

Table 2. Bond lengths (Å) and angles (°)

Fe—P	2.338 (4)	Si2—C21	1.88 (2)
Fe—C1	1.77 (2)	Si2—C22	1.87 (2)
Fe—C2	1.74 (2)	Si2—C23	1.92 (2)
Fe—C3	1.84 (2)	Si3—C31	1.89 (2)
Fe—C4	1.77 (2)	Si3—C32	1.93 (2)
P—Si1	2.279 (6)	Si3—C33	1.89 (2)
P—Si2	2.289 (6)	O1—C1	1.14 (2)
P—Si3	2.294 (6)	O2—C2	1.15 (2)
Si1—C11	1.91 (2)	O3—C3	1.15 (2)
Si1—C12	1.89 (2)	O4—C4	1.123 (12)
Si1—C13	1.86 (2)		
P—Fe—C1	91.7 (5)	C11—Si1—C12	107.8 (9)
P—Fe—C2	174.9 (6)	C11—Si1—C13	110. (1)
P—Fe—C3	89.2 (5)	C12—Si1—C13	110.9 (9)
P—Fe—C4	90.0 (7)	P—Si2—C21	110.9 (7)
C1—Fe—C2	92.5 (8)	P—Si2—C22	107.6 (6)
C1—Fe—C3	116.9 (8)	P—Si2—C23	107.9 (6)
C1—Fe—C4	117.9 (9)	C21—Si2—C22	110.3 (8)
C2—Fe—C3	86.2 (8)	C21—Si2—C23	108.3 (9)
C2—Fe—C4	91. (1)	C22—Si2—C23	111.9 (9)
C3—Fe—C4	125.2 (8)	P—Si3—C31	107.3 (7)
Fe—P—Si1	112.6 (2)	P—Si3—C32	109.1 (7)
Fe—P—Si2	112.0 (2)	P—Si3—C33	107.7 (6)
Fe—P—Si3	113.2 (2)	C31—Si3—C32	112.2 (8)
Si1—P—Si2	106.3 (3)	C31—Si3—C33	109. (1)
Si1—P—Si3	106.6 (3)	C32—Si3—C33	111. (1)
Si2—P—Si3	105.6 (2)	Fe—C1—O1	174. (2)
P—Si1—C11	108.0 (7)	Fe—C2—O2	177. (2)
P—Si1—C12	110.0 (6)	Fe—C3—O3	174. (2)
P—Si1—C13	110.5 (7)	Fe—C4—O4	173. (1)

$= [S^2(C + R^2B) + \{P(F_0)^2\}^2]/Lp^2$ , where  $S$  = scan rate,  $C$  = total integrated peak count,  $R$  = ratio of scan time to background counting time,  $B$  = total background count,  $Lp$  = Lorentz-polarization factor. Final  $R = 0.0590$ ,  $wR = 0.0659$ .  $\Delta/\sigma(\max.) = 0.127$ , the largest peak in final  $\Delta F$  map  $\pm 0.4$  (3) e Å<sup>-3</sup>. Scattering-factor data from *International Tables for X-ray Crystallography* (1974). Final atomic parameters are listed in

*Acta Cryst.* (1988). **C44**, 751–753

## Structure of Di[bis(triphenylphosphine)iminium] Hexachlororhenate(IV)

By CHUNG-NIN CHAU, ROBERT W. M. WARDLE AND JAMES A. IBERS

*Department of Chemistry, Northwestern University, Evanston, Illinois 60208, USA*

(Received 14 October 1987; accepted 25 November 1987)

**Abstract.**  $[(C_6H_5)_3PNP(C_6H_5)_3]_2[ReCl_6]$ ,  $M_r = 1476.1$ , orthorhombic,  $Pbca$  ( $D_{2h}^{15}$ ),  $a = 16.59$  (1),  $b = 19.49$  (1),  $c = 19.88$  (1) Å,  $V = 6428$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.53$  g cm<sup>-3</sup>,  $Mo K\alpha$ ,  $\lambda(K\alpha_1) = 0.7093$  Å,  $\mu = 23.1$  cm<sup>-1</sup>,  $F(000) = 2970$  (including anomalous dispersion),  $T = 123$  K,  $R(F^2) = 0.109$  for 4127 independent reflections. The  $ReCl_6^{2-}$  anion has crystallographically imposed symmetry  $\bar{1}$ , the Re—Cl distances are 2.351 (3), 2.355 (3) and 2.376 (3) Å, and

Table 1.\* Computer programs used: *SDP-Plus* (Frenz, 1981), *ORTEPII* (Johnson, 1976). The molecule and the numbering scheme are shown in Fig. 1. Bond lengths and angles are given in Table 2.

**Related literature.** Bennett, Neustadt, Parry & Cagle (1978), Kilbourn, Raeburn & Thompson (1969), Pickardt, Rösch & Schumann (1976).

We thank the Robert A. Welch Foundation, the US Army Research Office, and the National Science Foundation for financial support.

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44595 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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the Cl—Re—Cl angles are 89.72 (11), 91.10 (10) and 91.23 (10)°.

**Experimental.** Dark yellowish-green crystals of bis(triphenylphosphine)iminium hexachlororhenate,  $[PPN]_2[ReCl_6]$ , were obtained by combining 0.5 g rhenium pentachloride, 0.1 g rhenium powder, 0.2 g selenium powder, and 1.0 g  $[PPN]Cl$  with 1.0 g bis(dimethyloctylsilyl) selenide (Chau, Wardle & Ibers,

Table 1. Positional parameters and equivalent isotropic thermal parameters

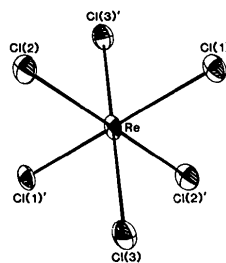
$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Re	x	y	z	$B_{eq}(\text{\AA}^2)$
Re	0	0	0	1.25 (1)
Cl(1)	0.44643 (16)	0.04833 (15)	0.59865 (15)	1.76 (7)
Cl(2)	0.61643 (15)	0.06844 (14)	0.50912 (16)	1.98 (8)
Cl(3)	0.56048 (17)	-0.08828 (15)	0.56513 (15)	2.03 (8)
P(1)	0.53584 (17)	0.11400 (16)	0.87442 (16)	1.47 (8)
P(2)	0.10591 (17)	0.31126 (15)	0.24338 (16)	1.36 (7)
N	0.55602 (53)	0.17161 (45)	0.82232 (45)	1.6 (3)
C(1)	0.53080 (63)	0.02974 (55)	0.83891 (55)	1.2 (2)
C(2)	0.54790 (72)	-0.02882 (59)	0.87630 (61)	2.1 (2)
C(3)	0.54097 (78)	-0.09265 (64)	0.84966 (64)	2.7 (3)
C(4)	0.51616 (69)	-0.09999 (60)	0.78341 (60)	2.4 (3)
C(5)	0.49952 (74)	-0.04420 (52)	0.74486 (58)	1.8 (2)
C(6)	0.50636 (70)	0.02129 (47)	0.77338 (51)	1.4 (2)
C(7)	0.43872 (64)	0.13501 (53)	0.91017 (55)	1.3 (2)
C(8)	0.40952 (66)	0.20064 (56)	0.90411 (58)	1.7 (2)
C(9)	0.33506 (70)	0.21835 (60)	0.93307 (61)	2.1 (2)
C(10)	0.29062 (66)	0.16823 (57)	0.96601 (59)	1.6 (2)
C(11)	0.32036 (74)	0.10262 (63)	0.97060 (63)	2.4 (3)
C(12)	0.39435 (66)	0.08476 (58)	0.94238 (58)	1.7 (2)
C(13)	0.60681 (64)	0.11010 (55)	0.94331 (56)	1.3 (2)
C(14)	0.58734 (63)	0.13223 (54)	1.00610 (63)	1.9 (2)
C(15)	0.64356 (73)	0.13325 (62)	1.05834 (63)	2.4 (3)
C(16)	0.72179 (69)	0.11069 (60)	1.04409 (60)	2.0 (2)
C(17)	0.74252 (72)	0.08889 (61)	0.98169 (58)	2.3 (3)
C(18)	0.68581 (71)	0.08664 (61)	0.92981 (62)	2.1 (2)
C(19)	0.03793 (66)	0.29568 (55)	0.31276 (57)	1.4 (2)
C(20)	0.06310 (67)	0.29381 (55)	0.37742 (57)	1.6 (2)
C(21)	0.00861 (75)	0.28377 (58)	0.42966 (60)	2.4 (2)
C(22)	-0.07258 (71)	0.27208 (59)	0.41394 (62)	2.2 (2)
C(23)	-0.09692 (70)	0.27091 (60)	0.34957 (61)	2.1 (2)
C(24)	-0.04438 (68)	0.28432 (55)	0.29705 (57)	1.5 (2)
C(25)	0.16383 (66)	0.23516 (57)	0.22779 (56)	1.7 (2)
C(26)	0.22454 (67)	0.21479 (58)	0.27334 (58)	1.8 (2)
C(27)	0.26915 (66)	0.15522 (58)	0.26004 (62)	2.1 (3)
C(28)	0.25069 (83)	0.11580 (71)	0.20535 (68)	3.3 (3)
C(29)	0.19007 (79)	0.13391 (69)	0.16127 (66)	3.0 (3)
C(30)	0.14555 (72)	0.19387 (62)	0.17367 (61)	2.3 (2)
C(31)	0.17597 (61)	0.37602 (53)	0.26993 (52)	1.1 (2)
C(32)	0.15900 (67)	0.42239 (58)	0.31936 (58)	1.7 (2)
C(33)	0.21219 (70)	0.47398 (58)	0.33571 (60)	2.0 (2)
C(34)	0.28553 (68)	0.47874 (55)	0.30061 (59)	1.8 (2)
C(35)	0.30358 (69)	0.43295 (56)	0.25098 (66)	2.0 (2)
C(36)	0.24881 (71)	0.38104 (57)	0.23517 (56)	1.8 (2)

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

Re—Cl(1)	2.351 (3)	Cl(1)—Re—Cl(2)	91.10 (10)
Re—Cl(2)	2.355 (3)	Cl(1)—Re—Cl(3)	89.72 (11)
Re—Cl(3)	2.376 (3)	Cl(2)—Re—Cl(3)	91.23 (10)
P(1)—N	1.564 (9)	P(1)—N—P(2)	144.14 (61)
P(2)—N	1.582 (9)	N—P(1)—C(1)	114.0 (5)
P(1)—C(1)	1.789 (10)	N—P(1)—C(7)	106.8 (5)
P(1)—C(7)	1.809 (10)	N—P(1)—C(13)	113.1 (5)
P(1)—C(13)	1.808 (10)	C(1)—P(1)—C(7)	108.7 (5)
P(2)—C(19)	1.809 (11)	C(1)—P(1)—C(13)	106.9 (5)
P(2)—C(25)	1.795 (11)	C(7)—P(1)—C(13)	107.0 (5)
P(2)—C(31)	1.795 (10)	N—P(2)—C(19)	109.8 (5)
C—C*	1.39 (2)	N—P(2)—C(25)	108.2 (5)
C—C—C*	120 (1)	N—P(2)—C(31)	115.7 (5)
		C(19)—P(2)—C(25)	109.1 (5)
		C(19)—P(2)—C(31)	107.3 (5)
		C(25)—P(2)—C(31)	106.5 (5)

\* This is the standard deviation of a single observation on the assumption that the values averaged are from the same population. The fact that the value is the same as that estimated for a single observation from the inverse matrix indicates that the standard deviations so estimated are reliable.

Fig. 1. Perspective view of the  $\text{ReCl}_6^{2-}$  ion in  $[\text{PPN}]_2[\text{ReCl}_6]$  (50% probability thermal ellipsoids) along with atom-numbering scheme.

1987) in 30 mL DMF. The brown-yellow solution was filtered after stirring at room temperature for 24 h. Addition of ether caused the precipitation of small black crystals that were filtered on a sintered-glass funnel, washed twice with ether, and suction dried. Infrared measurements on the product gave a Re—Cl stretch at  $290 \text{ cm}^{-1}$ . The above reaction did not yield a Re/Se anion, as desired. Single crystals of  $[\text{PPN}]_2[\text{ReCl}_6]$  were obtained by slow diffusion of ether into a DMF solution of the material.

A single crystal approximately  $0.38 \times 0.23 \times 0.09 \text{ mm}$  was mounted on a glass fiber with the long dimension as the axis of rotation. The crystal was bounded by  $\{001\}$  and  $\{111\}$ ; crystal volume  $6.13 \times 10^{-3} \text{ mm}^3$ . Unit-cell parameters were derived from a least-squares analysis of the setting angles of 25 reflections that had been centered at 123 K in the range  $13 < \theta < 18^\circ$  on an Enraf-Nonius CAD-4 X-ray diffractometer; graphite-monochromated Mo K $\alpha$  radiation [ $\lambda(K\alpha_1) = 0.7093 \text{ \AA}$ ]. As opposed to the limitations of the usual CAD-4 software, we imposed the condition  $\alpha = \beta = \gamma = 90^\circ$ . Intensity data were collected with the  $\omega$ -scan technique; intensities of six

standards monitored every 3 h throughout data collection were constant within intensity statistics. X-ray diffraction intensities, 4646 measured, 4127 unique, 2395 observed [ $I \geq 3\sigma(I)$ ] collected to  $(\sin \theta_{\text{max}})/\lambda = 0.5509 \text{ \AA}^{-1}$ , in the index range  $0 \leq h \leq 18$ ,  $0 \leq k \leq 21$ ,  $0 \leq l \leq 19$ , were corrected for absorption with use of the analytical method (de Meulenaer & Tompa, 1965); min. and max. transmission factors were 0.652 and 0.820.

The initial position for the Re atom was determined from a Patterson synthesis. Positions for the Cl, N, P and C atoms were obtained from subsequent electron density syntheses; full-matrix least-squares refinement on  $F$  and final refinement cycles on  $F^2$ ; phenyl H atoms included were derived with the assumption of idealized geometry; all non-H and non-C atoms refined anisotropically. Scattering factors and anomalous-dispersion terms were taken from the usual sources (Cromer & Waber, 1974; Cromer, 1974). All calculations were performed on a Harris 1000 computer with methods and programs standard for this laboratory (Waters & Ibers, 1977). The final refinement, based on 205 variables and 4127 observations, resulted in  $R(F^2) = 0.109$ ;  $wR(F^2) = 0.134$ ;  $w = 1/\sigma^2(F^2)$ ;  $S(F^2) = 1.51$ ;  $R(F)[F_o^2 \geq 3\sigma(F_o^2)] = 0.057$ ; with max.  $\Delta/\sigma = 0.046$ . A final difference electron density map exhibited no features with heights greater than 0.9% of an Re atom.

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  $\text{ReCl}_6^{2-}$  ion is shown in Fig. 1.

**Related literature.** The Re—Cl distances found here may be compared with those found in  $\text{K}_2\text{ReCl}_6$ , 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).

This research was supported by the US National Science Foundation — grant No. CHE87-01007.

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*Acta Cryst.* (1988). **C44**, 753–755

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

BY WILLIAM CLEGG

*Department of Inorganic Chemistry, The University, Newcastle upon Tyne NE1 7RU, England*

AND C. DAVID GARNER AND COLIN D. SCATTERGOOD

*Chemistry Department, Manchester University, Manchester M13 9PL, England*

(Received 13 November 1987; accepted 6 January 1988)

**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)°,  $V = 6248.5$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.929$  Mg m<sup>-3</sup>,  $F(000) = 3552$ ,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu = 6.55$  mm<sup>-1</sup>,  $T = 293$  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