

$wR(F^2) = 0.0733$
 $S = 0.837$
 1676 reflections
 63 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 1.12P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

Extinction correction:
SHELXL93 (Sheldrick, 1993)
 Extinction coefficient:
 0.064 (2)
 Atomic scattering factors
 from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.
 Stoe & Cie (1987a). *DIF4. Data Collection Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.
 Stoe & Cie (1987b). *REDU4. Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

| | <i>x</i> | <i>y</i> | <i>z</i> | U_{eq} |
|-------|------------|------------|------------|-----------------|
| Cr(1) | 0.6215 (1) | 0.5992 (1) | 0.3424 (1) | 0.023 (1) |
| N(1) | 0.6467 (1) | 0.0935 (2) | 0.2932 (2) | 0.031 (1) |
| C(1) | 0.5460 (1) | 0.0954 (2) | 0.3288 (2) | 0.026 (1) |
| O(1) | 1/2 | 0.4925 (2) | 1/4 | 0.030 (1) |
| O(2) | 0.6398 (1) | 0.7336 (2) | 0.2116 (2) | 0.039 (1) |
| O(3) | 0.6179 (1) | 0.7009 (2) | 0.5023 (2) | 0.043 (1) |
| O(4) | 0.7132 (1) | 0.4539 (2) | 0.3900 (1) | 0.034 (1) |

Table 2. Selected geometric parameters (\AA , $^\circ$)

| | | | |
|-----------------|-------------|--------------------------------|-------------|
| Cr(1)—O(3) | 1.617 (2) | N(1)—C(1) | 1.494 (2) |
| Cr(1)—O(2) | 1.625 (2) | C(1)—C(1 ¹) | 1.520 (3) |
| Cr(1)—O(4) | 1.6300 (11) | O(1)—Cr(1 ¹) | 1.7818 (10) |
| Cr(1)—O(1) | 1.7818 (10) | | |
| O(3)—Cr(1)—O(2) | 109.96 (9) | O(2)—Cr(1)—O(1) | 108.37 (7) |
| O(3)—Cr(1)—O(4) | 110.94 (8) | O(4)—Cr(1)—O(1) | 107.59 (6) |
| O(2)—Cr(1)—O(4) | 110.42 (7) | N(1)—C(1)—C(1 ¹) | 110.15 (14) |
| O(3)—Cr(1)—O(1) | 109.49 (6) | Cr(1)—O(1)—Cr(1 ¹) | 124.27 (8) |

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

Data collection and lattice-constant refinement used *DIF4* (Stoe & Cie, 1987a). Lorentz and polarization corrections were applied (*REDU4*; Stoe & Cie, 1987b). The structure was solved by the Patterson method using *SHELXS86* (Sheldrick, 1990). All non-H atoms were refined anisotropically with *SHELXL93* (Sheldrick, 1993). H atoms were refined with restraints: distances N—H and C—H = 1.08 \AA ; isotropic displacement factors = 0.043 and 0.033 \AA^2 for H atoms bonded to N and C, respectively. Molecular graphics were produced using *ORTEPII* (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: SH1107). 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. (1995). **C51**, 1075–1078

A Chelate-Stabilized Molybdenum–Imido–Alkylidene Complex, [TpMo{CHC(Me)₂Ph}(NAr)(OTf)]

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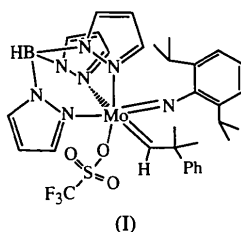
Abstract

The title compound, (2,6-diisopropylphenylimido)[hydrotris(1-pyrazolyl-*N*³)borato](2-methyl-2-phenylpropylidene)(trifluoromethanesulfonato)molybdenum(VI), [Mo(CF₃O₃S)(C₁₀H₁₂)(C₁₂H₁₇N)(C₉H₁₀BN₆)], has been synthesized and its crystal structure determined. The geometry around the Mo atom, imposed by the tridentate tris(pyrazolyl)borate ligand, is that of a distorted octahedron. There appears to be significant interaction between the ligands and the transition metal center. Principal distances include Mo—N(imido) 1.753 (8), Mo—C(propylidene) 1.949 (10), Mo—O(trifluoromethanesulfonato) 2.121 (7) and Mo—N(pyrazolyl) 2.167 (8)–2.311 (9) \AA .

Comment

The chemistry of high-oxidation-state metal–alkylidene complexes is of particular interest because of their role in olefin metathesis reactions and polymerizations (Nugent & Mayer, 1988; Schrock, 1986). Our efforts have focused on employing ancillary chelating ligands to form metal–alkylidene complexes of increased thermal stability (Blosch, Gamble, Abboud & Boncella, 1992; Gamble & Boncella, 1993). As part of our investigation of the use of hydrotris(pyrazolyl)borate (Tp) ligands (Trofimenko, 1971) in the stabilization of metal–alkylidene complexes, we have determined the structure of the title compound

[TpMo{CHC(Me)₂Ph}(NAr)(OTf)] (Ar = 2,6-*i*-Pr₂C₆H₃, Ph = C₆H₅, OTf = OSO₂CF₃), (I). Details of the synthesis and characterization of (I) will appear elsewhere (Vaughan, Abboud & Boncella, 1994).



A displacement ellipsoid drawing (*SHELXTL-Plus*; Sheldrick, 1990) of the title molecule, together with the atom-labeling scheme, is given in Fig. 1. There are several pertinent features relating to the structure of (I) that are analogous to features of other structurally characterized Tp or imido-alkylidene complexes (Schrock, Murdzek, Bazan, Robbins, DiMare & O'Regan, 1990). The coordination geometry around the Mo atom in (I) is that of a distorted octahedron. The geometric constraints of the facially bound Tp ligand force the neophylidene, arylimido and trifluoromethanesulfonate ligands to be mutually *cis*. The N—Mo bond lengths of the chelating pyrazolyl rings [Mo—N1 2.311 (8), Mo—N3 2.167 (8) and Mo—N5 2.311 (9) Å] are consistent with the decreasing *trans* influence of the ligands, *i.e.* imido \approx alkylidene > trifluoromethanesulfonate.

The *cis* orientation of the arylimido and alkylidene ligands allows for maximum $d\pi$ — $p\pi$ bonding between

the Mo atom and the multiply bonded ligands (Nugent & Mayer, 1988). The Mo—N bond length of 1.753 (8) Å and Mo—N—C11 bond angle of 170.9 (7)° are within normal ranges for molybdenum-imido complexes in which the Mo—N bond can be considered to be triply bound, with the lone pair of the N atom donating to an empty $d\pi$ orbital of the d^0 Mo atom.

The Mo—C1 bond length of 1.949 (10) Å lies within the expected range for Mo=C double bonds. The —CMe₂Ph group of the alkylidene ligand is in the *syn* or *s-cis* orientation with respect to the imido ligand. This *syn* orientation of the neophylidene ligand is also present in the solution structure as determined by nuclear Overhauser difference spectra (Vaughan, Abboud & Boncella, 1994). The steric congestion imposed by the *syn* orientation of the neophylidene ligand results in the aryl ring of the imido ligand being situated perpendicular to the plane containing atoms N, Mo, C1 and C2.

The Mo—OTf bond distance of 2.121 (7) Å is long compared with the Mo—OMe bond length of 1.960 (7) Å found in the related complex [TpMo{CHC(Me)₂Ph}(NAr)(OMe)] (Vaughan, Abboud & Boncella, 1995). However, the Mo—OTf bond length is within the range for covalently bound trifluoromethanesulfonate ligands (Lawrence, 1986). The presence of a bound trifluoromethanesulfonate ligand was also supported by the presence of characteristic S=O stretches in the IR spectrum of (I) (Vaughan, Abboud & Boncella, 1994). Though the trifluoromethanesulfonate anion is often thought of as being non-coordinating, the high electrophilicity of the metal center and the ability of the Tp ligand to efficiently polarize metal orbitals into an octahedral array (Curtis & Shiu, 1985) lead to bonding between the metal center and the relatively ionic poor π -donating trifluoromethanesulfonate ligand.

Experimental

The compound was synthesized according to the procedure of Vaughan, Abboud & Boncella (1994). The crystal used for structure determination was crystallized from a minimal portion of hexanes at 313 K, which was slowly cooled to ambient temperature.

Crystal data

[Mo(CF₃O₃S)(C₁₀H₁₂)-
(C₁₂H₁₇N)(C₉H₁₀BN₆)]
M_r = 765.51
Orthorhombic
*P*2₁2₁2₁
a = 10.434 (2) Å
b = 13.699 (2) Å
c = 25.148 (4) Å
V = 3595 (1) Å³
Z = 4
D_x = 1.415 Mg m⁻³

Mo *K*α radiation
 λ = 0.71073 Å
Cell parameters from 32 reflections
 θ = 10–11°
 μ = 0.48 mm⁻¹
T = 293 K
Needle
0.58 × 0.10 × 0.10 mm
Orange–yellow

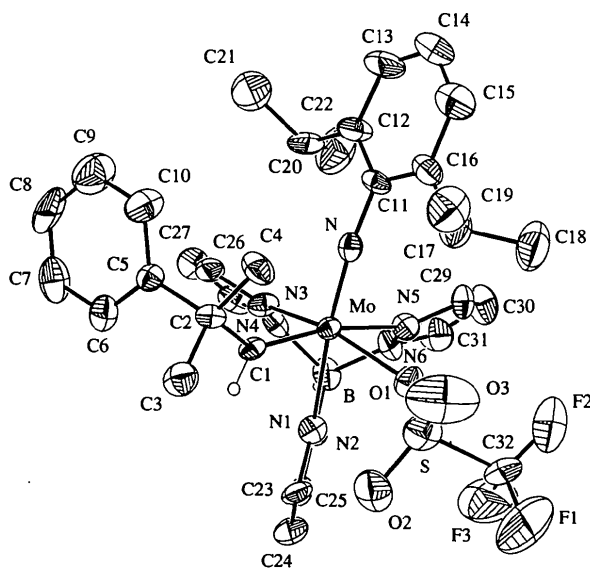


Fig. 1. The molecular structure of (I), with 50% probability ellipsoids, showing the atom-numbering scheme.

Data collection

Siemens P3m/V diffractometer
 ω scans
 Absorption correction: analytical
 $T_{\min} = 0.951$, $T_{\max} = 0.960$
 3603 measured reflections
 3575 independent reflections

2489 observed reflections
 $[F > 6\sigma(F)]$
 $\theta_{\max} = 25^\circ$
 $h = 0 \rightarrow 12$
 $k = 0 \rightarrow 16$
 $l = 0 \rightarrow 29$
 4 standard reflections monitored every 100 reflections
 intensity decay: 4%

Refinement

Refinement on F^2
 $R = 0.0530$
 $wR = 0.0546$
 $S = 1.48$
 2489 reflections
 437 parameters
 $w = 1/[\sigma^2(F) + 0.0004F^2]$

$(\Delta/\sigma)_{\max} = 0.0001$
 $\Delta\rho_{\max} = 0.50 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.54 \text{ e } \text{Å}^{-3}$
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

| | x | y | z | U_{eq} |
|-----|--------------|--------------|-------------|-----------------|
| Mo | 0.02932 (9) | 0.06468 (6) | 0.11559 (3) | 0.0288 (2) |
| S | 0.2775 (3) | 0.1472 (3) | 0.1893 (2) | 0.0614 (13) |
| F1 | 0.354 (2) | 0.2176 (2) | 0.2772 (6) | 0.236 (10) |
| F2 | 0.245 (2) | 0.0858 (10) | 0.2851 (4) | 0.201 (8) |
| F3 | 0.158 (2) | 0.2210 (11) | 0.2684 (5) | 0.187 (9) |
| O1 | 0.1602 (7) | 0.0942 (6) | 0.1781 (3) | 0.043 (3) |
| O2 | 0.2804 (9) | 0.2433 (7) | 0.1674 (4) | 0.075 (4) |
| O3 | 0.3881 (9) | 0.0893 (3) | 0.1845 (6) | 0.131 (6) |
| N | 0.0679 (7) | -0.0592 (6) | 0.1083 (3) | 0.036 (3) |
| N1 | -0.0468 (8) | 0.2193 (6) | 0.1344 (3) | 0.039 (3) |
| N2 | -0.1711 (9) | 0.2343 (7) | 0.1456 (3) | 0.040 (3) |
| N3 | -0.1466 (8) | 0.0698 (8) | 0.0699 (3) | 0.039 (3) |
| N4 | -0.2606 (8) | 0.0980 (6) | 0.0918 (4) | 0.039 (3) |
| N5 | -0.1054 (9) | 0.0331 (6) | 0.1865 (4) | 0.041 (3) |
| N6 | -0.2238 (9) | 0.0731 (8) | 0.1893 (3) | 0.045 (3) |
| B | -0.2661 (14) | 0.1492 (10) | 0.1481 (5) | 0.050 (5) |
| C1 | 0.1267 (10) | 0.1205 (8) | 0.0569 (4) | 0.028 (3) |
| C2 | 0.2187 (10) | 0.0892 (7) | 0.0143 (4) | 0.036 (4) |
| C3 | 0.3115 (12) | 0.1752 (8) | 0.0034 (5) | 0.052 (4) |
| C4 | 0.2995 (11) | 0.0032 (8) | 0.0321 (5) | 0.049 (4) |
| C5 | 0.1382 (9) | 0.0680 (9) | -0.0352 (3) | 0.035 (3) |
| C6 | 0.0661 (11) | 0.1406 (10) | -0.0588 (5) | 0.054 (5) |
| C7 | -0.0121 (12) | 0.1213 (11) | -0.1014 (5) | 0.070 (6) |
| C8 | -0.0198 (15) | 0.0282 (12) | -0.1218 (5) | 0.084 (6) |
| C9 | 0.0498 (15) | -0.0435 (12) | -0.0986 (6) | 0.089 (7) |
| C10 | 0.1277 (12) | -0.0246 (9) | -0.0564 (5) | 0.062 (5) |
| C11 | 0.0880 (10) | -0.1598 (7) | 0.1101 (5) | 0.035 (3) |
| C12 | -0.0007 (13) | -0.2232 (8) | 0.0866 (5) | 0.058 (5) |
| C13 | 0.018 (2) | -0.3224 (8) | 0.0922 (5) | 0.077 (6) |
| C14 | 0.121 (2) | -0.3596 (9) | 0.1181 (6) | 0.082 (6) |
| C15 | 0.210 (2) | -0.2976 (10) | 0.1389 (5) | 0.076 (6) |
| C16 | 0.1967 (12) | -0.1968 (8) | 0.1364 (4) | 0.049 (4) |
| C17 | 0.3005 (13) | -0.1323 (10) | 0.1613 (5) | 0.059 (5) |
| C18 | 0.297 (2) | -0.1426 (12) | 0.2217 (5) | 0.092 (7) |
| C19 | 0.4320 (11) | -0.1515 (10) | 0.1398 (6) | 0.074 (6) |
| C20 | -0.1187 (13) | -0.1839 (9) | 0.0594 (5) | 0.057 (5) |
| C21 | -0.137 (2) | -0.2303 (11) | 0.0048 (6) | 0.098 (7) |
| C22 | -0.237 (2) | -0.1908 (14) | 0.0920 (6) | 0.115 (9) |
| C23 | 0.0115 (12) | 0.3092 (8) | 0.1337 (4) | 0.047 (4) |
| C24 | -0.0788 (13) | 0.3777 (9) | 0.1461 (5) | 0.059 (5) |
| C25 | -0.1918 (13) | 0.3295 (9) | 0.1525 (4) | 0.051 (5) |
| C26 | -0.1773 (12) | 0.0456 (8) | 0.0202 (5) | 0.045 (4) |
| C27 | -0.3051 (13) | 0.0495 (11) | 0.0103 (6) | 0.062 (5) |
| C28 | -0.3564 (11) | 0.0861 (9) | 0.0579 (6) | 0.058 (5) |
| C29 | -0.0977 (14) | -0.0306 (9) | 0.2248 (4) | 0.058 (5) |

| | | | | |
|-----|--------------|--------------|------------|------------|
| C30 | -0.2101 (13) | -0.0339 (11) | 0.2537 (5) | 0.069 (6) |
| C31 | -0.2881 (14) | 0.0372 (10) | 0.2306 (6) | 0.070 (6) |
| C32 | 0.260 (3) | 0.1658 (13) | 0.2602 (8) | 0.134 (13) |

Table 2. Selected geometric parameters (Å , $^\circ$)

| | | | |
|-----------|------------|-----------|------------|
| O1—Mo | 2.121 (7) | N2—N1 | 1.344 (12) |
| N—Mo | 1.753 (8) | C23—N1 | 1.373 (14) |
| N1—Mo | 2.311 (8) | B—N2 | 1.53 (2) |
| N3—Mo | 2.167 (8) | C25—N2 | 1.333 (15) |
| N5—Mo | 2.311 (9) | N4—N3 | 1.366 (12) |
| C1—Mo | 1.949 (10) | C26—N3 | 1.332 (14) |
| O1—S | 1.451 (8) | B—N4 | 1.58 (2) |
| O2—S | 1.427 (10) | C28—N4 | 1.32 (2) |
| O3—S | 1.406 (11) | N6—N5 | 1.354 (13) |
| C32—F1 | 1.28 (3) | C29—N5 | 1.303 (14) |
| C32—F2 | 1.27 (2) | B—N6 | 1.53 (2) |
| C32—F3 | 1.32 (3) | C31—N6 | 1.33 (2) |
| C11—N | 1.396 (12) | C2—C1 | 1.501 (14) |
| O1—Mo—N | 96.5 (3) | N2—N1—Mo | 121.0 (6) |
| O1—Mo—N1 | 84.0 (3) | C23—N1—Mo | 131.9 (7) |
| O1—Mo—N3 | 158.8 (3) | B—N2—C25 | 129.4 (10) |
| O1—Mo—N5 | 81.7 (3) | B—N2—N1 | 121.1 (9) |
| N—Mo—N1 | 170.8 (3) | C25—N2—N1 | 109.4 (9) |
| N—Mo—N3 | 99.8 (4) | N4—N3—C26 | 103.8 (8) |
| N—Mo—N5 | 92.2 (3) | N4—N3—Mo | 122.2 (6) |
| N—Mo—C1 | 100.5 (4) | C26—N3—Mo | 133.9 (7) |
| N1—Mo—N3 | 77.7 (3) | B—N4—C28 | 127.1 (10) |
| N1—Mo—N5 | 78.8 (3) | B—N4—N3 | 121.2 (9) |
| N1—Mo—C1 | 88.5 (4) | C28—N4—N3 | 111.3 (9) |
| N3—Mo—N5 | 84.3 (3) | N6—N5—C29 | 106.8 (10) |
| N3—Mo—C1 | 91.5 (4) | N6—N5—Mo | 121.3 (7) |
| N5—Mo—C1 | 167.2 (4) | C29—N5—Mo | 131.2 (8) |
| C1—Mo—O1 | 98.7 (4) | B—N6—C31 | 129.3 (11) |
| O1—S—O2 | 113.9 (5) | B—N6—N5 | 120.2 (9) |
| O2—S—O3 | 118.0 (7) | C31—N6—N5 | 110.5 (10) |
| O3—S—O1 | 113.1 (6) | N2—B—N4 | 106.1 (9) |
| Mo—O1—S | 141.5 (5) | N2—B—N6 | 111.0 (10) |
| C11—N—Mo | 170.9 (7) | N4—B—N6 | 107.0 (10) |
| N2—N1—C23 | 107.0 (8) | C2—C1—Mo | 139.6 (8) |

The ω -scan width was symmetrical over 1.2° about the $K\alpha_{1,2}$ maximum with the background offset by 1.0 and -1.0° in ω from the $K\alpha_{1,2}$ maximum. The scan speed varied between 3 and 6° min^{-1} depending upon intensity. The structure was solved by the heavy-atom method. The H atoms were refined as riding (C—H 0.96 Å), except for H1 bonded to C1, for which all parameters were refined, and the H atom bonded to the B atom, the coordinates of which were obtained from a difference map.

SHELXTL-Plus (Sheldrick, 1990) was used for cell refinement, data collection, data reduction, structure solution and molecular graphics. *SHELX76* (Sheldrick, 1976) was used for structure refinement (full-matrix least squares).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1016). 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. (1995). **C51**, 1078–1080

trans-Dichlorooxo(2-propoxido)bis-(triphenylphosphine)rhenium(V)

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Abstract

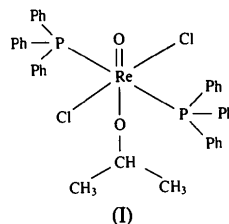
The title compound crystallizes with discrete [ReOCl₂-(C₃H₇O)(C₁₈H₁₅P)₂] units. The coordination sphere of the metal atom is approximately octahedral with the bulky phosphine ligands *trans* with respect to each other. The O=Re—O unit is nearly linear with an angle of 174.25 (9)°. Both Re—Cl bond lengths, as well as both Re—P lengths, are equal within their standard deviations.

Comment

[ReOX₃(Ph₃P)₂] complexes are frequently used as precursors for the synthesis of new rhenium(V)-oxo complexes. The structure of the chloro complex has recently been reported (Lebuis & Beauchamp, 1993). The chloro ligand *trans* to the oxo ligand is only weakly coordinated because of the strong structural *trans* influence of the π-donating oxo ligand and can easily be replaced.

In the course of our studies of the coordination behaviour of tellurium-containing organic ligands, we attempted the reaction of [ReOCl₃(Ph₃P)₂] with bis(2,4,6-

triphenylphenyl)ditelluride. We could not isolate a rhenium species with Re—Te bonds. However, large violet blocks of [ReOCl₂(C₃H₇O)(Ph₃P)₂], (I), were obtained after attempted recrystallization of the brown crude product from CH₂Cl₂/2-propanol solution.



The coordination sphere of the Re atom is approximately octahedral (Fig. 1). Deviations from idealized geometry are found for the O(1)—Re—P angles of 86.7 (1) and 87.5 (1)°. These values are somewhat smaller than those obtained for the related complexes [ReOX₂(OEt)(Ph₃P)₂] (X = Cl, Br) (Graziani, Casellato, Rossi & Marchi, 1985; Lebuis, Roux & Beauchamp, 1993; Lebuis & Beauchamp, 1993), which range between 89.0 (1) and 91.55 (3)°. The differences can be explained by the requirements of the more bulky 2-propoxido ligand *trans* to the oxo ligand. In contrast to the ethoxo ligands in [ReOCl₂(OEt)(Ph₃P)₂] (Graziani, Casellato, Rossi & Marchi, 1985) and [ReOBr₂(OEt)(Ph₃P)₂] (Lebuis, Roux & Beauchamp, 1993; Lebuis & Beauchamp, 1993), no disorder could be detected for the alkoxy group in the title compound.

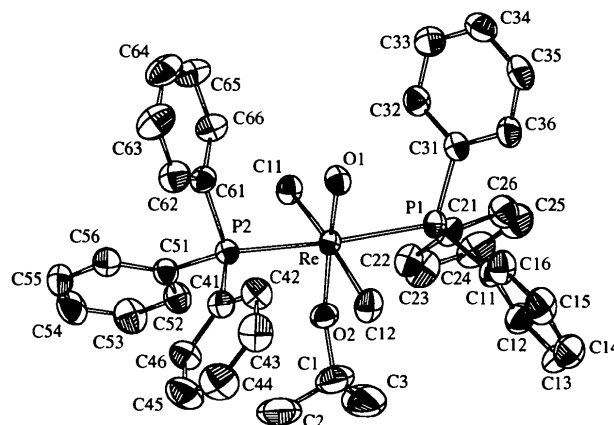


Fig. 1. ORTEP (Johnson, 1976) plot of [ReOCl₂(C₃H₇O)(Ph₃P)₂] along with the atomic numbering scheme (displacement ellipsoids represent 40% probability).

The Re—O(1) bond distance of 1.698 (2) Å comes very close to the mean value for Re—O bonds in monooxo-rhenium(V) complexes (1.69 Å). In agreement with the situation in the comparable complexes [ReO(OR)X₂(Ph₃P)₂] (R = Et, Me; X = Cl, Br, I)