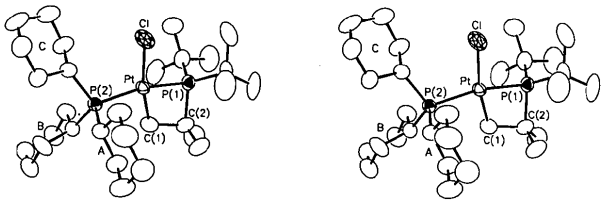

Fig. 1. An *ORTEP* stereoview of $Cl(PCy_3)Pt[P(tert\text{-}Bu)_2\text{-}C(CH_3)_2CH_2]$ with 50% probability ellipsoids.

Sappenfield & Grossie, 1986; Mullica, Oliver & Grossie, 1987). Organometallic compounds containing PR_3 ligands have been shown to be excellent catalysts for hydrogen-transfer and polymerization reactions. The steric contribution of such ligands greatly influences both rates and stereochemistries of products (Chow, Clark, Fraenkel, Grossie, Hampden-Smith, Mullica & Ruegger, 1991). Ligand bulk calculations using the program *CONE* (Smith & Oliver, 1978) help evaluate the stereochemistry.

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Structure of a Heptanuclear Tungsten–Copper Complex, $(Ph_4P)_2[Cu_5W_2O_2S_6(S_2CNEt_2)_3].DMF$

BY RONG CAO, XINJIAN LEI AND HANQIN LIU*

Fujian Institute of Research on the Structure of Matter and Fuzhou Laboratory of Structural Chemistry, Chinese Academy of Sciences, Fuzhou, Fujian 350002, *People's Republic of China*

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Abstract. Bis(tetraphenylphosphonium) bis(μ-*N*,*N*-diethyldithiocarbamato-3κS:4κS;5κS:6κS)-(diethyldithiocarbamato-7κS)-hexa-μ_3-sulfido-1$\kappa^3 S$:2$\kappa^3 S$:3$\kappa^2 S$:4$\kappa^3 S$:6$\kappa^3 S$:7$\kappa^2 S$-pentacopperdi(oxotungsten)–dimethylformamide (1/1), $2[(C_6H_5)_4P]^+.[Cu_5W_2(C_5H_{10}NS_2)_3O_2S_6]^{2-}.C_3H_7NO$, $M_r = 2106{\cdot}5$, monoclinic, $P2_1/c$, $a = 25{\cdot}210\ (3)$, $b = 22{\cdot}507\ (10)$, $c = 14{\cdot}038\ (9)$ Å, $\beta = 100{\cdot}70\ (5)°$, $V = 7826{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}788$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, $\mu = 47{\cdot}2$ cm^{-1}, $F(000) = 4160$, $T = 296\ (1)$ K, $R = 0{\cdot}059$ and $wR = 0{\cdot}064$ for 6359 reflections with $I > 3\sigma(I)$. The anion consists of two defective cubane-like units OWS_3Cu_2 and OWS_3Cu_3 linked by two weak Cu—S bonds and two bridging $S_2CNEt_2^-$ ligands.

Experimental. The title compound was prepared by reaction of $(NH_4)_2WO_2S_2$, CuCl, $NaS_2CNEt_2.3H_2O$ and Ph_4PBr in DMF solution and a crystal suitable for X-ray analysis was grown in a solution of DMF/Et_2O. Details of the sample preparation and the crystal growth are similar to that of $(Et_4N)_2[Mo_2Cu_5O_2S_6(S_2CNMe_2)_3]$ (Liu, Cao, Lei, Wu, Wei, Huang, Hong & Kang, 1990).

Diffraction intensities were collected from a purple-red crystal $0{\cdot}65 \times 0{\cdot}30 \times 0{\cdot}20$ mm in the θ–2θ scan mode on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromatized Mo $K\alpha$ radiation. Cell constants were obtained from least-squares refinement of 24 reflections, using the setting angles around 14°, measured by the computer-controlled diagonal slit method of centering. 11490 reflections were collected in the range $2 < 2\theta < 46°$ ($-27 < h < 0$, $-24 < k < 0$, $-15 < l < 15$). The intensities were monitored by 3 representative reflections. The data were corrected for fluctuation in the monitored reflections (between 1·000 and 0·818), the Lp factor and empirical absorption (between 1·042 and 0·917),

* To whom correspondence should be addressed.

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Table 1. *Positional parameters and their e.s.d.'s for* $(Ph_4P)_2[W_2Cu_5O_2S_6(S_2CNEt_2)_3]$.DMF

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	*y*	*z*	B_{eq}(Å^2)
W(1)	0·86602 (3)	−0·00648 (3)	0·91886 (5)	2·76 (2)
W(2)	0·66399 (3)	0·00658 (3)	0·94696 (5)	2·61 (1)
Cu(1)	0·81745 (9)	0·0712 (1)	0·7886 (2)	3·47 (5)
Cu(2)	0·80849 (9)	−0·09051 (9)	0·8151 (2)	3·58 (5)
Cu(3)	0·73666 (9)	0·0811 (1)	0·8864 (2)	3·63 (5)
Cu(4)	0·7249 (1)	−0·0879 (1)	0·9106 (2)	3·97 (6)
Cu(5)	0·7246 (1)	0·0116 (1)	1·1252 (2)	5·61 (7)
S(1)	0·8329 (2)	0·0815 (2)	0·9516 (4)	3·4 (1)
S(2)	0·8249 (2)	−0·0817 (2)	0·9778 (4)	3·5 (1)
S(3)	0·8534 (2)	−0·0165 (2)	0·7561 (3)	3·4 (1)
S(4)	0·6855 (2)	−0·0722 (2)	1·0445 (4)	3·7 (1)
S(5)	0·7002 (2)	−0·0061 (2)	0·8129 (3)	3·3 (1)
S(6)	0·6994 (2)	0·0885 (2)	1·0235 (3)	3·4 (1)
S(11)	0·7954 (2)	0·1419 (2)	0·6824 (4)	3·8 (1)
S(12)	0·7149 (2)	0·1700 (2)	0·8122 (4)	4·0 (1)
S(21)	0·7798 (2)	−0·1659 (2)	0·7234 (4)	3·9 (1)
S(22)	0·7076 (2)	−0·1839 (2)	0·8712 (4)	4·4 (1)
S(31)	0·8092 (3)	−0·0024 (4)	1·2248 (5)	7·9 (2)
S(32)	0·7100 (3)	0·0311 (4)	1·2884 (5)	8·5 (2)
O(1)	0·9326 (5)	−0·0085 (6)	0·9699 (9)	4·3 (3)
O(2)	0·5958 (5)	0·0164 (6)	0·915 (1)	4·9 (3)
N(1)	0·7366 (6)	0·2404 (6)	0·675 (1)	3·6 (4)
N(2)	0·7216 (6)	−0·2614 (6)	0·735 (1)	4·3 (4)
N(3)	0·8002 (9)	−0·0029 (9)	1·414 (1)	8·5 (6)
C(10)	0·7473 (7)	0·1884 (7)	0·720 (1)	3·1 (4)
C(11)	0·6957 (7)	0·2834 (8)	0·705 (2)	4·7 (5)
C(12)	0·6434 (8)	0·270 (1)	0·642 (2)	7·1 (7)
C(13)	0·7628 (8)	0·2597 (9)	0·592 (1)	4·4 (5)
C(14)	0·8091 (9)	0·302 (1)	0·629 (2)	6·3 (6)
C(20)	0·7351 (7)	−0·2073 (8)	0·776 (1)	3·5 (4)
C(21)	0·7433 (8)	−0·2859 (8)	0·650 (1)	5·1 (5)
C(22)	0·795 (1)	−0·321 (1)	0·692 (2)	7·1 (7)
C(23)	0·675 (1)	−0·2966 (8)	0·769 (2)	10 (1)
C(24)	0·702 (1)	−0·335 (2)	0·829 (3)	14 (1)
C(30)	0·7757 (9)	0·008 (1)	1·320 (2)	6·4 (6)
C(31)	0·770 (1)	0·014 (1)	1·496 (2)	11 (1)
C(32)	0·744 (1)	−0·041 (1)	1·527 (2)	9·8 (9)
C(33)	0·859 (2)	−0·021 (2)	1·436 (3)	16 (2)
C(34)	0·851 (2)	−0·078 (2)	1·451 (4)	14 (2)
O	0·5136 (6)	−0·0034 (7)	0·659 (1)	6·9 (4)*
N	0·4439 (7)	0·0434 (8)	0·711 (1)	5·9 (4)*
C(1)	0·4945 (9)	0·0157 (9)	0·728 (2)	6·3 (6)*
C(2)	0·413 (1)	0·052 (1)	0·611 (2)	8·0 (7)*
C(3)	0·423 (1)	0·063 (1)	0·800 (2)	9·8 (9)*
P(1)	0·0114 (2)	0·7642 (2)	0·3920 (4)	3·4 (1)*
P(2)	0·4843 (2)	0·8302 (2)	0·6588 (4)	3·3 (1)*
C(111)	0·0016 (7)	0·8174 (8)	0·298 (1)	3·4 (4)*
C(112)	0·0457 (8)	0·8348 (8)	0·253 (1)	4·1 (4)*
C(113)	0·0370 (8)	0·8769 (9)	0·175 (2)	4·8 (5)*
C(114)	−0·0134 (9)	0·8998 (9)	0·144 (2)	5·1 (5)*
C(115)	−0·0554 (9)	0·883 (1)	0·187 (2)	5·8 (5)*
C(116)	−0·0510 (8)	0·8409 (9)	0·263 (2)	5·1 (5)*
C(121)	0·0724 (7)	0·7826 (8)	0·475 (1)	3·4 (4)*
C(122)	0·0987 (8)	0·7363 (8)	0·531 (1)	4·1 (4)*
C(123)	0·1456 (9)	0·746 (1)	0·596 (2)	5·9 (6)*
C(124)	0·1672 (8)	0·8056 (9)	0·603 (2)	4·9 (5)*
C(125)	0·1400 (9)	0·852 (1)	0·550 (2)	5·8 (5)*
C(126)	0·0919 (8)	0·8408 (9)	0·479 (2)	4·9 (5)*
C(131)	−0·0439 (8)	0·7665 (8)	0·455 (1)	4·0 (4)*
C(132)	−0·0942 (8)	0·7390 (8)	0·410 (1)	4·0 (4)*
C(133)	−0·1369 (9)	0·7451 (9)	0·461 (2)	5·3 (5)*
C(134)	−0·1315 (9)	0·774 (1)	0·547 (2)	5·5 (5)*
C(135)	−0·0808 (9)	0·801 (1)	0·590 (2)	6·6 (6)*
C(136)	−0·0366 (9)	0·7968 (9)	0·544 (2)	5·3 (5)*
C(141)	0·0194 (7)	0·6895 (7)	0·347 (1)	3·2 (4)*
C(142)	0·0510 (8)	0·6844 (9)	0·279 (1)	4·7 (5)*
C(143)	0·065 (1)	0·628 (1)	0·255 (2)	6·8 (6)*
C(144)	0·0471 (9)	0·577 (1)	0·303 (2)	6·0 (6)*
C(145)	0·0140 (9)	0·5855 (9)	0·374 (2)	5·2 (5)*
C(146)	0·0003 (8)	0·6428 (9)	0·397 (2)	4·7 (5)*
C(211)	0·4883 (7)	0·8675 (7)	0·773 (1)	3·1 (4)*
C(212)	0·5364 (8)	0·8652 (9)	0·843 (1)	4·4 (5)*
C(213)	0·5354 (8)	0·8890 (8)	0·936 (1)	4·2 (4)*
C(214)	0·4879 (8)	0·9152 (9)	0·955 (2)	5·3 (5)*
C(215)	0·4408 (9)	0·919 (1)	0·885 (2)	5·8 (5)*
C(216)	0·4414 (8)	0·8932 (8)	0·790 (1)	4·4 (5)*
C(221)	0·4315 (7)	0·8592 (8)	0·568 (1)	3·3 (4)*
C(222)	0·3781 (8)	0·8412 (9)	0·569 (1)	4·7 (5)*
C(223)	0·3356 (9)	0·8677 (9)	0·498 (2)	5·4 (5)*
C(224)	0·3499 (9)	0·909 (1)	0·432 (2)	5·8 (5)*
C(225)	0·4017 (8)	0·9291 (9)	0·438 (1)	4·8 (5)*
C(226)	0·4432 (8)	0·9036 (8)	0·505 (1)	4·0 (4)*
C(231)	0·4674 (7)	0·7529 (8)	0·681 (1)	3·5 (4)*
C(232)	0·4809 (8)	0·7305 (8)	0·771 (1)	4·3 (4)*
C(233)	0·4659 (9)	0·671 (1)	0·789 (2)	6·1 (6)*
C(234)	0·4376 (8)	0·6394 (9)	0·711 (2)	5·0 (5)*
C(235)	0·4253 (8)	0·6612 (9)	0·621 (2)	5·0 (5)*
C(236)	0·4393 (8)	0·7209 (9)	0·600 (1)	4·5 (5)*
C(241)	0·5470 (7)	0·8321 (8)	0·617 (1)	3·5 (4)*
C(242)	0·5601 (9)	0·7841 (9)	0·562 (2)	5·3 (5)*
C(243)	0·6106 (9)	0·786 (1)	0·530 (2)	6·1 (6)*
C(244)	0·6460 (9)	0·8318 (9)	0·560 (2)	5·4 (5)*
C(245)	0·6337 (9)	0·878 (1)	0·614 (2)	5·8 (5)*
C(246)	0·5830 (8)	0·8794 (8)	0·644 (1)	4·3 (4)*

* Atoms refined isotropically.

Table 2. *Selected atomic distances* (Å) *and bond angles* (°) *for* $(Ph_4P)_2[W_2Cu_5O_2S_6(S_2CNEt_2)_3]$.DMF, *with e.s.d.'s in parentheses*

W(1)—Cu(1)	2·659 (2)	Cu(1)—S(11)	2·181 (5)
W(1)—Cu(2)	2·650 (2)	Cu(2)—S(2)	2·253 (4)
W(2)—Cu(3)	2·733 (2)	Cu(2)—S(3)	2·256 (4)
W(2)—Cu(4)	2·726 (2)	Cu(2)—S(21)	2·171 (4)
W(2)—Cu(5)	2·681 (2)	Cu(3)—S(1)	2·429 (4)
Cu(1)—Cu(3)	2·669 (3)	Cu(3)—S(5)	2·327 (4)
Cu(2)—Cu(4)	2·701 (3)	Cu(3)—S(6)	2·298 (4)
W(1)—S(1)	2·230 (3)	Cu(3)—S(12)	2·276 (5)
W(1)—S(2)	2·224 (4)	Cu(4)—S(2)	2·526 (4)
W(1)—S(3)	2·259 (4)	Cu(4)—S(4)	2·311 (4)
W(1)—O(1)	1·700 (8)	Cu(4)—S(5)	2·311 (4)
W(2)—S(4)	2·245 (4)	Cu(4)—S(22)	2·252 (4)
W(2)—S(5)	2·258 (3)	Cu(5)—S(4)	2·323 (4)
W(2)—S(6)	2·236 (4)	Cu(5)—S(6)	2·258 (4)
W(2)—O(2)	1·71 (1)	Cu(5)—S(31)	2·346 (5)
Cu(1)—S(1)	2·261 (4)	Cu(5)—S(32)	2·427 (6)
Cu(1)—S(3)	2·254 (4)		

S(1)—W(1)—S(2)	112·4 (1)	S(4)—Cu(4)—S(5)	104·6 (1)
S(1)—W(1)—S(3)	108·1 (1)	S(4)—Cu(4)—S(22)	104·7 (2)
S(1)—W(1)—O(1)	108·4 (3)	S(5)—Cu(4)—S(22)	126·9 (2)
S(2)—W(1)—S(3)	108·2 (1)	S(4)—Cu(5)—S(6)	105·7 (1)
S(2)—W(1)—O(1)	108·4 (3)	S(4)—Cu(5)—S(31)	115·9 (3)
S(3)—W(1)—O(1)	111·5 (3)	S(4)—Cu(5)—S(32)	118·8 (3)
S(4)—W(2)—S(5)	108·6 (1)	S(6)—Cu(5)—S(31)	126·7 (2)
S(4)—W(2)—S(6)	109·2 (1)	S(6)—Cu(5)—S(32)	112·6 (2)
S(4)—W(2)—O(2)	112·3 (4)	S(31)—Cu(5)—S(32)	75·1 (2)
S(5)—W(2)—S(6)	108·5 (1)	W(1)—S(1)—Cu(1)	72·6 (1)
S(5)—W(2)—O(2)	110·1 (4)	W(1)—S(1)—Cu(3)	107·7 (2)
S(6)—W(2)—O(2)	108·1 (3)	Cu(1)—S(1)—Cu(3)	69·3 (1)
S(1)—Cu(1)—S(3)	107·1 (1)	W(1)—S(2)—Cu(2)	72·6 (1)
S(1)—Cu(1)—S(11)	126·2 (2)	W(1)—S(2)—Cu(4)	113·9 (2)
S(3)—Cu(1)—S(11)	124·2 (2)	Cu(2)—S(2)—Cu(4)	68·5 (1)
S(2)—Cu(2)—S(3)	107·3 (1)	W(1)—S(3)—Cu(1)	72·2 (1)
S(2)—Cu(2)—S(21)	130·4 (2)	W(1)—S(3)—Cu(2)	71·9 (1)
S(3)—Cu(2)—S(21)	119·6 (2)	Cu(1)—S(3)—Cu(2)	109·0 (2)
S(1)—Cu(3)—S(5)	117·1 (1)	W(2)—S(4)—Cu(4)	73·5 (1)
S(1)—Cu(3)—S(6)	102·7 (2)	W(2)—S(4)—Cu(5)	71·8 (1)
S(1)—Cu(3)—S(12)	107·9 (1)	Cu(4)—S(4)—Cu(5)	108·3 (2)
S(5)—Cu(3)—S(6)	104·2 (1)	W(2)—S(5)—Cu(3)	73·2 (2)
S(5)—Cu(3)—S(12)	119·8 (2)	W(2)—S(5)—Cu(4)	73·3 (1)
S(6)—Cu(3)—S(12)	102·6 (2)	Cu(3)—S(5)—Cu(4)	111·7 (1)
S(2)—Cu(4)—S(4)	103·7 (2)	W(2)—S(6)—Cu(3)	74·1 (1)
S(2)—Cu(4)—S(5)	108·5 (1)	W(2)—S(6)—Cu(5)	73·2 (1)
S(2)—Cu(4)—S(22)	106·1 (2)	Cu(3)—S(6)—Cu(5)	111·3 (2)

but no extinction correction was made. 6359 reflections with $I > 3\sigma(I)$ were used for structure solution and refinement. Calculations were performed on a VAX 11/785 computer with the *SDP* program package (Frenz, 1978). All metal atoms were located from

the E map. A Fourier map phased by the metal atoms contained most of the remaining non-H atoms. Full-matrix least-squares refinement with anisotropic thermal parameters for all non-H atoms of the anion and isotropic thermal parameters for the non-H atoms of two units of the Ph_4P^+ cation and one molecule of DMF led to convergence with $R = 0.059$ and $wR = 0.064$. Function minimized was $\sum w(|F_o| - |F_c|)^2$, where w is defined by Killean & Lawrence (1969). Atomic scattering factors were taken from Cromer & Waber (1974). The goodness of fit (S) is 1·800. The largest Δ/σ value in the final cycle is 0·49 while the extreme peaks in the difference electron density map are 1·61 and -1.51 e Å^{-3}. Final positional parameters are given in Table 1, and selected atomic distances and bond angles are listed in Table 2.* The atomic labelling scheme and the structure of the anion are shown in Fig. 1.

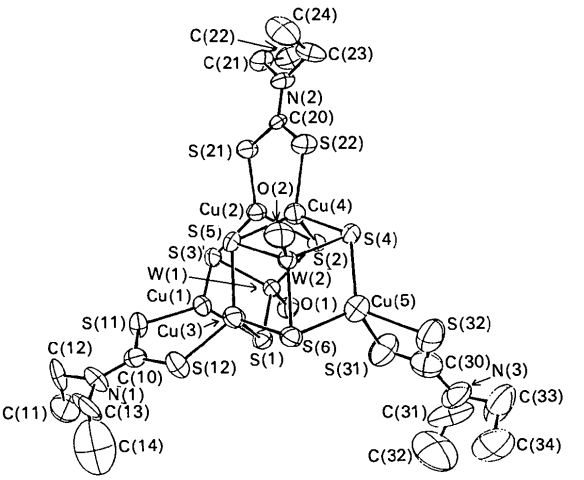

Fig. 1. Molecular structure and atomic labelling scheme for the anion $[W_2Cu_5O_2S_6(S_2CNEt_2)_3]^{2-}$.

Related literature. Crystal structures of related compounds have been studied: $(Et_4N)_2[Mo_2Cu_5S_8(S_2CNMe_2)_3].2H_2O$ (Lei, Huang, Liu, Hong & Liu, 1989); $(Et_4N)_2[W_2Cu_5S_8(S_2CNMe_2)_3]$ (Lei, Huang, Hong, Liu & Liu, 1989); $(Et_4N)_2[Mo_2Cu_5O_2S_6(S_2CNMe_2)_3]$ (Liu, Cao, Lei, Wu, Wei, Huang, Hong & Kang, 1990); $(Et_4N)_2[MoCu_3S_4(S_2CNC_5H_{10})_3].DMF$ (Lei, Liu & Liu, 1988).

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* Tables of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53620 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Chloro[tris(*m*-tolyl)phosphine]gold(I)

BY CAMERON S. W. HARKER AND EDWARD R. T. TIEKINK

Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, Adelaide, South Australia 5001, *Australia*

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Abstract. $[AuCl\{P(C_7H_7)_3\}]$, $M_r = 536.8$, orthorhombic, $P2_12_12_1$, $a = 11.236$ (1), $b = 12.994$ (4), $c = 13.371$ (2) Å, $V = 1952$ (2) Å^3, $Z = 4$, $D_x = 1.827$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å, $\mu = 7.702$ mm^{-1}, $F(000) = 1032$, $T = 293$ (1) K, $R = 0.028$ for 2341 observed reflections. The linearly coordinated Au atom is bonded to a Cl atom [2·288 (2) Å] and a P atom [2·235 (2) Å] such that Cl—Au—P is 175·1 (1)°. The dihedral angles between the mean planes through the phosphine-bound *m*-tolyl groups are 111·1, 97·0 and 101·9°.

Experimental. The title compound was prepared according to the standard literature method (Al-

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