found to be on an inversion center. Remaining atomic positions were determined from difference Fourier maps. Scattering factors, including terms for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All non-H atoms were refined anisotropically. H atoms were placed in idealized positions with $U_{\rm iso} = 1.2 \times C_{\rm iso}$; the H atom on the solvent O atom was not modeled. Refinement was based on F with weights of the form $w^{-1} = [\sigma^2(F_o) + |g|(F_o^2)]$. The value of g was refined by fitting $(F_o - F_c)^2$ to $[\sigma^2(F_o) + gF^2]/k$ (k = scale factor) to put weights on an approximately absolute scale; final refined $g = 0.00042$. The original data, without an absorption correction, refined to a conventional $R = 0.0367$, $wR = 0.0378$ (GOF = 1.222). An absorption correction was applied to the isotropic model by use of *DIFABS* (Walker & Stuart, 1983). Convergence to conventional R values of $R = 0.0174$ and $wR = 0.0177$ (GOF = 1.001) was obtained using 214 variable parameters and 2303 reflections with $|F_o|^2 > 3\sigma|F_o|^2$. The largest shift/e.s.d. in the last least-squares cycle was 0.005; the maximum and minimum residual electron densities in the final difference Fourier map were 0.66 and -0.57 e Å^{-3}, respectively.

The Pt atom lies on the inversion center. The bond distances for Pt—C and Pt—P of 1.991 (4) and 2.332 (1) Å are in the normal range. The bond angles for P—Pt—P′ and C—Pt—C are required by symmetry to be 180°. The bond angle of Pt—C—N is a normal value of 178.7°. A thermal ellipsoid diagram showing the molecular structure of $[\mathrm{Pt(CN)_2(PPh_3)_2}].2\mathrm{CH_3OH}$ is shown in Fig. 1. Final positional and thermal parameters are presented in Table 1.* Selected interatomic distances, angles and torsion angles are listed in Table 2.

Related literature. The structures of the related compounds *trans*-$[\mathrm{Pt(CNMe)_2\{C(NHCH_3)(SCH_2CH_3)\}_2}]2\mathrm{PF_6}$ (Buttler & Enemark, 1973) and *cis*-$[\mathrm{PtCl_2(CNEt)(PEt_2Ph)}]$, *cis*-$[\mathrm{PtCl_2(CNPh)_2}]$ (Jovanović & Manojlovic-Muir, 1972) have been characterized.

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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. SUP 55319 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0418]

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Structure of Tetrakis(*N*,*N*-dimethylbenzenecarbothioamide-*S*)cadmium(II) Diperchlorate Monohydrate

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Abstract. $[\mathrm{Cd(C_9H_{11}NS)_4}](\mathrm{ClO_4})_2.\mathrm{H_2O}$, $M_r = 990.33$, orthorhombic, $Pbca$, $a = 17.488$ (3), $b = 22.287$ (4), $c = 22.811$ (3) Å, $V = 8890.8$ Å^3, $Z = 8$, $D_x = 1.480$ g cm^{-3}, λ(Mo $K\alpha$) = 0.70930 Å, $\mu = 8.42$ cm^{-1}, $F(000) = 4064$, $T = 293$ K, final $R = 0.044$ for 4255 observed $[I > 3\sigma(I)]$ reflections. In the

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Table 1. *Positional and equivalent isotropic thermal parameters* (Å^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cd	0.74284 (3)	0.18419 (2)	0.21599 (2)	0.0447 (2)
Ligands				
S(1)	0.6203 (1)	0.15379 (9)	0.26741 (8)	0.054 (1)
S(2)	0.8654 (1)	0.19320 (8)	0.27473 (9)	0.058 (1)
S(3)	0.8031 (1)	0.11090 (9)	0.14351 (8)	0.057 (1)
S(4)	0.6911 (1)	0.28376 (9)	0.18156 (9)	0.062 (1)
N(1)	0.5777 (3)	0.0637 (3)	0.3329 (2)	0.049 (3)
N(2)	0.9223 (3)	0.2889 (3)	0.3254 (3)	0.056 (3)
N(3)	0.7812 (3)	0.0820 (3)	0.0340 (2)	0.055 (4)
N(4)	0.7501 (4)	0.3796 (2)	0.1339 (2)	0.055 (3)
C(11)	0.6353 (4)	0.0919 (3)	0.3096 (3)	0.040 (3)
C(12)	0.7139 (4)	0.0707 (3)	0.3217 (3)	0.043 (3)
C(13)	0.7462 (4)	0.0816 (3)	0.3761 (3)	0.052 (4)
C(14)	0.8207 (4)	0.0631 (4)	0.3866 (3)	0.063 (4)
C(15)	0.8617 (4)	0.0337 (3)	0.3446 (4)	0.065 (5)
C(16)	0.8291 (4)	0.0227 (3)	0.2905 (4)	0.063 (4)
C(17)	0.7554 (4)	0.0416 (3)	0.2788 (3)	0.054 (4)
C(18)	0.5858 (5)	0.0092 (3)	0.3689 (4)	0.068 (5)
C(19)	0.4990 (4)	0.0837 (4)	0.3247 (4)	0.069 (5)
C(21)	0.8602 (4)	0.2586 (3)	0.3131 (3)	0.045 (4)
C(22)	0.7863 (4)	0.2810 (3)	0.3349 (3)	0.046 (4)
C(23)	0.7411 (5)	0.2447 (3)	0.3703 (3)	0.063 (4)
C(24)	0.6696 (5)	0.2643 (4)	0.3893 (4)	0.078 (5)
C(25)	0.6438 (5)	0.3188 (4)	0.3727 (4)	0.081 (5)
C(26)	0.6854 (5)	0.3545 (4)	0.3369 (3)	0.076 (5)
C(27)	0.7582 (5)	0.3364 (3)	0.3187 (3)	0.063 (4)
C(28)	0.9262 (5)	0.3382 (4)	0.3667 (4)	0.082 (5)
C(29)	0.9960 (5)	0.2743 (4)	0.2992 (5)	0.090 (6)
C(31)	0.7503 (4)	0.1045 (3)	0.0814 (3)	0.044 (3)
C(32)	0.6688 (4)	0.1236 (3)	0.0804 (3)	0.042 (4)
C(33)	0.6192 (4)	0.1007 (3)	0.1211 (3)	0.053 (4)
C(34)	0.5432 (5)	0.1202 (4)	0.1211 (4)	0.074 (5)
C(35)	0.5197 (5)	0.1623 (4)	0.0814 (4)	0.083 (6)
C(36)	0.5700 (5)	0.1859 (4)	0.0418 (4)	0.084 (6)
C(37)	0.6451 (4)	0.1665 (4)	0.0410 (4)	0.066 (5)
C(38)	0.8629 (5)	0.0689 (4)	0.0308 (4)	0.071 (5)
C(39)	0.7375 (6)	0.0644 (4)	−0.0183 (3)	0.087 (5)
C(41)	0.7546 (4)	0.3218 (3)	0.1392 (3)	0.046 (3)
C(42)	0.8140 (4)	0.2892 (3)	0.1056 (3)	0.048 (4)
C(43)	0.8828 (4)	0.2748 (4)	0.1302 (4)	0.068 (5)
C(44)	0.9366 (4)	0.2431 (4)	0.0956 (5)	0.089 (6)
C(45)	0.9190 (5)	0.2275 (4)	0.0405 (4)	0.095 (6)
C(46)	0.8511 (6)	0.2410 (5)	0.0161 (4)	0.095 (7)
C(47)	0.7980 (5)	0.2731 (4)	0.0492 (4)	0.075 (5)
C(48)	0.8031 (5)	0.4147 (4)	0.0977 (4)	0.075 (5)
C(49)	0.6905 (6)	0.4147 (4)	0.1637 (4)	0.085 (6)
Anions				
Cl(1)	0.5414 (1)	−0.06594 (9)	0.21602 (9)	0.062 (1)
O(11)	0.4836 (4)	−0.0660 (3)	0.2604 (3)	0.107 (5)
O(12)	0.6015 (4)	−0.1017 (4)	0.2330 (3)	0.149 (6)
O(13)	0.5126 (5)	−0.0879 (5)	0.1640 (4)	0.156 (7)
O(14)	0.5650 (6)	−0.0092 (3)	0.2077 (4)	0.165 (7)
Cl(2)	0.5922 (1)	0.11551 (9)	0.51057 (9)	0.062 (1)
O(21)	0.6598 (4)	0.0814 (3)	0.5091 (3)	0.108 (4)
O(22)	0.5987 (5)	0.1655 (3)	0.5453 (4)	0.150 (7)
O(23)	0.5703 (5)	0.1309 (4)	0.4537 (3)	0.156 (6)
O(24)	0.5361 (5)	0.0807 (4)	0.5359 (5)	0.173 (8)
Solvent				
O(1)	0.504 (1)	0.5282 (6)	0.5279 (6)	30.5 (7)*

* This atom was refined isotropically.

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

Cd—S(1)	2.535 (2)	Cd—S(2)	2.536 (2)
Cd—S(3)	2.553 (2)	Cd—S(4)	2.522 (3)
S(1)—C(11)	1.702 (6)	N(1)—C(11)	1.301 (8)
N(1)—C(18)	1.47 (1)	N(1)—C(19)	1.46 (1)
C(11)—C(12)	1.479 (9)	C(12)—C(13)	1.384 (9)
C(12)—C(17)	1.381 (9)	C(13)—C(14)	1.39 (2)
C(14)—C(15)	1.36 (1)	C(15)—C(16)	1.38 (1)
C(16)—C(17)	1.39 (2)		
S(2)—C(21)	1.703 (7)	N(2)—C(21)	1.309 (9)
N(2)—C(28)	1.45 (2)	N(2)—C(29)	1.46 (2)
C(21)—C(22)	1.471 (9)	C(22)—C(23)	1.40 (2)
C(22)—C(27)	1.39 (1)	C(23)—C(24)	1.39 (1)
C(24)—C(25)	1.36 (1)	C(25)—C(26)	1.35 (1)
C(26)—C(27)	1.40 (1)		
S(3)—C(31)	1.699 (7)	N(3)—C(31)	1.308 (8)
N(3)—C(38)	1.46 (1)	N(3)—C(39)	1.48 (2)
C(31)—C(32)	1.49 (1)	C(32)—C(33)	1.371 (9)
C(32)—C(37)	1.38 (2)	C(33)—C(34)	1.40 (2)
C(34)—C(35)	1.37 (1)	C(35)—C(36)	1.37 (1)
C(36)—C(37)	1.38 (1)		
S(4)—C(41)	1.700 (7)	N(4)—C(41)	1.297 (8)
N(4)—C(48)	1.47 (2)	N(4)—C(49)	1.47 (1)
C(41)—C(42)	1.479 (9)	C(42)—C(43)	1.37 (2)
C(42)—C(47)	1.36 (2)	C(43)—C(44)	1.42 (1)
C(44)—C(45)	1.35 (1)	C(45)—C(46)	1.35 (1)
C(46)—C(47)	1.40 (1)		
Cl(1)—O(11)	1.430 (7)	Cl(2)—O(21)	1.406 (7)
Cl(1)—O(12)	1.375 (8)	Cl(2)—O(22)	1.372 (8)
Cl(1)—O(13)	1.379 (8)	Cl(2)—O(23)	1.395 (7)
Cl(1)—O(14)	1.343 (8)	Cl(2)—O(24)	1.378 (9)
S(1)—Cd—S(2)	119.42 (6)	S(1)—Cd—S(3)	118.58 (6)
S(1)—Cd—S(4)	94.35 (6)	S(2)—Cd—S(3)	92.47 (6)
S(2)—Cd—S(4)	113.47 (7)	S(3)—Cd—S(4)	120.66 (7)
C(11)—N(1)—C(18)	123.4 (6)	C(11)—N(1)—C(19)	122.2 (6)
C(18)—N(1)—C(19)	114.4 (6)	S(1)—C(11)—N(1)	120.1 (5)
S(1)—C(11)—C(12)	120.5 (5)	N(1)—C(11)—C(12)	119.4 (6)
C(11)—C(12)—C(13)	119.4 (6)	C(11)—C(12)—C(17)	120.4 (6)
C(13)—C(12)—C(17)	120.2 (6)	C(12)—C(13)—C(14)	119.1 (6)
C(13)—C(14)—C(15)	121.0 (7)	C(14)—C(15)—C(16)	119.7 (7)
C(15)—C(16)—C(17)	120.3 (7)	C(12)—C(17)—C(16)	119.8 (7)
C(21)—N(2)—C(28)	124.7 (6)	C(21)—N(2)—C(29)	122.1 (6)
C(28)—N(2)—C(29)	113.2 (6)	S(2)—C(21)—N(2)	120.4 (5)
S(2)—C(21)—C(22)	120.7 (5)	N(2)—C(21)—C(22)	118.9 (6)
C(21)—C(22)—C(23)	119.9 (7)	C(21)—C(22)—C(27)	121.7 (6)
C(23)—C(22)—C(27)	118.3 (7)	C(22)—C(23)—C(24)	120.6 (8)
C(23)—C(24)—C(25)	119.7 (8)	C(24)—C(25)—C(26)	121.2 (8)
C(25)—C(26)—C(27)	120.1 (8)	C(22)—C(27)—C(26)	120.2 (7)
C(31)—N(3)—C(38)	121.4 (6)	C(31)—N(3)—C(39)	123.9 (6)
C(38)—N(3)—C(39)	114.6 (7)	S(3)—C(31)—N(3)	119.8 (5)
S(3)—C(31)—C(32)	120.7 (5)	N(3)—C(31)—C(32)	119.6 (6)
C(31)—C(32)—C(33)	119.3 (6)	C(31)—C(32)—C(37)	119.9 (6)
C(33)—C(32)—C(37)	120.8 (6)	C(32)—C(33)—C(34)	119.1 (7)
C(33)—C(34)—C(35)	120.0 (7)	C(34)—C(35)—C(36)	120.5 (8)
C(35)—C(36)—C(37)	120.0 (8)	C(32)—C(37)—C(36)	119.6 (7)
C(41)—N(4)—C(48)	123.0 (6)	C(41)—N(4)—C(49)	121.8 (6)
C(48)—N(4)—C(49)	115.3 (6)	S(4)—C(41)—N(4)	120.5 (5)
S(4)—C(41)—C(42)	120.5 (5)	N(4)—C(41)—C(42)	118.9 (6)
C(41)—C(42)—C(43)	121.4 (6)	C(41)—C(42)—C(47)	118.2 (6)
C(43)—C(42)—C(47)	120.3 (8)	C(42)—C(43)—C(44)	118.3 (8)
C(43)—C(44)—C(45)	120.0 (8)	C(44)—C(45)—C(46)	122.1 (9)
C(45)—C(46)—C(47)	118.7 (9)	C(42)—C(47)—C(46)	120.6 (8)
O(11)—Cl(1)—O(12)	109.9 (4)	O(21)—Cl(2)—O(22)	112.5 (5)
O(11)—Cl(1)—O(13)	110.5 (4)	O(21)—Cl(2)—O(23)	110.0 (5)
O(11)—Cl(1)—O(14)	108.6 (6)	O(21)—Cl(2)—O(24)	107.7 (5)
O(12)—Cl(1)—O(13)	108.5 (5)	O(22)—Cl(2)—O(23)	111.2 (5)
O(12)—Cl(1)—O(14)	110.6 (5)	O(22)—Cl(2)—O(24)	106.0 (6)
O(13)—Cl(1)—O(14)	109.0 (6)	O(23)—Cl(2)—O(24)	109.5 (6)

cation the four ligand molecules are coordinated to the Cd atom through the thiocarbonyl S atom in a slightly distorted tetrahedral geometry, with Cd—S distances between 2.522 (3) and 2.553 (2) Å.

Experimental. The title compound was synthesized by reaction of $Cd(ClO_4)_2$ with $C_6H_5C(S)N(CH_3)_2$ in ethanol. Recrystallization from ethanol afforded prismatic colourless single crystals. A specimen (0.10 × 0.10 × 0.20 mm) was mounted on an Enraf–Nonius CAD-4 diffractometer. The unit cell was determined and refined from angular data of 25 randomly selected reflections in the range $14 < \theta <$

19° (CAD-4 routine). Intensities were measured with filtered Mo $K\alpha$ radiation using ω–θ scans for $3 < \theta < 24°$. 7605 reflections were collected in the range h 0 →20, k 0→25, l 0→26. 4255 reflections were unique with $I > 3\sigma(I)$, and were used in structure determination and refinement. Intensities of two standard reflections (10,0,12 and 11,0,8) measured every 60 min showed no intensity variation. Lorentz–polarization corrections were applied, as well as an empirical absorption correction [*DIFABS* (Walker & Stuart, 1983); absorption: minimum 0.712, maximum 1.181]. Structure solution was by direct methods (Sheldrick, 1986), least-squares and difference Fourier techniques, and refinement (on F) by full-matrix least squares with unit weights and anisotropic displacement parameters for non-H atoms. All H atoms were introduced at calculated positions (C—H = 0.95 Å) and used only for structure-factor calculations with $B_{\rm iso}$ fixed at 5.0 Å^2. A secondary-extinction coefficient refined to $g = 8.684 \times 10^{-9}$ $\{F_c = F_c/[1 + g(F_c)^2{\rm Lp}]\}$ (Zachariasen, 1963). $(\Delta/\sigma)_{\rm max} = 0.001$, $(\Delta\rho)_{\rm max} = 0.629$ e Å^{-3}. Final $R = 0.044$ ($wR = 0.046$) for 502 parameters; $S = 4.20$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2A). Calculations were performed on a MicroVAX 3500 computer with the *SDP-VAX* program package (Frenz, 1978). Final positional parameters are in Table 1, and bond lengths and bond angles are in Table 2.* The atomic labelling scheme and molecular structure (Keller, 1988) are shown in Fig. 1, and a stereoview of the unit cell in Fig. 2.

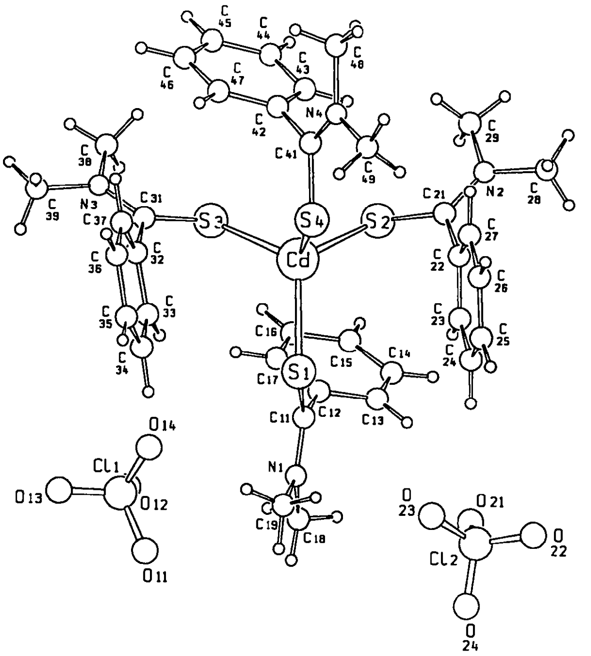

Fig. 1. Molecular structure of $[{\rm Cd}\{{\rm C_6H_5C(S)N(CH_3)_2}\}_4]({\rm ClO_4})_2$.

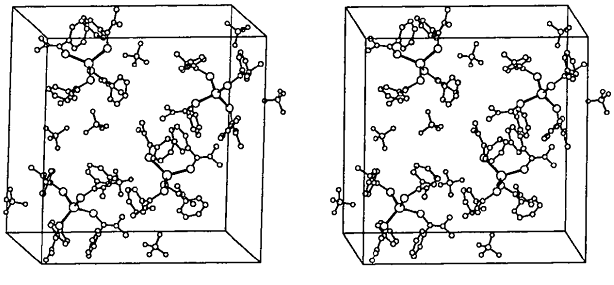

Fig. 2. Stereoview of the unit cell. **c** is vertical, **a** is horizontal and **b** is in plane of the paper.

Related literature. The crystal structure of *N*,*N*-dimethylbenzenecarbothioamide has been reported (Walter, Harto & Voss, 1976). Complexes of benzenecarbothioamide with CuCl (Lenstra, Tavernier & Versichel, 1977) and with the halides of Group IIB metals (Castiñeiras, Arquero & Masaguer, 1984; Castiñeiras, Arquero, Masaguer, Ruíz-Amil, Martinez-Carrera & García-Blanco, 1985; Hiller, Castiñeiras, Arquero & Masaguer, 1986) and of benzenecarbothioamides *N*,*N*-disubstituted with these dihalides (Arquero, Masaguer & Castiñeiras, 1985; Castiñeiras, Arquero, Masaguer, Martinez-Carrera & García-Blanco, 1986) have also been studied.

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* Lists of structure factors, H-atom positions, complete bond distances and angles, torsion angles, least-squares planes and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55388 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BX0581]

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Structure of Tetrakis(triphenylphosphine oxide)lithium Dibromocuprate(I)

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Abstract. $[Li\{OP(C_6H_5)_3\}_4][CuBr_2]$, $M_r = 1343.4$, triclinic, $P\bar{1}$, $a = 14.60$ (1), $b = 16.28$ (1), $c = 17.13$ (1) Å, $\alpha = 70.20$ (5), $\beta = 69.07$ (6), $\gamma = 63.68$ (6)°, $V = 3328$ (5) Å^3, $Z = 2$, $D_x = 1.34$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.66$ mm^{-1}, $F(000) = 1372$, $T = 293$ K, $R = 0.0473$ for 3163 unique observed reflections. The structure consists of a tetrahedrally coordinated $[Li(OPPh_3)_4]^+$ (Li—O ≃ 1.90 Å) cation in a general position and two linear $[CuBr_2]^-$ anions with the Cu atoms on inversion centers.

Experimental. Reaction of methyllithium (1.4 *M* solution in diethyl ether) with damp triphenylphosphine oxide in THF gave a yellow solution, which gave a dark green precipitate upon addition of copper(II) bromide. The mixture was filtered and colorless crystals were obtained by recrystallization from a tetrahydrofuran/diethyl ether solution.

A crystal with dimensions 0.25 × 0.25 × 0.30 mm was mounted on a glass fiber. Intensity data were collected on a Nicolet *R3m/E* diffractometer (graphite-monochromated Mo $K\alpha$ radiation) using the ω-scanning technique in bisecting geometry. Refined cell parameters were obtained from the setting angles of 23 reflections with $5 < 2\theta < 20°$. Intensities were measured for 4617 unique reflections (h 0 to 15, k −14 to 16, l −15 to 17) with $2 < 2\theta < 40°$ (scan rate variable, 3–30° min^{-1}). The intensities of three check reflections ($23\bar{5}$, $\bar{2}1\bar{1}$ and $\bar{1}\bar{2}\bar{5}$) varied less than 2%. Data were corrected for Lorentz and polarization effects. No absorption correction was applied. The structure was solved by direct methods and subsequent electron-density difference maps. Non-H atoms were refined anisotropically. Phenyl rings were refined as hexagons (C—C = 1.395 Å, C—C—C = 120°). H atoms were included in calculated positions (C—H 0.96 Å), assigned isotropic thermal parameters of $U(H) = 1.2U_{eq}(C)$, and allowed to ride on their parent C atoms. Block-cascade-matrix least-squares refinement gave $R = 0.0473$, $wR = 0.0495$ and $S = 1.422$ for 3163 reflections with $F > 2.5\sigma(F)$, and 616 refined parameters. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.00016F^2$. Maximum and minimum residual electron densities were 0.26 and −0.28 e Å^{-3}, respectively. All calculations were performed using *SHELXTL* (Sheldrick, 1985).

Fractional atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,† and selected bond distances and angles in Table 2. Table

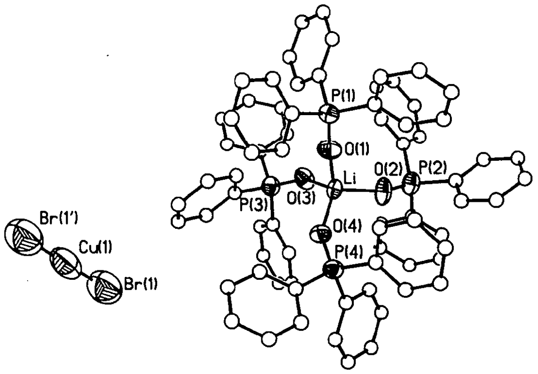

Fig. 1. $[Li(OPPh_3)_4][CuBr_2]$ showing the atom-labeling scheme. Phenyl-group C atoms are of arbitrary radii. Thermal ellipsoids have been drawn at the 50% probability level. H atoms have been omitted for clarity. The $[CuBr_2]$ anion is shown with the Cu atom at the inversion center.

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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. SUP 55427 (45 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0305]

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