPPh_3$ distances (equivalent by symmetry) are halfway between those in the ordered $P2_1/c$ molecule. The rest of the structural parameters are normal and not worthy of dicussion.

For the [$MoOI(dmpe)_2$]I compound (cation shown in Fig. 2), the positional parameters and equivalent isotropic thermal parameters are given in Table 4, and selected bond distances and angles are in Table

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* Lists of structure factors, anisotropic thermal parameters, calculated H-atom positions, and complete intramolecular bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55412 (42 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR0199]

5.* The $I^-$ anion is well separated from the cation. The closest contact is 4.029 Å from C(2), and the distance from Mo is 4.780 Å. The geometry can be described as approximately octahedral, with the Mo atom deviating 0.36 Å from the least-squares equatorial plane determined by the four P donors, toward the O atom. Although the [$MoOI(dmpe)_2$]$^+$ complex has, to the best of our knowledge, not been reported before, analogous $Mo^{IV}$ cationic complexes have been reported, including the very similar [$MoOCl(dppe)_2$]$^+$ cation (dppe = $Ph_2PCH_2CH_2PPh_2$) (Adam, Gregory & Kilbourn, 1970). The Mo—O distance is very close to those reported for the above mentioned [$MoOCl(dppe)_2$]$^+$ cation (1.69 Å) and for other $Mo^{IV}$–oxo species, *e.g.* [$MoOCl(CNMe)_4$]$^+$ [1.64 (4) Å] (Lam, Lewis & Lippard, 1976). The Mo—P distances, on the other hand, are significantly shorter than the reported average distance for [$MoOCl(dppe)_2$]$^+$ (2.57 Å). This effect may be attributed to the decreased steric bulk of the dmpe ligand and/or to its superior $\sigma$-donor properties with respect to the dppe ligand. While iodide complexes of $Mo^V$, *e.g.* [$MoOI_4(thf)$]$^-$ (Cotton & Poli, 1987) and $Mo^{III}$, *e.g.* [$MoI_3(PMe_3)_3$] (Owens, Poli & Rheingold, 1989), are known, with average Mo—I distances of, respectively, 2.746 (16) and 2.73 (3) Å, we are not aware of other $Mo^{IV}$—I linkages being reported in the literature. The Mo—I distance in [$MoOI(dmpe)_2$]$^+$ is extremely long, most likely because of the strong *trans* influence exerted by the oxo ligand.

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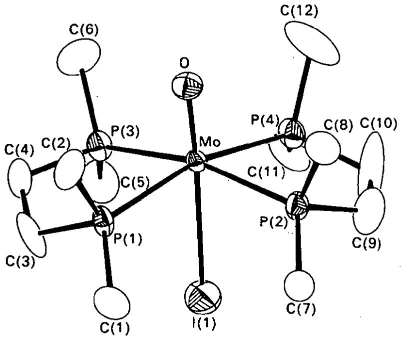

Fig. 2. Molecular configuration and atomic labelling scheme for [$MoOI(dmpe)_2$]$^+$.

Table 4. *Positional parameters and equivalent isotropic thermal parameters* (Å$^2$) *for* [MoIO(*dmpe*)$_2$]I

$B_{eq}$ is defined as one third of the trace of the orthogonalized $B_{ij}$ tensor.

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| I(1) | 0.7855 (1) | 0.26964 (8) | 0.23097 (8) | 5.24 (6) |
| I(2) | 1.27223 (8) | −0.00016 (7) | 0.16936 (7) | 4.36 (5) |
| Mo | 0.86414 (7) | 0.11316 (7) | 0.34286 (6) | 1.93 (4) |
| P(1) | 0.8612 (3) | 0.0603 (2) | 0.1709 (2) | 2.9 (1) |
| P(2) | 1.0586 (3) | 0.1919 (3) | 0.3583 (2) | 3.1 (2) |
| P(3) | 0.6484 (3) | 0.0737 (2) | 0.3069 (2) | 2.9 (1) |
| P(4) | 0.8450 (3) | 0.2053 (3) | 0.4893 (2) | 3.3 (2) |
| O | 0.9067 (8) | 0.0208 (6) | 0.3989 (6) | 3.4 (4) |
| C(1) | 0.929 (1) | 0.115 (1) | 0.076 (1) | 5.3 (9) |
| C(2) | 0.922 (1) | −0.048 (1) | 0.168 (1) | 4.9 (8) |
| C(3) | 0.706 (1) | 0.052 (1) | 0.117 (1) | 5.1 (8) |
| C(4) | 0.627 (1) | 0.019 (1) | 0.186 (1) | 4.6 (8) |
| C(5) | 0.531 (1) | 0.152 (1) | 0.305 (1) | 5.0 (8) |
| C(6) | 0.601 (2) | −0.009 (1) | 0.389 (2) | 6 (1) |
| C(7) | 1.125 (1) | 0.237 (1) | 0.259 (1) | 4.9 (8) |
| C(8) | 1.176 (1) | 0.125 (1) | 0.416 (1) | 5.3 (8) |
| C(9) | 1.051 (2) | 0.281 (1) | 0.440 (1) | 7 (1) |
| C(10) | 0.973 (2) | 0.279 (2) | 0.508 (2) | 15 (2) |
| C(11) | 0.715 (1) | 0.270 (1) | 0.503 (1) | 5.3 (8) |
| C(12) | 0.861 (2) | 0.147 (2) | 0.603 (1) | 9 (1) |

Table 5. *Selected intramolecular distances* (Å) *and angles* (°) *for* [MoIO(*dmpe*)$_2$]

| | | | |
|---|---|---|---|
| Mo—I(1) | 2.964 (2) | Mo—P(3) | 2.510 (3) |
| Mo—P(1) | 2.499 (3) | Mo—P(4) | 2.502 (3) |
| Mo—P(2) | 2.505 (3) | Mo—O | 1.68 (1) |
| I(1)—Mo—P(1) | 79.0 (1) | P(1)—Mo—O | 97.1 (3) |
| I(1)—Mo—P(2) | 81.4 (1) | P(2)—Mo—P(3) | 163.9 (1) |
| I(1)—Mo—P(3) | 82.6 (1) | P(2)—Mo—P(4) | 78.9 (1) |
| I(1)—Mo—P(4) | 84.1 (1) | P(2)—Mo—O | 100.3 (3) |
| I(1)—Mo—O | 176.0 (3) | P(3)—Mo—P(4) | 97.3 (1) |
| P(1)—Mo—P(2) | 99.1 (1) | P(3)—Mo—O | 95.8 (3) |
| P(1)—Mo—P(3) | 80.0 (1) | P(4)—Mo—O | 99.8 (3) |
| P(1)—Mo—P(4) | 163.1 (1) | | |

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* See deposition footnote.