Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (Fair, 1990)
$T_{\min} = 0.716$, $T_{\max} = 0.825$
9841 measured reflections
9841 independent reflections
4834 reflections with $I > 3\sigma(I)$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -28 \rightarrow 28$
3 standard reflections every 300 reflections intensity decay: 2.3%

Refinement

Refinement on F
$R = 0.054$
$wR = 0.060$
$S = 1.16$
4834 reflections
649 parameters
H atoms not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.020$
$\Delta\rho_{\max} = 0.60$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

Mo—Cu1	2.709 (2)	Cu1—P1	2.219 (3)
Mo—Cu2	2.729 (2)	Cu2—S1	2.348 (3)
Mo—Cu3	2.777 (2)	Cu2—S3	2.278 (3)
Mo—S1	2.255 (3)	Cu2—S4	2.457 (3)
Mo—S2	2.257 (3)	Cu2—P2	2.220 (3)
Mo—S3	2.252 (3)	Cu3—S2	2.297 (3)
Mo—O	1.716 (9)	Cu3—S3	2.301 (3)
Cu1—S1	2.343 (4)	Cu3—P3	2.305 (3)
Cu1—S2	2.293 (3)	Cu3—N	2.103 (9)
Cu1—S4	2.459 (3)	S4—C1	1.741 (9)
S1—Mo—S2	108.15 (9)	S2—Cu3—P3	112.7 (2)
S1—Mo—S3	107.2 (1)	S2—Cu3—N	111.5 (2)
S1—Mo—O	110.3 (2)	S3—Cu3—P3	104.2 (1)
S2—Mo—S3	106.3 (1)	S3—Cu3—N	112.3 (2)
S2—Mo—O	112.4 (3)	P3—Cu3—N	112.2 (2)
S3—Mo—O	112.2 (2)	Mo—S1—Cu1	72.16 (8)
S1—Cu1—S2	104.0 (2)	Mo—S1—Cu2	72.68 (7)
S1—Cu1—S4	92.0 (2)	Cu1—S1—Cu2	83.5 (2)
S1—Cu1—P1	123.0 (1)	Mo—S2—Cu1	73.08 (8)
S2—Cu1—S4	112.87 (9)	Cu1—S2—Cu3	96.5 (2)
S2—Cu1—P1	119.2 (2)	Mo—S3—Cu2	74.08 (7)
S4—Cu1—P1	102.5 (2)	Mo—S3—Cu3	75.16 (9)
S1—Cu2—S3	103.3 (2)	Cu2—S3—Cu3	97.1 (2)
S1—Cu2—S4	91.9 (1)	Cu1—S4—Cu2	78.9 (1)
S1—Cu2—P2	126.7 (2)	Cu1—S4—C1	112.5 (3)
S3—Cu2—S4	112.8 (1)	Cu2—S4—C1	113.6 (4)
S3—Cu2—P2	110.4 (1)	Cu3—N—C1	126.5 (6)
S4—Cu2—P2	110.5 (1)	Cu3—N—C5	115.0 (6)
S2—Cu3—S3	103.4 (2)		

The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for non-H atoms. H atoms were placed in calculated positions and not refined. Structure solution and refinement were carried out on a COMPAQ PROLINEA 4/50 computer using the *MolEN* (Fair, 1990) program package. Other programs used included *ORTEP*II (Johnson, 1976) for the molecular graphics.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1304). Services for accessing these data are described at the back of the journal.

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trans-Tetracarbonylbis[tris(4-chlorophenyl)-phosphine-*P*]molybdenum(0)

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Abstract

As a result of its *trans* geometry, the title compound, $[Mo\{(C_6H_4Cl)_3P\}_2(CO)_4]$, has short Mo—P bonds [mean value 2.483 (1) Å] in spite of the presence of bulky chloro-substituted phenyl rings. The Mo atom displays nearly perfect octahedral coordination. Average Mo—C and C—O distances are 2.021 (5) and 1.141 (6) Å, respectively.

Comment

The unit cell of the title compound, (I), contains two independent molecules with their Mo atoms lying on inversion centres (Fig. 1). Each Mo atom displays octahedral coordination. The two chloro-substituted phosphine ligands are *trans* with respect to each other, with an average Mo—P distance of 2.483 (1) Å, which is shorter than comparable values in $[Mo(CO)_5PPh_3]$ [2.560 (1) Å; Cotton, Darensbourg & Ilsley, 1981], $[Mo(CO)_5\{P(4-$

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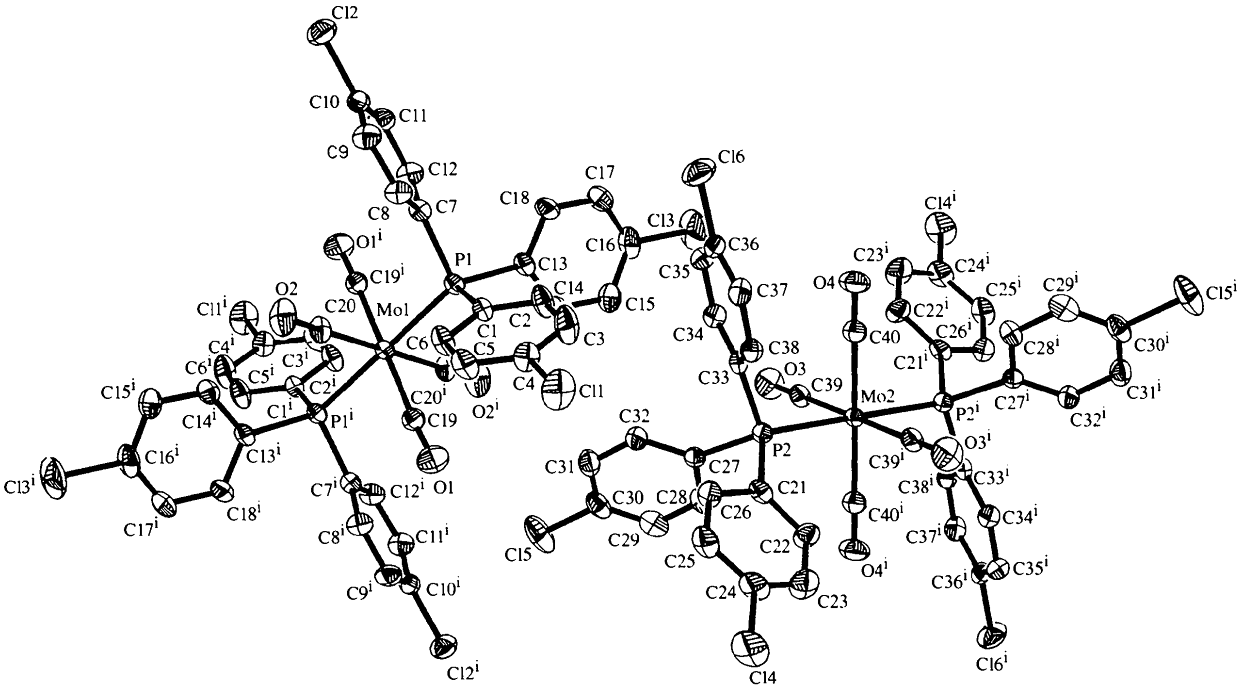
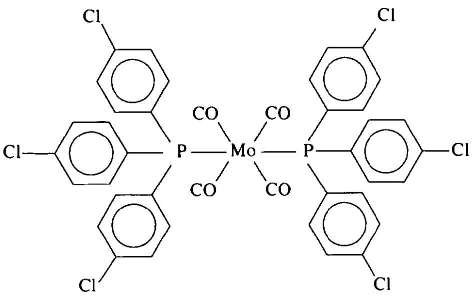


Fig. 1. Displacement ellipsoid plot (50% probability) of compound (I) with the atom-numbering scheme. H atoms are shown as spheres of arbitrary radii. Each Mo atom lies on an inversion centre.

CH$_3$C$_6$H$_4$)$_3$}] [2.558 (1) Å; Alyea, Ferguson & Somogyvari, 1983] and *cis*-[Mo(CO)$_4$(PPh$_3$)$_2$] [2.577 (2) Å; Cotton, Darensbourg & Klein, 1982].



(I)

Comparison of the mean Mo—C and C—O distances of 2.021 (5) and 1.141 (6) Å, respectively, in (I) with those of 2.059 (3) and 1.125 (5) Å, respectively, in Mo(CO)$_6$ (Mak, 1984) indicates stronger bonding due to the back-bonding abilities of the *trans*-tris(chlorophenyl)phosphines. The carbonyl groups are almost at right angles to each other (C—Mo—C 87–93°) and form planar Mo(CO)$_4$ units. One of the chlorophenyl groups is parallel to the Mo(CO)$_4$ plane which alleviates the steric strain and is exemplified by the relatively low C7—P1—Mo1 angle of 111.8 (2)°.

The mean P—C bond length is 1.837 (5) Å which compares well with the values reported for free tris(4-chlorophenyl)phosphine [mean value 1.834 (2) Å; Shawkataly, Singh, Sivakumar & Fun, 1996] and PPh$_3$ [1.831 (2) Å; Dunne & Orpen, 1991; Daly, 1964]. The bond parameters associated with the phenyl rings are normal.

Experimental

Compound (I) was synthesized by refluxing an ethanolic solution of Mo(CO)$_6$ and tris(4-chlorophenyl)phosphine (1:2 ratio) in the presence of NaBH$_4$ as a catalyst for about 1 h under dry nitrogen gas. Suitable single crystals were obtained by slow evaporation of a solution of (I) in a mixture of dichloromethane, *n*-heptane and toluene (2:1:1).

Crystal data

[Mo(C$_{18}$H$_{12}$Cl$_3$P)$_2$(CO)$_4$]
M_r = 939.17
Triclinic
$P\bar{1}$
a = 10.725 (1) Å
b = 11.126 (2) Å
c = 18.368 (3) Å
α = 93.41 (1)°
β = 90.05 (1)°
γ = 112.61 (1)°
V = 2019.1 (5) Å^3
Z = 2
D_x = 1.545 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 40 reflections
θ = 5.38–12.43°
μ = 0.841 mm^{-1}
T = 293 (2) K
Rectangular slab
0.30 × 0.26 × 0.14 mm
Yellow

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction:
empirical ψ scans
(*XSCANS*; Siemens, 1994)
$T_{\min} = 0.691$, $T_{\max} = 0.892$
10 659 measured reflections
9197 independent reflections
4804 reflections with
$I > 2\sigma(I)$

$R_{\rm int} = 0.041$
$\theta_{\max} = 27.51°$
$h = -1 \rightarrow 13$
$k = -14 \rightarrow 13$
$l = -23 \rightarrow 23$
3 standard reflections
every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.121$
$S = 0.810$
9197 reflections
577 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 1.03$ e Å^{-3} (0.93 Å from Mo2)
$\Delta\rho_{\min} = -0.77$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Mo1—C19	2.017 (5)	P2—C33	1.825 (5)
Mo1—C20	2.024 (5)	P2—C27	1.838 (5)
Mo1—P1	2.4900 (12)	P2—C21	1.844 (5)
Mo2—C40	2.017 (5)	Cl1—C4	1.743 (5)
Mo2—C39	2.029 (6)	Cl2—C10	1.732 (5)
Mo2—P2	2.4760 (12)	Cl3—C16	1.746 (5)
P1—C13	1.836 (4)	Cl4—C24	1.739 (6)
P1—C7	1.838 (5)	Cl5—C30	1.750 (5)
P1—C1	1.843 (5)	Cl6—C36	1.733 (5)
C19—Mo1—C20	90.6 (2)	C7—P1—Mo1	111.85 (15)
C19—Mo1—P1	89.45 (14)	C1—P1—Mo1	116.71 (15)
C20—Mo1—P1	87.15 (13)	C33—P2—C27	103.9 (2)
C40—Mo2—C39	89.8 (2)	C33—P2—C21	103.0 (2)
C40—Mo2—P2	88.86 (13)	C27—P2—C21	97.8 (2)
C39—Mo2—P2	87.00 (14)	C33—P2—Mo2	110.96 (14)
C13—P1—C7	102.3 (2)	C27—P2—Mo2	118.6 (2)
C13—P1—C1	100.5 (2)	C21—P2—Mo2	120.3 (2)
C7—P1—C1	103.8 (2)	O3—C39—Mo2	177.5 (5)
C13—P1—Mo1	119.5 (2)	O4—C40—Mo2	179.4 (5)

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All the H atoms were located from difference Fourier maps and refined isotropically. Computer program *PARST* (Nardelli, 1983) was used for geometrical calculations.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*.

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Tetracarbonyl[2-(diphenylphosphino)aniline-*N*,*P*]molybdenum(0)

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Abstract

In the title complex, $[Mo(CO)_4(C_{18}H_{16}NP)]$, coordination about the central metal is distorted octahedral. The five-membered $MoNC_2P$ chelate ring has an 'envelope' conformation with the Mo atom deviating from the plane defined by the four non-metal atoms.

Comment

The title compound, (I), $[Mo(CO)_4\{(C_6H_5)_2PC_6H_4-NH_2\}]$, was prepared as part of a series of our continuing studies on transition metal derivatives containing NH, OH and SH functional PR_3 ligands (Dahlenburg, Herbst & Kühnlein, 1997; Dahlenburg & Kühnlein, 1997; Dahlenburg & Herbst, 1997; Dahlenburg, Herbst & Liehr, 1997). This crystal structure was determined in order to make comparisons with related tetracarbonylmolybdenum complexes bearing bidentate ligands with NH_2 and $P(C_6H_5)_2$ donor groups.