C40	−0.121 (2)	−0.0411 (9)	0.136 (1)	4.3 (6)
C41	−0.016 (3)	−0.0302 (8)	0.093 (1)	4.3 (6)
C42	0.082 (2)	0.0013 (8)	0.109 (1)	3.6 (6)
C43	0.088 (2)	0.0266 (8)	0.171 (1)	3.9 (6)
C44	−0.018 (2)	0.0189 (8)	0.221 (1)	3.6 (5)
C45	−0.118 (2)	−0.0156 (7)	0.202 (1)	3.1 (5)
C46	−0.494 (2)	0.1011 (8)	0.297 (1)	3.5 (5)
C47	−0.563 (2)	0.0801 (8)	0.236 (1)	3.8 (6)
C48	−0.552 (2)	0.0345 (7)	0.248 (1)	2.6 (5)
C49	−0.449 (2)	−0.0103 (8)	0.365 (1)	3.9 (6)
C50	−0.392 (2)	−0.0054 (8)	0.438 (1)	3.3 (5)
C51	−0.353 (2)	0.0361 (9)	0.464 (1)	4.2 (6)
C52	−0.380 (2)	0.0747 (8)	0.425 (1)	3.3 (5)
C53	−0.447 (2)	0.0716 (8)	0.349 (1)	3.6 (5)
C54	−0.485 (2)	0.0249 (8)	0.320 (1)	3.7 (6)
C55	−0.276 (3)	0.231 (1)	0.694 (2)	5.6 (7)
C56	−0.190 (3)	0.1277 (9)	0.266 (1)	4.6 (6)
C1′	−0.497 (3)	0.1693 (9)	0.610 (2)	5.4 (7)
C1″	−0.624 (3)	0.140 (1)	0.569 (2)	8.0 (9)
C10′	−0.349 (3)	0.246 (1)	0.869 (2)	5.3 (7)
C10″	−0.392 (3)	0.258 (1)	0.946 (2)	8.2 (9)
C19′	0.054 (3)	0.252 (1)	0.664 (2)	5.5 (7)
C19″	0.201 (4)	0.263 (1)	0.645 (2)	11 (1)
C28′	−0.159 (2)	0.1467 (8)	0.087 (1)	3.6 (5)
C28″	−0.014 (3)	0.137 (1)	0.094 (2)	5.8 (7)
C37′	0.054 (3)	0.0620 (9)	0.336 (2)	5.0 (6)
C37″	0.175 (3)	0.040 (1)	0.382 (2)	6.7 (8)
C46′	−0.502 (3)	0.150 (1)	0.305 (2)	6.3 (8)
C46″	−0.636 (4)	0.166 (1)	0.331 (2)	9 (1)

Table 2. *Selected distances* (Å), *angles* (°) *and torsion angles* (°)

Ind. denotes the centroid of the five-membered indenyl ring

Molecule Th1

Th1—C55	2.48 (3)

Ind. I		Ind. II		Ind. III	
Th1—C1	2.94 (3)	Th1—C10	2.83 (2)	Th1—C19	2.84 (3)
Th1—C2	2.73 (2)	Th1—C11	2.78 (2)	Th1—C20	2.76 (3)
Th1—C3	2.70 (2)	Th1—C12	2.77 (2)	Th1—C21	2.69 (2)
Th1—C8	2.97 (2)	Th1—C17	2.96 (3)	Th1—C26	2.98 (3)
Th1—C9	2.92 (2)	Th1—C18	2.92 (3)	Th1—C27	2.95 (2)
Th1—Ind. I	2.59 (3)	Th—Ind. II	2.58 (3)	Th—Ind. III	2.58 (3)

C55—Th1—Ind. I	98.0 (8)	Ind. I—Th1—Ind. II	116.0 (7)
C55—Th1—Ind. II	100.1 (8)	Ind. I—Th1—Ind. III	118.0 (7)
C55—Th1—Ind. III	99.4 (8)	Ind. II—Th1—Ind. III	118.6 (7)

Ind. I	
C8—C1—C1′—C1″	−85 (3)
C2—C1—C1′—C1″	96 (3)
Ind. II	
C17—C10—C10′—C10″	−90 (3)
C11—C10—C10′—C10″	75 (3)
Ind. III	
C26—C19—C19′—C19″	−90 (3)
C20—C19—C19′—C19″	65 (4)

Molecule Th2

Th2—C56	2.50 (3)

Ind. I		Ind. II		Ind. III	
Th2—C28	2.89 (2)	Th2—C37	2.89 (2)	Th2—C46	2.84 (2)
Th2—C29	2.74 (2)	Th2—C38	2.72 (2)	Th2—C47	2.76 (2)
Th2—C30	2.72 (2)	Th2—C39	2.72 (2)	Th2—C48	2.73 (2)
Th2—C35	2.98 (2)	Th2—C44	3.04 (2)	Th2—C53	2.96 (3)
Th2—C36	2.92 (2)	Th2—C45	2.95 (2)	Th2—C54	2.95 (2)
Th2—Ind. I	2.58 (2)	Th2—Ind. II	2.60 (2)	Th2—Ind. III	2.57 (3)

C56—Th2—Ind. I	99.7 (8)	Ind. I—Th2—Ind. II	116.1 (6)
C56—Th2—Ind. II	98.8 (7)	Ind. I—Th2—Ind. III	118.1 (7)
C56—Th2—Ind. III	97.3 (8)	Ind. II—Th2—Ind. III	119.3 (6)

Ind. I	
C35—C28—C28′—C28″	179 (3)
C29—C28—C28′—C28″	−13 (3)
Ind. II	
C44—C37—C37′—C37″	−83 (3)
C38—C37—C37′—C37″	81 (3)
Ind. III	
C53—C46—C46′—C46″	−88 (3)
C47—C46—C46′—C46″	77 (3)

Space group $P2_1/c$ was indicated by the systematic absences. Data were corrected for Lp effects. The structure was solved by direct methods and subsequent $\Delta\rho$ maps. The maximum and minimum excursions of density in the final difference map were close to the Th atom. Refinement was performed by full-matrix least-squares techniques. Only the thermal motions of the Th atoms were refined. H atoms were not included in the refinement. Computer programs used were from the Enraf-Nonius (1986) *SDP*, including *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic thermal parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55908 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1017]

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Structure of $[Cu_2(dmpe)_3Cl_2]_n.2CH_2Cl_2$

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Abstract

Recrystallization of the product obtained from the reaction of copper(I) acetate and 1,2-bis(dimethylphosphino)ethane (dmpe) from methylene chloride and hexane resulted in the formation of the air-stable colorless polymeric compound *catena*-poly[(chlorocopper)-bis-μ-{1,2-

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bis(dimethylphosphino)ethane-*P*:*P'*}-(chlorocopper)-μ-{1,2-bis(dimethylphosphino)ethane-*P*:*P'*}] bis(dichloromethane) solvate, $[Cu_2(dmpe)_3Cl_2]_n.2CH_2Cl_2$. The polymer consists of ten-membered $[Cu_2(dmpe)_2Cl_2]$ rings which are bridged through a unidentate dmpe ligand in such a way as to form a one-dimensional chain which propagates along the *bc* diagonal of the unit cell. The coordination geometry of each copper(I) atom is tetrahedral.

Comment

It has been demonstrated that dppe [1,2-bis(diphenylphosphino)ethane] and other related phosphines exhibit enhanced cytotoxity and antitumor activity when complexed to copper(I) (Berners-Price, Mirabelli, Johnson, Faucette, McCabe & Sadler, 1987; Synder, Mirabelli, Johnson, Sung, Faucette, McCabe, Zimmerman, Whitman, Hemple & Crooke, 1986). Several diphosphine copper(I) complexes have been investigated; most exist as polymeric species containing bridging phosphines and halides (Camus, Marsich, Randaccio & Nardin, 1976; Leoni, Pasquali & Ghilardi, 1983; Gaughan, Ziolo & Dori, 1971; Albano, Belton & Ciani, 1972). Both monomeric (Darensbourg, Chao, Reibenspies & Bischoff, 1990) and dimeric (Mohr, Brooks, Rath & Deutsch, 1991) copper(I) dmpe [1,2-bis(dimethylphosphino)ethane] complexes have recently been reported. Each exists in cationic form as the respective monocation, $[Cu(dmpe)_2]^+$, and dication, $[Cu(dmpe)_2]_2^{2+}$. The title compound exists as a polymer. The polymer consists of $[Cu_2(dmpe)_2Cl_2]$ units. The two Cu atoms in the units are bridged by two dmpe ligands to form a ten-membered $Cu_2P_4C_4$ ring. Tetrahedral coordination around each copper(I) atom is completed by a unidentate dmpe ligand and a chloride. The unidentate dmpe ligand bridges adjacent $[Cu_2(dmpe)_2Cl_2]$ units to form a one-dimensional chain running parallel to the *bc* diagonal of the unit cell.

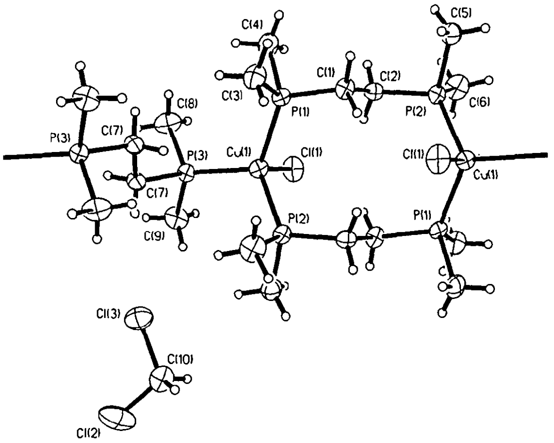

Fig. 1. Thermal ellipsoid plot (50% probability) of $[Cu_2(dmpe)_3Cl_2]_n.2CH_2Cl_2$. H atoms have been drawn as spheres of arbitrary radii.

Experimental

Crystal data

$[Cu_2Cl_2(C_6H_{16}P_2)_3].2CH_2Cl_2$
$M_r = 818$
Triclinic
$P\bar{1}$
$a = 9.025$ (4) Å
$b = 10.093$ (4) Å
$c = 11.987$ (6) Å
$\alpha = 110.60$ (3)°
$\beta = 94.22$ (4)°
$\gamma = 103.47$ (3)°
$V = 979.4$ (7) Å^3
$Z = 1$

$D_x = 1.387$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 15.22-25.15°$
$\mu = 1.76$ mm^{-1}
$T = 197$ K
Plate
0.38 × 0.23 × 0.01 mm
Colorless

Data collection

Siemens *R3m/V* diffractometer
ω scans
Absorption correction: Semi-empirical
$T_{min} = 0.504$, $T_{max} = 0.902$
3625 measured reflections
3625 independent reflections

1688 observed reflections $[F > 8.0\sigma(F)]$
$R_{int} = 0.060$
$\theta_{max} = 25.00°$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 97 min
intensity variation: 2%

Refinement

Refinement on F
Final $R = 0.0614$
$wR = 0.0662$
$S = 1.9137$
1688 reflections
154 parameters
H-atom parameters not refined; for all H atoms U_{iso} fixed at 0.08 Å^2
Calculated weights $w = 1/\sigma^2(F) + 0.001F^2$

$(\Delta/\sigma)_{max} = 0.0018$
$\Delta\rho_{max} = 0.9980$ e Å^{-3}
$\Delta\rho_{min} = -1.1220$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Data collection: *P3VAX* 3.42 (Siemens, 1990). Data reduction: *REFRED* (Reibenspies, 1992*a*). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*; *CIFREP* (Reibenspies, 1992*b*).

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

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu(1)	0.0796 (2)	0.2899 (2)	0.2071 (1)	0.0250 (1)
Cl(1)	0.2766 (4)	0.3407 (3)	0.0914 (3)	0.0384 (1)
Cl(2)	−0.4527 (4)	0.8426 (4)	0.2323 (4)	0.0711 (2)
Cl(3)	−0.2862 (4)	0.6649 (4)	0.3062 (3)	0.0521 (2)
P(1)	0.1245 (3)	0.1042 (3)	0.2565 (3)	0.0235 (1)
P(2)	0.1558 (4)	−0.2352 (3)	−0.0947 (3)	0.0253 (1)
P(3)	0.1453 (3)	0.5139 (3)	0.3577 (3)	0.0231 (1)
C(1)	0.0990 (12)	−0.0719 (11)	0.1327 (9)	0.0267 (5)
C(2)	0.2104 (13)	−0.0679 (12)	0.0424 (9)	0.0277 (5)
C(3)	0.0078 (14)	0.0465 (12)	0.3583 (10)	0.0390 (6)
C(4)	0.3171 (14)	0.1348 (13)	0.3346 (10)	0.0417 (6)
C(5)	0.1844 (15)	−0.3737 (12)	−0.0357 (10)	0.0398 (6)

C(6)	0.3243 (13)	−0.2115 (14)	−0.1704 (11)	0.0428 (6)
C(7)	0.0209 (14)	0.5580 (11)	0.4711 (9)	0.0284 (5)
C(8)	0.3367 (13)	0.5665 (14)	0.4517 (11)	0.0412 (6)
C(9)	0.1623 (14)	0.6694 (11)	0.3088 (11)	0.0366 (5)
C(10)	−0.4058 (14)	0.6757 (13)	0.1859 (10)	0.0427 (6)

Table 2. *Geometric parameters* (Å, °)

Cu(1)—Cl(1)	2.405 (4)	P(2)—C(2)	1.826 (9)
Cu(1)—P(1)	2.264 (4)	P(2)—C(5)	1.832 (15)
Cu(1)—P(3)	2.251 (3)	P(2)—C(6)	1.839 (13)
Cu(1)—P(2^{i})	2.268 (4)	P(2)—Cu(1^{i})	2.268 (4)
Cl(2)—C(10)	1.742 (14)	P(3)—C(7)	1.827 (12)
Cl(3)—C(10)	1.787 (14)	P(3)—C(8)	1.844 (12)
P(1)—C(1)	1.822 (9)	P(3)—C(9)	1.839 (14)
P(1)—C(3)	1.828 (14)	C(1)—C(2)	1.534 (17)
P(1)—C(4)	1.816 (13)	C(7)—C(7^{i})	1.548 (26)
Cl(1)—Cu(1)—P(1)	104.8 (1)	C(5)—P(2)—C(6)	101.5 (7)
Cl(1)—Cu(1)—P(3)	97.4 (1)	C(2)—P(2)—Cu(1^{i})	116.8 (4)
P(1)—Cu(1)—P(3)	116.1 (1)	C(5)—P(2)—Cu(1^{i})	115.3 (4)
Cl(1)—Cu(1)—P(2^{i})	109.0 (1)	C(6)—P(2)—Cu(1^{i})	116.8 (4)
P(1)—Cu(1)—P(2^{i})	114.6 (1)	Cu(1)—P(3)—C(7)	119.9 (3)
P(3)—Cu(1)—P(2^{i})	112.8 (1)	Cu(1)—P(3)—C(8)	115.1 (4)
Cu(1)—P(1)—C(1)	117.0 (4)	C(7)—P(3)—C(8)	102.4 (5)
Cu(1)—P(1)—C(3)	117.6 (5)	Cu(1)—P(3)—C(9)	115.1 (4)
C(1)—P(1)—C(3)	101.0 (5)	C(7)—P(3)—C(9)	100.3 (6)
Cu(1)—P(1)—C(4)	116.9 (4)	C(8)—P(3)—C(9)	101.3 (6)
C(1)—P(1)—C(4)	101.3 (5)	P(1)—C(1)—C(2)	114.4 (7)
C(3)—P(1)—C(4)	100.1 (6)	P(2)—C(2)—C(1)	112.6 (7)
C(2)—P(2)—C(5)	101.8 (5)	P(3)—C(7)—C(7^{i})	113.4 (11)
C(2)—P(2)—C(6)	102.3 (5)	Cl(2)—C(10)—Cl(3)	110.8 (6)

Symmetry code: (i) $-x, -y, -z$.

The title compound was isolated upon recrystallization of a (dmpe)copper(I) acetate species by slow diffusion of hexane into a CH_2Cl_2 solution of the complex at room temperature under a nitrogen atmosphere. The (dmpe)copper(I) acetate species was synthesized from the reaction of copper(I) acetate (2.04 mmol) with 1,2-bis(dimethylphosphino)ethane (2.04 mmol) in 40 ml of benzene under nitrogen atmosphere. The presence of chloride was not observed in the starting materials. The synthesis of the $[Cu_2(dmpe)_3Cl_2]_n$ polymer in the absence of chloride ions in the starting material is proposed to proceed from the reaction of CH_2Cl_2 with the (dmpe)copper(I) acetate species, by an undetermined pathway, during recrystallization from CH_2Cl_2/hexane. A suitable crystal of $[Cu_2(dmpe)_3Cl_2]_n.2CH_2Cl_2$ was chosen and mounted on a glass fiber at room temperature with epoxy cement. Inspection of axial photographs about each of the three axes confirmed axis length and Laue symmetry. ω scans of several intense reflections indicated acceptable crystal quality. The Wyckoff scan mode was employed (Siemens, 1990). The ω scan width was 0.6° with a θ variable scan rate between 2.0 and 29.6° $\min^{-1}$. Background measurements were made by stationary crystal and stationary counter technique at the beginning and end of each reflection for half of the total scan time. C-bound H atoms were placed in idealized positions [C—H = 0.96 Å, U(H) = 0.08 $Å^2$ (fixed)]. The H-atom parameters were not refined.

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Structures of the Monoclinic Form of $Ph_3PAuRuRh_3(CO)_{12}$ (I) and its Cubic Form with $Ru_3(CO)_{12}$ (II)

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Abstract

$C_{30}H_{15}AuO_{12}PRh_3Ru$ crystallizes in the monoclinic form (I), penta-μ-carbonyl-1:2κ^2C;1:3κ^2C;2:3κ^2C;2:4κ^2C;3:4κ^2C-heptacarbonyl-1κ^3C,2κC,3κC,4κ^2C-triphenylphosphine-5κP-goldtrirhodiumruthenium(3*Au*—*Rh*)(3*Rh*—*Rh*)(3*Rh*—*Ru*), and the cubic form (II), tri-μ-carbonyl-2:3κ^2C;2:4κ^2C;3:4κ^2C-nonacarbonyl-1κ^3C,2κ^2C,3κ^2C,4κ^2C-triphenylphosphine-5κP-goldtrirhodiumruthenium(3*Au*—*Rh*)(3*Rh*—*Rh*)-

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55966 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1042]