

Table 1 lists fractional coordinates and equivalent isotropic thermal parameters. A list of selected bond distances and angles may be found in Table 2.* A perspective view of the ReCl_6^{2-} ion is shown in Fig. 1.

Related literature. The Re—Cl distances found here may be compared with those found in K_2ReCl_6 , Re at $m3m$, Re—Cl = 2.353 (4) Å (Grundy & Brown, 1970); $[\text{NH}_4]_2[\text{ReCl}_6]$, Re at $m3m$, Re—Cl = 2.361 (3) Å (Lisher, Cowlam & Gillott, 1979); $[\text{CH}_3\text{C}_6\text{H}_4\text{NH}_3]_2[\text{ReCl}_6]$, Re—Cl 2.34 (2) to 2.37 (2) Å (Adman & Margulis, 1967); and $[(\text{NH}_2)_2\text{CSSC}(\text{NH}_2)_2]_2\text{Cl}_2[\text{ReCl}_6] \cdot 3\text{H}_2\text{O}$, Re at mm , Re—Cl(1) = 2.358 (2), Re—Cl(2) = 2.363 (2), Re—Cl(3) = 2.362 (2) Å (Lis & Starynowicz, 1985). The PPN cation in the present structure has an internal geometry that is comparable with those observed in other structure determinations, for example $[\text{PPN}][\text{Cr}_2\text{O}_{10}(\mu\text{-D})]$ (Petersen, Brown, Williams & McMullan, 1979), and $[\text{PPN}][\text{UF}_6]$ (Eastman, Eller & Halstead, 1981). In particular, the P—N—P angle of 144.1 (6)° is in the range 135–145°

*Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Center as Supplementary Publication No. SUP 44575 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

observed for all PPN salts except the anomalous compound $[\text{PPN}][\text{V}(\text{CO})_6]$ where a linear P—N—P linkage is found (Wilson & Bau, 1974).

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Structure of Bis(tetraethylammonium) [Chlorotris{chlorocopper(I)}tetrathiorhenate(VII)]

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Abstract. $[\text{N}(\text{C}_2\text{H}_5)_4]_2[\text{ReS}_4(\text{CuCl}_3\text{Cl})]$, $M_r = 907.4$, monoclinic, $C2/c$, $a = 20.988$ (3), $b = 13.007$ (2), $c = 22.894$ (3) Å, $\beta = 91.19$ (2)°, $V = 6248.5$ Å³, $Z = 8$, $D_x = 1.929$ Mg m⁻³, $F(000) = 3552$, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 6.55$ mm⁻¹, $T = 293$ K. $R = 0.047$ for 2896 observed reflections. The Re and Cu atoms, together with three of the S atoms and the triply-bridging Cl, are arranged as a distorted cube with alternating metal and non-metal vertices; each metal atom is further bonded to a terminal atom (Re—S and Cu—Cl). Mean bond lengths are Re—S(terminal) 2.107 (3), Re—S(bridging) 2.215 (5), Cu—S 2.267 (9),

Cu—Cl(terminal) 2.160 (5), Re...Cu 2.618 (6) Å. The bridging of all three Cu atoms by the fourth Cl is distinctly asymmetrical, with long Cu—Cl bonds: 2.855 (3), 2.621 (3) and 2.643 (3) Å. The coordination of Re by four S atoms is close to ideal tetrahedral [S—Re—S angles range from 109.1 (1) to 110.1 (1)°], but the tetrahedral coordination of each Cu by two S and two Cl atoms is very distorted [angles range from 92.4 (1) to 126.0 (1)°].

Experimental. The compound was prepared from $[\text{NEt}_4][\text{ReS}_4]$, CuCl, and $[\text{NEt}_4]\text{Cl}$ in 1:3:1 molar

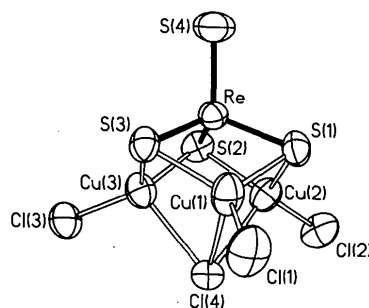
Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

$U_{eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} matrix).

	x	y	z	U_{eq}
Re	6737.8 (2)	5751.2 (3)	5022.6 (2)	51.0 (1)
Cu(1)	7606.0 (6)	5866.6 (10)	5852.2 (6)	70.4 (4)
Cu(2)	6572.8 (6)	4055.9 (8)	5619.9 (5)	64.1 (4)
Cu(3)	6076.1 (6)	6315.0 (9)	5926.9 (6)	66.0 (5)
S(1)	7489 (1)	4579 (2)	5196 (1)	61 (1)
S(2)	5800 (1)	5102 (2)	5249 (1)	63 (1)
S(3)	6936 (1)	7128 (2)	5562 (1)	59 (1)
S(4)	6727 (2)	6174 (2)	4134 (1)	92 (1)
Cl(1)	8465 (1)	6060 (2)	6373 (2)	93 (1)
Cl(2)	6426 (1)	2443 (2)	5798 (1)	83 (1)
Cl(3)	5393 (1)	7207 (2)	6410 (1)	79 (1)
Cl(4)	6693 (1)	5016 (2)	6623 (1)	63 (1)
N(1)	8222 (4)	2458 (6)	6914 (4)	68 (3)
C(11)	8886 (5)	2650 (10)	7165 (5)	87 (5)
C(12)	9094 (7)	3767 (13)	7136 (8)	122 (7)
C(13)	8180 (6)	2787 (10)	6265 (5)	89 (5)
C(14)	8634 (7)	2224 (16)	5864 (6)	129 (7)
C(15)	7715 (7)	3087 (11)	7220 (7)	108 (6)
C(16)	7741 (11)	2881 (16)	7888 (9)	160 (10)
C(17)	8116 (7)	1294 (9)	6975 (6)	99 (5)
C(18)	7449 (8)	951 (10)	6771 (8)	124 (7)
N(2)	5000	3272 (9)	7500	77 (5)
C(21)	5412 (14)	2583 (22)	7141 (17)	255 (19)
C(22)	5646 (29)	1725 (32)	7216 (17)	405 (36)
C(23)	4549 (19)	4016 (53)	7203 (25)	391 (38)
C(24)	4797 (16)	4395 (39)	6566 (31)	407 (37)
N(3)	0	5000	0	76 (5)
C(31x)	166 (10)	3817 (16)	-95 (13)	88 (11)
C(31y)	-419 (15)	4412 (17)	377 (13)	105 (11)
C(32)	-260 (7)	3142 (9)	265 (7)	102 (6)
C(33x)	36 (14)	5213 (19)	703 (9)	94 (11)
C(33y)	735 (10)	4935 (17)	133 (11)	76 (9)
C(34)	859 (10)	5194 (12)	795 (10)	156 (9)

Table 2. Selected bond lengths (\AA) and angles ($^\circ$)

Re—S(1)	2.222 (3)	Re—S(2)	2.213 (3)
Re—S(3)	2.211 (2)	Re—S(4)	2.107 (3)
Cu(1)—S(1)	2.260 (3)	Cu(1)—S(3)	2.252 (3)
Cu(1)—Cl(1)	2.155 (3)	Cu(1)—Cl(4)	2.855 (3)
Cu(2)—S(1)	2.276 (3)	Cu(2)—S(2)	2.269 (3)
Cu(2)—Cl(2)	2.160 (3)	Cu(2)—Cl(4)	2.621 (3)
Cu(3)—S(2)	2.279 (3)	Cu(3)—S(3)	2.266 (3)
Cu(3)—Cl(3)	2.166 (3)	Cu(3)—Cl(4)	2.643 (3)
Re...Cu(1)	2.609 (1)	Re...Cu(2)	2.622 (1)
Re...Cu(3)	2.622 (2)		
S(1)—Re—S(2)	109.1 (1)	S(1)—Re—S(3)	109.4 (1)
S(2)—Re—S(3)	109.6 (1)	S(1)—Re—S(4)	110.1 (1)
S(2)—Re—S(4)	109.5 (1)	S(3)—Re—S(4)	109.1 (1)
S(1)—Cu(1)—S(3)	106.6 (1)	S(1)—Cu(1)—Cl(1)	122.2 (1)
S(3)—Cu(1)—Cl(1)	126.0 (1)	S(1)—Cu(1)—Cl(4)	93.3 (1)
S(3)—Cu(1)—Cl(4)	92.4 (1)	Cl(1)—Cu(1)—Cl(4)	105.6 (1)
S(1)—Cu(2)—S(2)	105.3 (1)	S(1)—Cu(2)—Cl(2)	119.7 (1)
S(2)—Cu(2)—Cl(2)	123.4 (1)	S(1)—Cu(2)—Cl(4)	99.5 (1)
S(2)—Cu(2)—Cl(4)	95.6 (1)	Cl(2)—Cu(2)—Cl(4)	108.0 (1)
S(2)—Cu(3)—S(3)	105.4 (1)	S(2)—Cu(3)—Cl(3)	123.8 (1)
S(3)—Cu(3)—Cl(3)	118.6 (1)	S(2)—Cu(3)—Cl(4)	94.7 (1)
S(3)—Cu(3)—Cl(4)	97.9 (1)	Cl(3)—Cu(3)—Cl(4)	110.8 (1)
Re—S(1)—Cu(1)	71.2 (1)	Re—S(1)—Cu(2)	71.3 (1)
Cu(1)—S(1)—Cu(2)	91.1 (1)	Re—S(2)—Cu(2)	71.6 (1)
Re—S(2)—Cu(3)	71.4 (1)	Cu(2)—S(2)—Cu(3)	89.5 (1)
Re—S(3)—Cu(1)	71.5 (1)	Re—S(3)—Cu(3)	71.7 (1)
Cu(1)—S(3)—Cu(3)	92.8 (1)	Cu(1)—Cl(4)—Cu(2)	72.4 (1)
Cu(1)—Cl(4)—Cu(3)	72.9 (1)	Cu(2)—Cl(4)—Cu(3)	74.9 (1)

Fig. 1. Structure of $[\text{ReS}_4(\text{CuCl})_3\text{Cl}]^{2-}$, thermal ellipsoids drawn at 50% probability level.

proportions in MeCN solution at room temperature. Crystals were obtained after addition of Et₂O to the solution and cooling. D_m not measured. Crystal size for data collection, $0.5 \times 0.3 \times 0.02$ mm, mounted on a glass fibre. Siemens AED2 diffractometer with graphite monochromator. Cell dimensions from 2θ values (20 – 23°) of 32 reflections measured at $\pm\omega$. Data collection in ω/θ scan mode, scan range 0.6° below α_1 to 0.6° above α_2 , scan time 17–68 s, $2\theta_{\text{max}}$ 50° , index ranges h $0 \rightarrow 15$, k $-15 \rightarrow 2$, l $-27 \rightarrow 27$. Correction for small intensity decay of three standard reflections, together with Lp and semi-empirical absorption corrections (transmission 0.29–0.88). 4764 reflections measured, 4218 unique, 2896 with $F > 4\sigma_c(F)$ (σ_c from counting statistics only), $R_{\text{int}} = 0.028$.

Structure solved by Patterson and difference syntheses, blocked-cascade refinement on F , $w^{-1}[\sigma^2(F)] = \sigma_c^2(F) + 284 - 5G + 936G^2 - 724S + 687S^2 - 1214GS$ optimized from a variance analysis, with $G = F_o/F_{\text{max}}$, $S = (\sin\theta)/(\sin\theta_{\text{max}})$ (Hong & Robertson, 1985). Anisotropic thermal parameters, H atoms not included, twofold disorder for one cation on a centre of symmetry, no extinction correction. Final $R = 0.047$, $wR = 0.031$, $S = 1.09$ for 291 parameters, mean $(\Delta/\sigma) = 0.04$, max. = 0.28, max. $(\Delta\rho) = 0.76$, min. = -0.55 e \AA^{-3} . Scattering factors from *International Tables for X-ray Crystallography* (1974). *SHELXTL* (Sheldrick, 1985) and local computer programs.

Atomic coordinates are given in Table 1, bond lengths and angles for the anion in Table 2. Fig. 1 shows the structure of the cluster anion.*

Related literature. The anion geometry is as previously observed in the tetra-*n*-propylammonium salt, in which gross disorder of the cations adversely affected the precision attained (Scattergood, Garner & Clegg, 1987), and resembles that of $[\text{MoS}_4(\text{CuPPh}_3)_3\text{Cl}]$ (Müller, Bögge & Schimanski, 1980), where an asymmetrical μ_3 -Cl bridge is also observed. The only other reported Re—S—Cu—Cl cluster structure is that of $[\text{ReS}_4\text{Cu}_5\text{Cl}_7]^{3-}$ (Müller, Krickemeyer & Bögge, 1986), in which two cubane cores are fused together.

* Lists of structure factors, anisotropic thermal parameters, and geometry of the cations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44663 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Diphenyllead 2,6-Pyridinedicarboxylate Hydrate

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Abstract. $[\text{Pb}(\text{C}_6\text{H}_5)_2][\text{C}_7\text{H}_3\text{NO}_4] \cdot \text{H}_2\text{O}$, $M_r = 544.5$, monoclinic, $P2_1/c$, $a = 9.481(4)$, $b = 10.156(18)$, $c = 19.309(8)$ Å, $\beta = 98.52(4)^\circ$, $V = 1838.7$ Å³, $Z = 4$, $D_x = 1.967$ Mg m⁻³, $F(000) = 1032$, $\lambda(\text{Ag } K\alpha) = 0.5608$ Å, $\mu = 5.0$ mm⁻¹, $T = 291(1)$ K, final $R = 0.024$ for 2472 unique observed [$I \geq 1.96\sigma(I)$] diffractometer data. The coordination polyhedron around Pb is a pentagonal bipyramid, the phenyl groups being in the apical positions. One of the carboxylate groups of the tridentate dianionic ligand is bridging, thereby forming an infinite chain. The chains are probably linked by pairs of H bonds between each water molecule coordinated in the equatorial plane and each non-bridging carboxylate group of two units.

Experimental. The title compound is obtained from diphenyllead diacetate and 2,6-pyridinedicarboxylic acid by refluxing in methanol. Colourless crystals from methanol, dimensions $0.16 \times 0.40 \times 0.04$ mm, $\omega/2\theta$ scans, scan speed $2.0\text{--}6.67^\circ \text{ min}^{-1}$ in θ , Nonius CAD-4 diffractometer, graphite-monochromated Ag $K\alpha$; lattice parameters from least-squares fit of 25 reflections up to $2\theta = 24.6^\circ$; six standard reflections recorded every 2.5 h, only random deviations; 7559 reflections measured, $1 \leq \theta \leq 20^\circ$, $-11 \leq h \leq 11$, $0 \leq k \leq 12$, $-23 \leq l \leq 23$; after averaging ($R_{\text{int}} = 0.026$) 3713 unique reflections, 2472 with $I \geq 1.96\sigma(I)$; Lorentz–polarization correction; absorption correction via ψ scans; max./min. transmission 1.00/0.67; systematic absences $h0l$ $l = 2n + 1$, $0k0$ $k = 2n + 1$ conform to space group $P2_1/c$; structure solution via direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic tempera-

ture factor for H atoms, which were placed in geometrically calculated positions (C–H 0.95 Å) for the phenyl and pyridine rings; refinement on F with 2472 reflections and 236 refined parameters; $w = 4F_o^2/[\sigma^2(F_o^2) + (0.065F_o^2)^2]$; $S = 0.72$, $R = 0.024$, $wR = 0.033$, $(\Delta/\sigma)_{\text{max}} = 0.01$, no extinction correction; largest peak in final ΔF map $\pm 0.8(3) \text{ e } \text{Å}^{-3}$, atomic scattering factors and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *Structure Determination Package* (Frenz, 1981), *SHELXTL PLUS* (Sheldrick, 1987), *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980).

The molecule and the numbering scheme are shown in Fig. 1. Fig. 2 shows the infinite chains which are

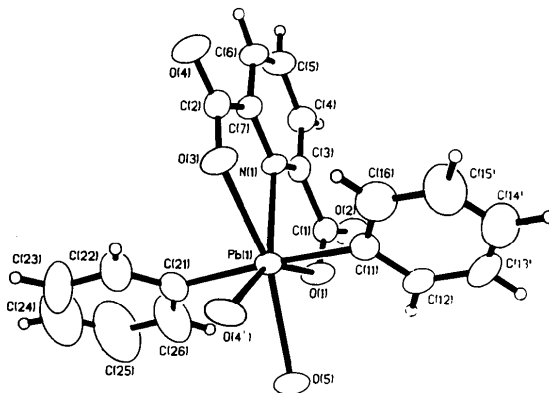


Fig. 1. General view of one formula unit plus the bridging O(4), showing the atom-numbering scheme [i] corresponds to the symmetry operation $-x, \frac{1}{2} + y, \frac{1}{2} - z$]. H atoms at O(5) are omitted.