Marumo & Saito, 1970), for which the N—Co—N angles were 85·0, 86·7 and 86·0°. With the exception of these rather small angles and the axial methyl group, there was little evidence of significant steric problems in the complex.

The two perchlorates were somewhat disordered and could only be refined satisfactorily by imposing geometrical constraints. This disorder, commonly encountered for this anion, presumably contributed to the unusually high value of R. Another contributing factor was the use of Cu radiation, which was convenient for comparing this Co complex with another which had a lattice parameter in excess of 53 Å. The high R value in no way limits the value of the structure, since the major question, the chirality of the complex, is clearly established.

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Structure of Chlorotris(triphenylphosphine)copper(I)–Tetrahydrofuran (1/3)

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Abstract. $[CuCl(C_{18}H_{15}P)_3].3C_4H_8O$, $M_r = 1102·2$, hexagonal, $P6_3$, $a = 18·444\ (7)$, $c = 9·817\ (2)$ Å, $V = 2891·4$ Å^3, $Z = 2$, $D_x = 1·27$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 5·49$ cm^{-1}, $F(000) = 1160$, $T = 115$ K, $R = 0·035$ based on 1264 observed $[F > 3\sigma(F)]$ reflections. The pseudo-tetrahedral CuP_3Cl geometry (with tetrahydrofuran in the lattice) shows altered structural parameters from those in a previously studied material lacking lattice solvent.

Introduction. The influence of coordination number on Cu^I—PPh_3 bond length has been described (Gill *et al.*, 1976). Distances can range from 2·01 (1) (Abu Salah, Bruce & Redhouse, 1974) to 2·605 (11) Å (Engelhardt, Pakawatchai, White & Healy, 1985). We report here the results of a structural study of $CuCl(PPh_3)_3.3THF$ (THF = tetrahydrofuran), (1), which shows significantly different structural parameters from the previously studied crystalline material $CuCl(PPh_3)_3$, (2) (Gill *et al.*, 1976).

Experimental. The compound was synthesized by the method of Jardine, Rule & Vohra (1970) and was recrystallized from THF/pentane. A small crystal (0·40 × 0·40 × 0·40 mm) was selected and transferred to the goniostat where it was cooled to 115 K for characterization and data collection. A systematic search of a limited hemisphere of reciprocal space yielded a set of reflections which exhibited $6/m$ symmetry; the only observed extinction was that of $00l$ for $l = 2n+1$. The possible space groups were $P6_3/m$ or $P6_3$. The choice of the non-centrosymmetric space group $P6_3$ was confirmed by the successful solution and refinement of the structure. Data collection ($6 \leq 2\theta \leq 45°$) and processing methods, as well as programs

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Table 1. *Fractional coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Hamilton, 1959) *for non-hydrogen atoms*

	x	y	z	B_{eq}(Å^2 × 10)
Cu(1)	−3333*	−6667*	−4931 (2)	16
Cl(2)	−3333*	−6667*	−2540 (3)	24
P(3)	−2872 (1)	−7593 (1)	−5467*	16
C(4)	−1848 (3)	−7355 (3)	−4775 (7)	20
C(5)	−1125 (4)	−6650 (4)	−5230 (7)	23
C(6)	−346 (4)	−6461 (4)	−4741 (10)	36
C(7)	−274 (4)	−6959 (5)	−3761 (10)	37
C(8)	−991 (5)	−7651 (6)	−3282 (8)	42
C(9)	8218 (4)	2140 (4)	6222 (9)	29
C(10)	−3549 (3)	−8663 (3)	−4829 (7)	19
C(11)	−3705 (4)	−9366 (4)	−5583 (8)	26
C(12)	−4204 (4)	−10158 (4)	−5052 (9)	30
C(13)	−4537 (4)	−10257 (4)	−3753 (10)	33
C(14)	−4393 (4)	−9563 (5)	−3025 (8)	32
C(15)	−3908 (4)	−8770 (4)	−3549 (7)	24
C(16)	−2775 (4)	−7757 (3)	−7292 (6)	18
C(17)	−2135 (4)	−7866 (4)	−7853 (7)	23
C(18)	−2099 (5)	−7949 (4)	−9240 (7)	29
C(19)	−2691 (4)	−7941 (4)	−10108 (7)	26
C(20)	−3336 (4)	−7864 (4)	−9556 (7)	26
C(21)	−3378 (4)	−7771 (4)	−8160 (7)	21
O(22)	7713 (6)	9967 (6)	7276 (26)	170
C(23)	−2042 (14)	−466 (10)	−1707 (18)	116
C(24)	−1546 (13)	−779 (14)	−2414 (42)	187
C(25)	−1558 (19)	−640 (17)	−3980 (43)	196
C(26)	−2019 (14)	−161 (20)	−4056 (20)	173

* Parameters marked by an asterisk are fixed by symmetry.

employed, have been previously described (Huffman, Lewis & Caulton, 1980). 3730 reflections were reduced to a set of 1362 unique intensities. R for the averaging was 0·032 for 1357 reflections observed more than once. The structure was solved by locating the Cu atom by means of direct methods, the remaining non-hydrogen atoms being located by the use of difference Fourier methods. All hydrogen atoms were located in a subsequent difference Fourier map. The full-matrix least-squares refinement was carried out using anisotropic thermal parameters on all non-hydrogen atoms and isotropic thermal parameters on the hydrogen atoms. The maximum Δ/σ for the last refinement cycle was 0·05.

The coordinates listed in this report determine the configuration in a right-handed system. The final $R(F)$ and $wR(F)$ were 0·0355 and 0·0375, respectively. The other enantiomer converged at $R(F) = 0·038$ and $wR(F) = 0·040$. The final difference map was essentially featureless, the largest peak being 0·67 e Å^{-3}. Atoms O(22) through C(26) belong to the THF; the associated thermal parameters are physically unrealistic and indicate some disorder in the molecule. Results are displayed in Tables 1 and 2 and Fig. 1.*

Discussion. The unit cell of (1) contains pseudo-tetrahedral $CuCl(PPh_3)_3$ with crystallographic C_3 symmetry; for each such molecule there are three THF molecules in general positions and not coordinated to Cu. The unit-cell of solvent-free $CuCl(PPh_3)_3$, (2) (Gill *et al.*, 1976), contains three crystallographically independent molecules, each with C_3 symmetry; all three are extremely similar to one another, even to the rotational conformations about the Cu—P and P—C single bonds. In contrast, the molecules in (1) show

Table 2. *Bond distances* (Å) *and angles* (°)

Cu(1)—Cl(2)	2·3469 (27)	C(12)—C(13)	1·387 (12)
Cu(1)—P(3)	2·3183 (17)	C(13)—C(14)	1·369 (11)
P(3)—C(4)	1·841 (6)	C(14)—C(15)	1·376 (9)
P(3)—C(10)	1·839 (6)	C(16)—C(17)	1·406 (9)
P(3)—C(16)	1·840 (6)	C(16)—C(21)	1·391 (9)
C(4)—C(5)	1·390 (8)	C(17)—C(18)	1·375 (10)
C(4)—C(9)	1·399 (10)	C(18)—C(19)	1·392 (10)
C(5)—C(6)	1·385 (9)	C(19)—C(20)	1·376 (10)
C(6)—C(7)	1·381 (11)	C(20)—C(21)	1·389 (9)
C(7)—C(8)	1·383 (11)	O(22)—C(23)	1·484 (21)
C(8)—C(9)	1·397 (10)	O(22)—C(26)	1·46 (3)
C(10)—C(11)	1·393 (9)	C(23)—C(24)	1·48 (3)
C(10)—C(15)	1·388 (10)	C(24)—C(25)	1·56 (4)
C(11)—C(12)	1·381 (10)	C(25)—C(26)	1·51 (4)

Cl(2)—Cu(1)—P(3)	103·13 (5)	C(10)—C(11)—C(12)	120·2 (7)
P(3)—Cu(1)—P(3)′	115·00 (4)	C(11)—C(12)—C(13)	120·2 (7)
Cu(1)—P(3)—C(4)	116·79 (18)	C(12)—C(13)—C(14)	119·3 (6)
Cu(1)—P(3)—C(10)	114·27 (19)	C(13)—C(14)—C(15)	121·2 (7)
Cu(1)—P(3)—C(16)	116·31 (19)	C(10)—C(15)—C(14)	120·0 (7)
C(4)—P(3)—C(10)	101·28 (25)	P(3)—C(16)—C(17)	124·2 (5)
C(4)—P(3)—C(16)	103·09 (28)	P(3)—C(16)—C(21)	117·2 (5)
C(10)—P(3)—C(16)	103·0 (3)	C(17)—C(16)—C(21)	118·6 (6)
P(3)—C(4)—C(5)	119·7 (5)	C(16)—C(17)—C(18)	119·6 (7)
P(3)—C(4)—C(9)	121·4 (4)	C(17)—C(18)—C(19)	121·6 (8)
C(5)—C(4)—C(9)	119·0 (6)	C(18)—C(19)—C(20)	118·9 (7)
C(4)—C(5)—C(6)	121·0 (7)	C(19)—C(20)—C(21)	120·4 (7)
C(5)—C(6)—C(7)	120·4 (7)	C(16)—C(21)—C(20)	120·8 (7)
C(6)—C(7)—C(8)	119·0 (7)	C(23)—O(22)—C(26)	107·3 (17)
C(7)—C(8)—C(9)	121·4 (8)	O(22)—C(23)—C(24)	108·0 (18)
C(4)—C(9)—C(8)	119·2 (6)	C(23)—C(24)—C(25)	109·7 (18)
P(3)—C(10)—C(11)	122·1 (5)	C(24)—C(25)—C(26)	101·7 (16)
P(3)—C(10)—C(15)	118·8 (5)	O(22)—C(26)—C(25)	112·8 (22)
C(11)—C(10)—C(15)	119·1 (6)		

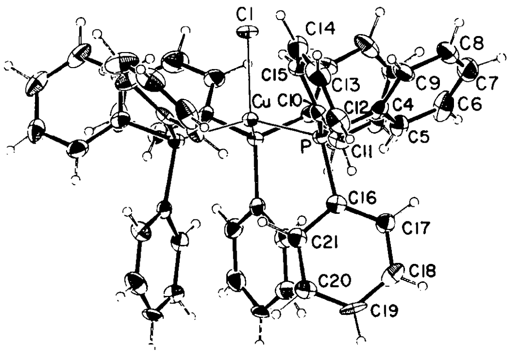

Fig. 1. Molecular structure and atom labeling in $CuCl(PPh_3)_3$. The Cu—Cl line defines the crystallographic C_3 axis which relates unlabeled atoms to those shown.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. 43833 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

clear differences from those in (2), having ∠ClCuP 6° smaller and Cu—P 0·033 Å [8σ(difference)] shorter. These changes may be attributed to conformational differences. The $CuCl(PPh_3)_3$ molecules in (2) all have ClCuP—C torsional angles (±1°) of 32, 155 and 85°, while they are 57·2, 60·7 and 179·4° in (1). Molecules in $CuCl(PPh_3)_3.3THF$ thus have a general Cu—P rotational conformation which puts one P—C vector more precisely *anti* to the Cu—Cl vector than is found in $CuCl(PPh_3)_3$. These differences are displayed in Fig. 2, prepared using program *BMFIT* (Yuen & Nyburg, 1979).

The Cu—Cl distance in $ClCu(PPh_3)_3.3THF$ does not differ significantly from the average value in (2), but the Cu—P distance is shorter by 8σ than the average value [2·351 (4) Å] in (2). Accompanying the shorter Cu—P distance in $ClCu(PPh_3)_3.3THF$ is a larger (115·0°) PCuP′ angle than the corresponding values [109·12 (6)–110·51 (6)°] in (2).

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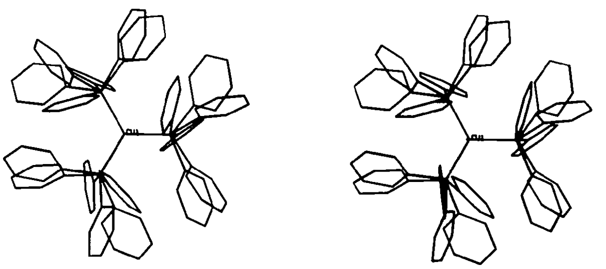

Fig. 2. Stereoview of the best least-squares fit of $CuCl(PPh_3)_3$ found in the unit cell of (1) to the same molecule as found in (2).

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Di-μ_3-oxo-bis(μ-trichloroacetato-*O,O′*)-bis(trichloroacetato)tetrakis[dimethyltin(IV)]

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Abstract. $[Sn_4(C_2Cl_3O_2)_4(CH_3)_8O_2]$, $[\{(CH_3)_2Sn(O_2CCCl_3)\}_2O]_2$, $M_r = 2 \times 638{\cdot}3 = 1276{\cdot}6$, triclinic, $P\bar{1}$, $a = 11{\cdot}778\ (5)$, $b = 10{\cdot}048\ (7)$, $c = 8{\cdot}773\ (5)$ Å, $\alpha = 95{\cdot}31\ (2)$, $\beta = 98{\cdot}07\ (2)$, $\gamma = 103{\cdot}34\ (2)°$, $V = 991{\cdot}7\ (18)$ Å^3, $Z = 1$, $D_x = 2{\cdot}14$ g cm^{-3}, μ(Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å) $= 31{\cdot}0$ cm^{-1}, $F(000) = 604$, room temperature, 1652 X-ray diffractometer measurements refined to give $wR = 0{\cdot}069$. The molecules are dimers with C_{2h} symmetry and the structure is virtually identical to those of both $[\{(C_4H_9)_2Sn(O_2CCCl_3)\}_2O]_2$ and $[\{(CH_3)_2Sn(O_2CCF_3)\}_2O]_2$. Both Sn atoms occupy distorted trigonal bipyramidal environments (the methyl groups in the equatorial plane) with one symmetrically bridging trichloroacetate group [Sn—O = 2·236 (11) and 2·235 (13) Å] and one very asymmetric monodentate trichloracetate group. The coordination is completed by one or two further O neighbours. The Cl atoms are disordered.

Introduction. $[\{(CH_3)_2Sn(O_2CCCl_3)\}_2O]_2$ was prepared from the attempted recrystallization of $(CH_3)_4Sn_2(O_2CCl_3)_2$ from chloroform. As with $[\{(CH_3)_2Sn(O_3CCF_3)\}_2O]_2$ (Faggiani, Johnson, Brown & Birchall, 1978), heating a chloroform solution of $(CH_3)_4Sn_2(O_2CCl_3)_2$ results in tin–tin cleavage and formation of the title compound as fibrous crystalline needles.

Experimental. A cylindrically shaped crystal of radius ~0·1 mm was mounted on a Syntex $P2_1$ diffractometer. Lattice parameters were calculated from the settings of 15 well centred reflections with $7 \le 2\theta \le 34°$ and

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