

Fig. 1. Stereopair showing the molecular conformation and the atomic numbering of $C_6H_{14}Cl_2N_2O_5Pt.H_2O$. The ellipsoids correspond to 50% probability, except for the H atoms which are shown as spheres of arbitrary size.

lucci, 1987); see also Okude, Ichida, Miyamoto & Sasaki (1989).

We would like to thank Le Ministère de l'Éducation du Québec, NSERC and the National Cancer Institute (Canada) for financial support.

Acta Cryst. (1991). **C47**, 1069–1072

Structures of Trimethylphosphonium *trans*-Tetrachlorobis(trimethylphosphine)-chromate(III), *mer*-Trichlorobis(dimethylphenylphosphine)(dimethylphenylphosphine oxide)chromium(III) and *mer*-Trichlorotris(trimethylphosphine oxide)-molybdenum(III) Dichloromethane Solvate

BY F. ALBERT COTTON* AND RUDY L. LUCK

Department of Chemistry and Laboratory for Molecular Structure and Bonding, Texas A&M University, College Station, TX 77843, USA

(Received 2 April 1990; accepted 22 October 1990)

Abstract. (I) $[C_3H_{10}P][CrCl_4(C_3H_9P)_2]$, $M_r = 423.05$, monoclinic, $C2/c$, $a = 15.334$ (4), $b = 10.202$ (2), $c = 13.145$ (2) Å, $\beta = 92.49$ (1)°, $V = 2054.4$ (7) Å³, $Z = 4$, $D_x = 1.368$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 12.8$ cm⁻¹, $F(000) = 876$, $T = 292$ K, $R = 0.043$ for 1348 ($F_o^2 > 3\sigma F_o^2$) reflections. (II) $[CrCl_3(C_8H_{11}OP)(C_8H_{11}P)_2]$, $M_r = 588.81$, orthorhombic, $Pnma$, $a = 23.234$ (4), $b = 13.677$ (2), $c = 9.064$ (1) Å, $V = 2880.3$ (7) Å³, $Z = 4$, $D_x = 1.358$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 8.5$ cm⁻¹, $F(000) = 1220$, $T = 292$ K, $R = 0.047$ for 1246 ($F_o^2 > 3\sigma F_o^2$) reflections. (III) $[MoCl_3(C_3H_9OP)_3].CH_2Cl_2$, $M_r = 563.47$, orthorhombic, $P2_12_12_1$, $a = 9.402$ (2), $b = 12.298$ (3), $c = 21.607$ (6) Å, $V = 2498.1$ (7) Å³, $Z = 4$, $D_x = 1.498$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu =$

12.5 cm⁻¹, $F(000) = 1140$, $T = 292$ K, $R = 0.041$ for 2176 ($F_o^2 > 3\sigma F_o^2$) reflections. $\bar{1}$ symmetry is imposed on the anion in (I). All bond angles at Cr are close to 90°. Twofold symmetry is imposed on the cation $[PMe_3H]^+$ which leads to disorder between one methyl group and the H atom bonded on P. Cr—Cl distances are 2.327 (1) and 2.353 (1) Å; Cr—P is 2.468 (1) Å. Mirror symmetry is imposed on (II); the Cr atom, three Cl atoms and the phenylphosphine oxide groups of the dimethylphenylphosphine oxide ligand are in the mirror plane. The Cr—Cl distances range from 2.319 (3) to 2.346 (3) Å and Cr—P is 2.489 (2) Å. Cr—O is 1.962 (6) Å. The angles between the *cis*-nonequivalent atoms range from 85.40 (5)° for Cl(3)—Cr—P(2) to 94.56 (5)° for P(2)—Cr—O(1). Cr—O(1)—P(1) is 154.6 (4)° and O(1)—P(1) is 1.501 (7) Å. For (III), the Mo—Cl distances range from 2.432 (3) to 2.441 (3) Å,

* To whom correspondence should be addressed.

References

- AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1973). *NRC Crystallographic Programs for the IBM 360 System*. Accession Nos. 133–137. *J. Appl. Cryst.* **6**, 309–346.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- HANESSIAN, S. & WANG, J. (1990). In preparation.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- OKUDE, K., ICHIDA, H., MIYAMOTO, T. K. & SASAKI, Y. (1989). *Chem. Lett.* pp. 119–120.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- WITIAK, D. T., ROTELLA, D. P., FILPPI, J. A. & GALLUCCI, J. (1987). *J. Med. Chem.* **30**, 1327–1336.

Mo—O from 2.102 (6) to 2.118 (6) Å. *cis* angles range from 86.9 (2)° for O(2)—Mo—O(3) to 93.8 (2)° for Cl(1)—Mo—O(3). The Mo—O—P angles range from 136.3 (4) to 141.0 (4)° and O—P distances from 1.498 (7) to 1.507 (6) Å.

Experimental. Green crystals of complex (I) were obtained after a reaction mixture of $\text{Cr}_2(\text{acetate})_4$, PMe_3 and Me_3SiCl in THF (Kranzl & Witkowska, 1960) was layered with hexanes. Crystals of complex (II) were obtained from layering a reaction mixture of $\text{MoCr}(\text{acetate})_4$, PMe_2Ph and Me_3SiCl in CH_2Cl_2 (Garner, Senior & King, 1976) with hexanes, and those of complex (III) were obtained by a reaction similar to that for (II) except PMe_3 was employed. The purpose of these reactions was to form the unbridged homonuclear $\text{Cr}_2\text{Cl}_4(\text{PMe}_3)_4$ and heteronuclear $\text{CrMoCl}_4(\text{PR}_3)_4$ ($\text{PR}_3 = \text{PMe}_2\text{Ph}$ and PMe_3) complexes. A similar preparative route had been used to produce $\text{Mo}_2\text{Cl}_4(\text{PR}_3)_4$ complexes (Agaskar, Cotton, Dunbar, Falvello & O'Connor, 1987). We cannot rationalize the formation of (I), (II) and (III) except to say that in the case of (II) and (III) there is solution evidence (^{31}P NMR) for the formation of the expected heteronuclear dimers which suggests that the present products may result from the decomposition of the desired products. The approximate crystal dimensions were 0.20 × 0.10 × 0.30 (I), 0.10 × 0.20 × 0.30 (II), 0.30 × 0.50 × 0.40 mm (III), all crystals mounted on glass fibers with epoxy resin; Rigaku AFC5R for (I), Enraf-Nonius CAD-4 for (II) and (III), cell constants derived from least-squares refinement based on 25 setting-angle reflections [$39 \leq 2\theta \leq 44$ (I), $20 \leq 2\theta \leq 30$ (II), $18 \leq 2\theta \leq 32.50^\circ$ (III)]. Intensity data for (I) were collected with ω - 2θ scans at a constant scan rate of 8° min^{-1} in 2θ but with a variable number of scans (maximum of three), determined from counting statistics with $4 < 2\theta < 50^\circ$ ($h = 0$ to 18, $k = 0$ to 12, $l = -16$ to 16) using graphite-monochromated $\text{Mo K}\alpha$ radiation. For (II) and (III), ω scans, scan width ($0.82 + 0.35\tan\theta$)° (II), ($0.87 + 0.35\tan\theta$)° (III) in the range $2 \leq 2\theta \leq 45$ (II) and $4 \leq 2\theta \leq 50^\circ$ (III) [$h = 0$ to 14, $k = 0$ to 25, $l = 0$ to 9 (II); $h = 0$ to 11, $k = 0$ to 14, $l = 0$ to 25 (III)], using graphite-monochromated $\text{Mo K}\alpha$ radiation. Three standard reflections (116, 243, 514; 802, 543, 510, 2; 266, 533, 413) measured every 100 reflections, 60 and 80 min for (I), (II) and (III), respectively, exhibited small (<0.2%) random variations.

1933, 2184, 2524 data were corrected for Lorentz and polarization effects, and for absorption effects based on ψ scans using the empirical method of North, Phillips & Mathews (1968); $T_{\text{min}} = 0.956$, 0.926 , 0.870 , $T_{\text{max}} = 1.000$, respectively, yielded 1348, 1246, 2176 unique observed data [$F_o^2 > 3\sigma(F_o^2)$]; $R_{\text{merge}}(F_o) = 0.025$ for (I). Scattering factors, includ-

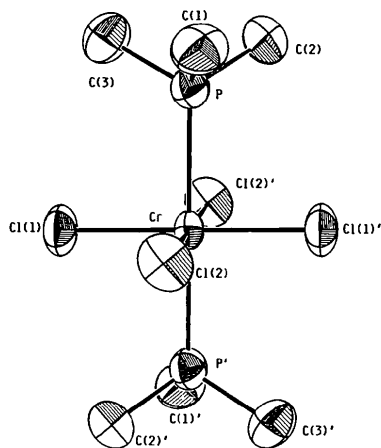


Fig. 1. An ORTEP drawing (Johnson, 1965) of $[\text{CrCl}_4(\text{PMe}_3)_2]^-$, the anion in (I), showing the atomic numbering scheme. Thermal ellipsoids have been drawn at the 50% probability level.

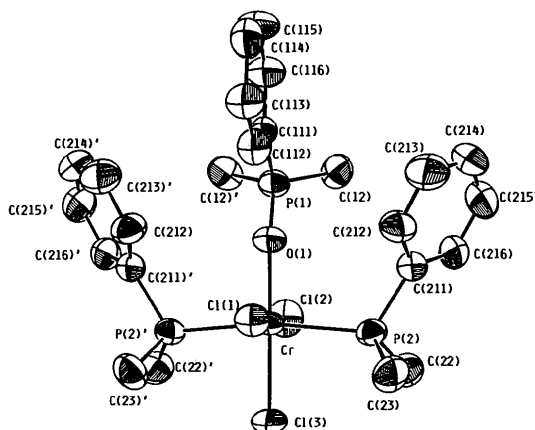


Fig. 2. An ORTEP drawing (Johnson, 1965) of $[\text{CrCl}_3(\text{PMe}_2\text{Ph})_2(\text{OPMe}_2\text{Ph})]$, (II), showing the atomic numbering scheme. Thermal ellipsoids have been drawn at the 50% probability level.

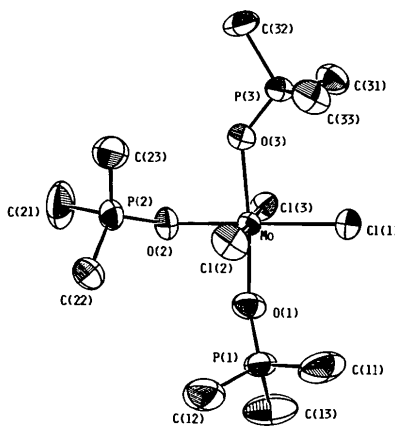


Fig. 3. An ORTEP drawing (Johnson, 1965) of $[\text{MoCl}_3(\text{OPMe}_3)_3]$, (III), showing the atomic numbering scheme. Thermal ellipsoids have been drawn at the 50% probability level.

Table 1. *Positional and equivalent isotropic thermal parameters and their e.s.d.'s for [PMe₃H][CrCl₄(PMe₃)₂], (I)*

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

	x	y	z	$B_{eq}(\text{\AA}^2)$
Cr	0.250	0.250	0.000	2.80 (2)
Cl(1)	0.2670 (1)	0.2245 (1)	0.17567 (9)	5.35 (3)
Cl(2)	0.10653 (8)	0.3247 (2)	0.0196 (1)	5.63 (3)
P	0.31182 (9)	0.4720 (1)	0.02244 (9)	4.08 (2)
C(1)	0.2385 (4)	0.5913 (6)	0.0740 (4)	6.3 (1)
C(2)	0.4076 (4)	0.4808 (6)	0.1066 (4)	6.7 (1)
C(3)	0.3470 (4)	0.5505 (6)	-0.0922 (4)	5.7 (1)
P'	0.000	0.5009 (3)	0.250	8.73 (7)
C/H	-0.0260 (8)	0.640 (1)	0.295 (1)	8.4 (3)
C'	0.0833 (4)	0.4082 (7)	0.3069 (5)	6.5 (1)

Table 2. *Positional and equivalent isotropic thermal parameters and their e.s.d.'s for [CrCl₃(PMe₃Ph)₂(OPMe₂Ph)], (II)*

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

	x	y	z	$B_{eq}(\text{\AA}^2)$
Cr	0.41512 (5)	0.250	0.2585 (2)	2.96 (3)
Cl(1)	0.3340 (1)	0.250	0.4127 (3)	4.19 (6)
Cl(2)	0.4900 (1)	0.250	0.0893 (3)	5.67 (8)
Cl(3)	0.4773 (1)	0.250	0.4602 (3)	5.37 (8)
P(1)	0.3438 (1)	0.250	-0.0665 (3)	3.85 (6)
P(2)	0.41899 (7)	0.0687 (1)	0.2787 (2)	3.68 (4)
O(1)	0.3605 (3)	0.250	0.0935 (7)	4.1 (2)
Cl(2)	0.3694 (4)	0.3542 (5)	-0.1697 (8)	5.4 (2)
C(22)	0.4922 (3)	0.0178 (6)	0.2599 (9)	5.8 (2)
C(23)	0.3949 (4)	0.0246 (6)	0.4589 (8)	5.6 (2)
Cl(111)	0.2671 (4)	0.250	-0.070 (1)	3.4 (2)
Cl(112)	0.2376 (5)	0.250	0.063 (1)	4.6 (3)
Cl(113)	0.1761 (5)	0.250	0.062 (1)	5.6 (3)
Cl(114)	0.1460 (5)	0.250	-0.070 (1)	5.4 (3)
Cl(115)	0.1774 (6)	0.250	-0.205 (1)	6.4 (4)
Cl(116)	0.2375 (5)	0.250	-0.206 (1)	5.6 (3)
C(211)	0.3752 (2)	-0.0016 (4)	0.1529 (5)	3.7 (2)
C(212)	0.3160 (2)	0.0157 (4)	0.1529 (5)	5.1 (2)
C(213)	0.2805 (2)	-0.0336 (4)	0.0533 (5)	7.1 (3)
C(214)	0.3043 (2)	-0.1001 (4)	-0.0462 (5)	6.8 (3)
C(215)	0.3635 (2)	-0.1173 (4)	-0.0462 (5)	6.8 (3)
C(216)	0.3989 (2)	-0.0681 (4)	0.0534 (5)	4.9 (2)

Table 3. *Positional and equivalent isotropic thermal parameters and their e.s.d.'s for [MoCl₃(OPMe₃)₃].CH₂Cl₂, (III)*

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

	x	y	z	$B_{eq}(\text{\AA}^2)$
Mo	0.93272 (8)	0.96408 (5)	0.10415 (3)	2.64 (1)
Cl(1)	1.1680 (3)	0.8902 (2)	0.1249 (1)	4.59 (5)
Cl(2)	1.0390 (3)	1.1332 (2)	0.0673 (1)	4.98 (6)
Cl(3)	0.8201 (3)	0.7969 (2)	0.1390 (1)	4.01 (5)
O(1)	0.9159 (7)	1.0236 (5)	0.1951 (3)	4.7 (1)
O(2)	0.7255 (6)	1.0254 (5)	0.0881 (3)	3.8 (1)
O(3)	0.9274 (7)	0.9053 (5)	0.0126 (2)	3.7 (1)
P(1)	0.9839 (3)	1.1036 (2)	0.2382 (1)	4.15 (5)
P(2)	0.6553 (3)	1.1119 (2)	0.0492 (1)	4.21 (5)
P(3)	1.0289 (2)	0.8476 (2)	-0.0302 (1)	3.26 (4)
Cl(11)	1.173 (1)	1.096 (1)	0.2350 (7)	11.2 (4)
Cl(12)	0.926 (2)	1.2402 (9)	0.2251 (6)	7.6 (3)
Cl(13)	0.930 (2)	1.067 (1)	0.3163 (6)	8.1 (4)
C(21)	0.465 (1)	1.096 (1)	0.0480 (9)	8.6 (4)
C(22)	0.686 (1)	1.2470 (8)	0.0780 (5)	5.5 (3)
C(23)	0.714 (2)	1.1070 (9)	-0.0288 (5)	6.9 (3)
C(31)	1.075 (1)	0.7141 (7)	-0.0028 (5)	4.9 (2)
C(32)	0.950 (1)	0.831 (1)	-0.1045 (5)	5.6 (2)
C(33)	1.186 (1)	0.9237 (9)	-0.0429 (6)	5.5 (2)
Cl(1)	0.445 (2)	0.450 (1)	0.2826 (8)	10.2 (5)
Cl(11)	0.4584 (5)	0.5919 (3)	0.2696 (2)	8.8 (1)
Cl(12)	0.461 (1)	0.3809 (5)	0.2163 (3)	19.4 (3)

Table 4. *Bond distances (\AA) and angles (°) with e.s.d.'s for [PMe₃H][CrCl₄(PMe₃)₂], (I)*

Cr—Cl(1)	2.327 (1)	P—C(2)	1.803 (6)
Cr—Cl(2)	2.353 (1)	P—C(3)	1.810 (6)
Cr—P	2.468 (1)	P'—C/H	1.593 (13)
P—C(1)	1.808 (6)	P'—C'	1.733 (6)
Cl(1)—Cr—Cl(2)	89.57 (5)	C(1)—P—C(2)	103.9 (3)
Cl(1)—Cr—P	87.59 (4)	C(1)—P—C(3)	103.1 (3)
Cl(2)—Cr—P	92.63 (5)	C(2)—P—C(3)	103.0 (3)
Cr—P—C(1)	114.9 (2)	C/H—P'—C'	121.2 (5)
Cr—P—C(2)	114.7 (2)	C'—P'—C'	113.8 (3)
Cr—P—C(3)	115.7 (2)		

Table 5. *Bond distances (\AA) and angles (°) with e.s.d.'s for [CrCl₃(PMe₂Ph)₂(OPMe₂Ph)], (II)*

Cr—Cl(1)	2.346 (3)	Cr—O(1)	1.962 (6)	P(2)—C(22)	1.845 (7)
Cr—Cl(2)	2.319 (3)	P(1)—O(1)	1.501 (7)	P(2)—C(23)	1.829 (7)
Cr—Cl(3)	2.330 (3)	P(1)—C(12)	1.805 (8)	P(2)—C(211)	1.806 (5)
Cr—P(2)	2.489 (2)	P(1)—C(111)	1.784 (9)		
Cl(1)—Cr—Cl(2)	175.2 (1)	Cl(3)—Cr—P(2)	85.40 (5)		
Cl(1)—Cr—Cl(3)	91.7 (1)	Cl(3)—Cr—O(1)	178.0 (2)		
Cl(1)—Cr—P(2)	89.15 (5)	P(2)—Cr—O(1)	94.56 (5)		
Cl(1)—Cr—O(1)	86.2 (2)	O(1)—P(1)—C(12)	114.6 (3)		
Cl(2)—Cr—Cl(3)	93.1 (1)	O(1)—P(1)—C(111)	105.9 (4)		
Cl(2)—Cr—P(2)	91.24 (5)	C(12)—P(1)—C(111)	108.7 (3)		
Cl(2)—Cr—O(1)	88.9 (2)	Cr—P(2)—C(22)	113.7 (3)		
Cr—P(2)—C(23)	112.5 (3)	C(22)—P(2)—C(211)	105.1 (3)		
Cr—P(2)—C(211)	117.6 (2)	C(23)—P(2)—C(211)	102.5 (3)		
C(22)—P(2)—C(23)	103.9 (4)	Cr—O(1)—P(1)	154.6 (4)		

Table 6. *Bond distances (\AA) and angles (°) with e.s.d.'s for [MoCl₃(OPMe₃)₃].CH₂Cl₂, (III)*

Mo—Cl(1)	2.433 (3)	O(2)—P(2)	1.507 (6)	P(2)—C(23)	1.78 (1)
Mo—Cl(2)	2.441 (3)	O(3)—P(3)	1.505 (6)	P(3)—C(31)	1.799 (9)
Mo—Cl(3)	2.432 (2)	P(1)—C(11)	1.78 (1)	P(3)—C(32)	1.78 (1)
Mo—O(1)	2.102 (6)	P(1)—C(12)	1.79 (1)	P(3)—C(33)	1.77 (1)
Mo—O(2)	2.118 (6)	P(1)—C(13)	1.82 (1)	C(1)—Cl(11)	1.77 (2)
Mo—O(3)	2.108 (5)	P(2)—C(21)	1.80 (1)	C(1)—Cl(12)	1.67 (2)
O(1)—P(1)	1.498 (7)	P(2)—C(22)	1.80 (1)		
Cl(1)—Mo—Cl(2)	90.33 (9)	O(1)—Mo—O(3)	174.3 (3)		
Cl(1)—Mo—Cl(3)	91.34 (9)	O(2)—Mo—O(3)	86.9 (2)		
Cl(1)—Mo—O(1)	91.5 (2)	Mo—O(1)—P(1)	141.0 (4)		
Cl(1)—Mo—O(2)	178.4 (2)	Mo—O(2)—P(2)	138.2 (4)		
Cl(1)—Mo—O(3)	93.8 (2)	Mo—O(3)—P(3)	136.3 (4)		
Cl(2)—Mo—Cl(3)	178.20 (9)	O(1)—P(1)—C(11)	111.7 (6)		
Cl(2)—Mo—O(1)	92.2 (2)	O(1)—P(1)—C(12)	113.0 (6)		
Cl(2)—Mo—O(2)	91.1 (2)	O(1)—P(1)—C(13)	107.3 (6)		
Cl(2)—Mo—O(3)	89.8 (2)	C(11)—P(1)—C(12)	110.1 (8)		
Cl(3)—Mo—O(1)	88.4 (2)	C(11)—P(1)—C(13)	107.4 (8)		
Cl(3)—Mo—O(2)	87.2 (2)	C(12)—P(1)—C(13)	107.1 (7)		
Cl(3)—Mo—O(3)	89.5 (2)	O(2)—P(2)—C(21)	111.6 (6)		
O(1)—Mo—O(2)	87.7 (2)	O(2)—P(2)—C(22)	112.9 (5)		
O(2)—P(2)—C(23)	111.7 (5)	O(3)—P(3)—C(33)	112.0 (4)		
C(21)—P(2)—C(22)	105.4 (7)	C(31)—P(3)—C(32)	107.2 (5)		
C(21)—P(2)—C(23)	106.9 (8)	C(31)—P(3)—C(33)	109.5 (5)		
C(22)—P(2)—C(23)	108.0 (5)	C(32)—P(3)—C(33)	105.4 (6)		
O(3)—P(3)—C(31)	112.3 (4)	Cl(11)—C(1)—Cl(12)	111 (1)		
O(3)—P(3)—C(32)	110.1 (5)				

ing anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All computations were carried out using the *SDP/V* package of programs (B. A. Frenz & Associates, Inc., 1985). For (I) and (II) the centrosymmetric space group consistent with the systematic absences $[hkl: h + k \neq 2n; h0l: h, l \neq 2n; 0kl: k \neq 2n; hk0: h + k \neq 2n; 0k0: k \neq 2n; h00: h \neq 2n; 00l: l \neq 2n \text{ (I)}; 0kl: k + l \neq 2n; hk0: h \neq 2n; h00: h \neq 2n; 0k0: k \neq 2n; 00l: l \neq 2n \text{ (II)}; h00: h \neq 2n; 0k0: k \neq 2n; 00l: l \neq 2n \text{ (III)}]$

was tried first and confirmed by successful refinement. The structures were solved by direct methods and refined by full-matrix least-squares methods. All non-H atoms were refined with anisotropic temperature factors. In (I) the H atoms on the anion were found in difference maps and refined freely. In (II), all H atoms except those bonded to the C₆ ring located on the mirror plane were inserted at calculated positions. The thermal parameters of the H atoms bonded to the methyl C atoms were constrained to one value which was refined. The thermal parameters of the H atoms on the phenyl group situated off the mirror plane were constrained to one value which was refined. No attempt was made to refine H atoms in (III). A ΔF map revealed a CH₂Cl₂ molecule which was refined with anisotropic thermal parameters for the C and two Cl atoms. $wR = \{[\sum w(|F_o| - |F_c|)^2] / [\sum w(F_o)^2]\}^{1/2}$ was minimized, where $w = \sigma^2(|F_o|)^{-1}$ and σ is the standard deviation in a single measurement; the final R values were 0.043, 0.047 and 0.041, $wR = 0.068$, 0.065 and 0.062, $S = 1.585$, 1.490 and 1.892 for 132, 162 and 199 variables for (I), (II) and (III), respectively. The largest shift/e.s.d. in the final least-squares cycle was 0.16, 0.028 and 0.08, respectively; the maximum and minimum residual electron densities in the ΔF maps were 0.73 and -0.56 , 0.30 and -0.23 , and 0.60 and -0.41 e \AA^{-3} , respectively.*

* Lists of structure factors, positional parameters [and H-bond distances and angles for (I)] for H atoms, stereoviews of the unit-cell packing, anisotropic thermal parameters, and intramolecular bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53676 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The molecules and the atomic labeling schemes are shown in Figs. 1, 2 and 3 for complexes (I), (II) and (III), respectively. Final positional and equivalent isotropic thermal parameters are listed in Tables 1, 2 and 3 [for complexes (I), (II) and (III), respectively]; important bond distances and angles are listed in Tables 4, 5 and 6.

Related literature. The IR and electronic spectra of $[\text{Ph}_4\text{P}][\text{CrCl}_4(\text{PR}_3)_2]$, the crystal structures of $[\text{N}^n\text{Pr}_4][\text{Cr}(\text{cis-Ph}_2\text{PCHCHPPh}_2)\text{Cl}_4]$ and *mer*- $[\text{Cr}\{\text{P}(\text{CH}_2\text{CH}_2\text{PPh}_2)_3\}\text{Cl}_3]$ have been reported (Bennett, Clark & Goodwin, 1970; Gray, Hale, Levason, McCullough & Webster, 1983, 1984).

These studies were supported by the National Science Foundation.

References

- AGASKAR, P. A., COTTON, F. A., DUNBAR, K. R., FALVELLO, L. R. & O'CONNOR, C. J. (1987). *Inorg. Chem.* **26**, 4051–4057.
 B. A. FRENZ & ASSOCIATES, INC. (1985). *SDP/V. Structure Determination Package*. Version 3.0. College Station, Texas 77840, USA.
 BENNETT, M. A., CLARK, R. J. H. & GOODWIN, A. D. J. (1970). *J. Chem. Soc. A*, pp. 541–544.
 GARNER, C. D., SENIOR, R. G. & KING, T. J. (1976). *J. Am. Chem. Soc.* **98**, 3526–3529.
 GRAY, L. R., HALE, A. L., LEVASON, W., MCCULLOUGH, F. P. & WEBSTER, M. (1983). *J. Chem. Soc. Dalton Trans.* pp. 2573–2580.
 GRAY, L. R., HALE, A. L., LEVASON, W., MCCULLOUGH, F. P. & WEBSTER, M. (1984). *J. Chem. Soc. Dalton Trans.* pp. 47–53.
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
 KRANZL, M. & WITKOWSKA, A. (1960). *Inorg. Synth.* **6**, 144–146.
 NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

Acta Cryst. (1991). **C47**, 1072–1074

Structure of Chloro(dicyclohexylphenylphosphine)gold(I)

BY JAMES A. MUIR, SANDRA I. CUADRADO AND MARIEL M. MUIR

Departments of Chemistry and Physics, University of Puerto Rico, Rio Piedras, PR 00931, USA

(Received 30 July 1990; accepted 25 October 1990)

Abstract. $[\text{AuCl}\{\text{P}(\text{C}_6\text{H}_5)(\text{C}_6\text{H}_{11})_2\}]$, $M_r = 506.8$, orthorhombic, $P2_12_12_1$, $a = 8.476$ (5), $b = 13.747$ (2), $c = 15.951$ (3) Å, $V = 1858.8$ (3) Å³, $Z = 4$, $D_x = 1.81 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 81.24 \text{ cm}^{-1}$, $F(000) = 984$, $T = 296 \text{ K}$, final $R = 0.031$

for 1860 unique observed reflections. The complex $\text{Cy}_2\text{PhPAuCl}$ adopts the two-coordinate linear geometry typical of Au^{I} complexes. The P—Au—Cl angle is 178.3 (1)° while Au—P and Au—Cl distances are 2.234 (2) and 2.281 (3) Å, respectively.