

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 $-1.51 \text{ e } \text{\AA}^{-3}$. 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.

Related literature. Crystal structures of related compounds have been studied: $(\text{Et}_4\text{N})_2[\text{Mo}_2\text{Cu}_5\text{S}_8(\text{S}_2\text{CNMe}_2)_3]\cdot 2\text{H}_2\text{O}$ (Lei, Huang, Liu, Hong & Liu, 1989); $(\text{Et}_4\text{N})_2[\text{W}_2\text{Cu}_5\text{S}_8(\text{S}_2\text{CNMe}_2)_3]$ (Lei, Huang, Hong, Liu & Liu, 1989); $(\text{Et}_4\text{N})_2[\text{Mo}_2\text{Cu}_5\text{O}_2\text{S}_6(\text{S}_2\text{CNMe}_2)_3]$ (Liu, Cao, Lei, Wu, Wei, Huang, Hong & Kang, 1990); $(\text{Et}_4\text{N})_2[\text{MoCu}_3\text{S}_4(\text{S}_2\text{CNC}_5\text{H}_{10})_3]\cdot\text{DMF}$ (Lei, Liu & Liu, 1988).

This work was partially supported by the National Natural Science Foundation.

* 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.

Acta Cryst. (1991). **C47**, 878–879

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

(Received 10 September 1990; accepted 11 October 1990)

Abstract. $[\text{AuCl}\{\text{P}(\text{C}_7\text{H}_7)_3\}]$, $M_r = 536.8$, orthorhombic, $P2_12_12_1$, $a = 11.236$ (1), $b = 12.994$ (4), $c = 13.371$ (2) \AA , $V = 1952$ (2) \AA^3 , $Z = 4$, $D_x = 1.827 \text{ Mg m}^{-3}$, $\text{Mo } K\alpha$ radiation, $\lambda = 0.7107 \text{ \AA}$, $\mu = 7.702 \text{ mm}^{-1}$, $F(000) = 1032$, $T = 293$ (1) K, $R = 0.028$ for 2341 observed reflections. The linearly coordinated Au atom is bonded to a Cl atom

0108-2701/91/040878-02\$03.00

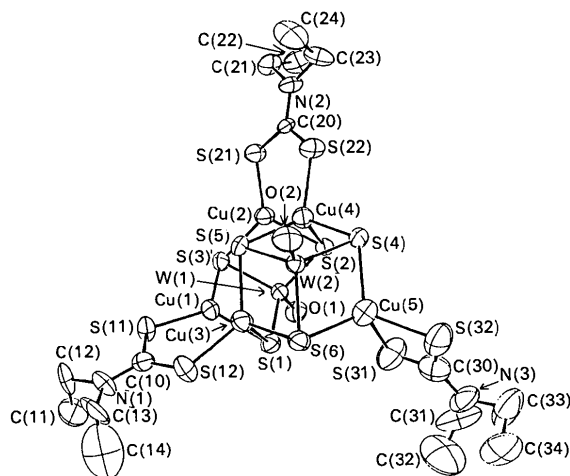


Fig. 1. Molecular structure and atomic labelling scheme for the anion $[\text{W}_2\text{Cu}_5\text{O}_2\text{S}_6(\text{S}_2\text{CNEt}_2)_3]^{2-}$.

References

- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- FRENZ, B. A. (1978). *The Enraf-Nonius CAD4 SDP - A Real-Time System of Concurrent X-ray Data Collection and Crystal Structure Solution*. In *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAEZKAMP, H. VAN KONINGSVELD & G. C. BASSI. Delft Univ. Press.
- KILLEAN, R. C. G. & LAWRENCE, J. L. (1969). *Acta Cryst.* **B25**, 1750–1752.
- LEI, X. J., HUANG, Z. Y., HONG, M. C., LIU, Q. T. & LIU, H. Q. (1989). *Inorg. Chim. Acta*, **164**, 119–121.
- LEI, X. J., HUANG, Z. Y., LIU, Q. T., HONG, M. C. & LIU, H. Q. (1989). *Inorg. Chem.* **28**, 4302–4304.
- LEI, X. J., LIU, Q. T. & LIU, H. Q. (1988). *J. Struct. Chem. (Chin.)*, **7**, 99–102.
- LIU, H. Q., CAO, R., LEI, X. J., WU, D. X., WEI, G. W., HUANG, Z. Y., HONG, M. C. & KANG, B. S. (1990). *J. Chem. Soc. Dalton Trans.* pp. 1023–1026.

[2.288 (2) \AA] and a P atom [2.235 (2) \AA] 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-

© 1991 International Union of Crystallography

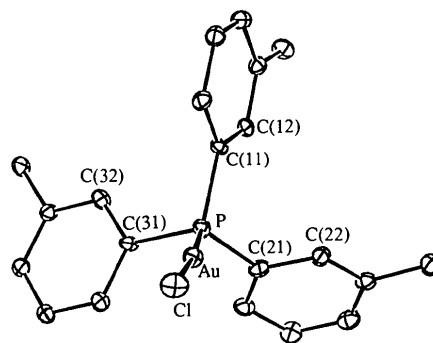
Table 1. Fractional atomic coordinates and B_{eq} values (\AA^2)
$$B_{\text{eq}} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
Au	-0.17198 (3)	-0.06978 (2)	-0.26475 (2)	3.27
Cl	-0.3597 (2)	-0.0294 (2)	-0.3183 (2)	5.05
P	0.0055 (2)	-0.1228 (2)	-0.2096 (2)	3.00
C(11)	0.0834 (7)	-0.2013 (6)	-0.2990 (6)	2.74
C(12)	0.2094 (7)	-0.2125 (6)	-0.2928 (6)	2.99
C(13)	0.2709 (8)	-0.2752 (7)	-0.3601 (7)	3.39
C(131)	0.4048 (8)	-0.2868 (9)	-0.3496 (9)	4.71
C(14)	0.2044 (8)	-0.3309 (7)	-0.4307 (7)	3.57
C(15)	0.0838 (10)	-0.3215 (7)	-0.4378 (7)	4.16
C(16)	0.0216 (9)	-0.2591 (7)	-0.3696 (7)	3.84
C(21)	0.1112 (8)	-0.0229 (6)	-0.1764 (7)	3.39
C(22)	0.1576 (8)	0.0376 (6)	-0.2542 (7)	3.49
C(23)	0.2471 (7)	0.1110 (7)	-0.2354 (8)	3.74
C(231)	0.2961 (10)	0.1707 (8)	-0.3217 (11)	5.43
C(24)	0.2808 (10)	0.1270 (9)	-0.1394 (10)	4.94
C(25)	0.2378 (9)	0.0706 (9)	-0.0618 (8)	4.88
C(26)	0.1519 (9)	-0.0076 (7)	-0.0785 (7)	4.10
C(31)	-0.0143 (7)	-0.1997 (6)	-0.0972 (6)	2.81
C(32)	0.0366 (8)	-0.2970 (8)	-0.0849 (6)	3.55
C(33)	0.0173 (8)	-0.3526 (6)	0.0009 (7)	3.54
C(331)	0.0754 (10)	-0.4573 (7)	0.0144 (8)	4.57
C(34)	-0.0572 (9)	-0.3155 (7)	0.0726 (8)	4.09
C(35)	-0.1111 (9)	-0.2186 (7)	0.0626 (7)	3.76
C(36)	-0.0923 (8)	-0.1612 (6)	-0.0241 (8)	3.68

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

Au—Cl	2.288 (2)	Au—P	2.235 (2)
P—C(11)	1.800 (8)	P—C(21)	1.814 (9)
P—C(31)	1.818 (8)		
Cl—Au—P	175.1 (1)	Au—P—C(11)	112.9 (3)
Au—P—C(21)	116.4 (3)	Au—P—C(31)	109.5 (3)
C(11)—P—C(21)	104.4 (4)	C(11)—P—C(31)	107.3 (3)
C(21)—P—C(31)	105.7 (4)		

Saady, McAuliffe, Parish & Sandbank, 1985). Crystals were grown by the slow evaporation of a CHCl_3 solution of the compound held at 277 K. Enraf-Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated $\text{Mo K}\alpha$ radiation; $\omega:2\theta$ scan technique. Cell parameters by least squares on 25 reflections ($6 \leq \theta \leq 19^\circ$) (Boer & Duisenberg, 1984) on a crystal $0.50 \times 0.17 \times 0.08$ mm. Analytical absorption correction applied; max. and min. transmission factors 0.557 and 0.249 (Sheldrick, 1976). Total of 2702 reflections ($1.5 \leq \theta \leq 25.0^\circ$) measured in the range $-13 \leq h < 1$, $0 \leq k \leq 15$, $-17 \leq l < 0$; some high-angle Friedel pairs were also included. No significant variation in the net intensities of two reference reflections ($2\bar{1}4$ and $2\bar{1}5$) measured every 7200 s. 2657 unique reflections ($R_{\text{int}} 0.019$) and 2341 satisfied $I \geq 2.5\sigma(I)$. Structure solved by Patterson method, full-matrix least-squares refinement on 218 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms included at their calculated positions. At convergence (for preferred

Fig. 1. Molecular structure and crystallographic numbering scheme employed for $[(\text{C}_{21}\text{H}_{21}\text{P})\text{AuCl}]$ (Johnson, 1971).

chirality) $R = 0.028$, $wR = 0.034$, $w = 0.14/[\sigma^2(F) + 0.008|F|^2]$, $S = 0.18$, $(\Delta/\sigma)_{\text{max}} \leq 0.001$, $\Delta\rho_{\text{max}} = 0.69$, $\Delta\rho_{\text{min}} = -0.66 \text{ e \AA}^{-3}$; no extinction correction applied. Scattering factors for neutral Au atom corrected for f' and f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV) and for the remaining atoms as incorporated in *SHELX76* (Sheldrick, 1976). All calculations on SUN4/280 computer system. Atomic parameters are given in Table 1, selected interatomic parameters in Table 2* and numbering scheme used is shown in Fig. 1 which was drawn with *ORTEPII* (Johnson, 1971) at 15% probability levels.

Related literature. The title compound has similar geometric parameters to those reported recently for chloro[tris(*o*-tolyl)phosphine]gold(I) (Harker & Tiekink, 1990).

The Australian Research Council is thanked for support.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, mean-plane data and all interatomic parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53650 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- AL-SAADY, A. K., MCAULIFFE, C. A., PARISH, R. V. & SANDBANK, J. A. (1985). *Inorg. Synth.* **23**, 191–194.
 BOER, J. L. DE & DUISENBERG, A. J. M. (1984). Enraf-Nonius CAD-4F diffractometer software update. February 1984. Groningen and Utrecht, The Netherlands.
 HARKER, C. W. S. & TIEKINK, E. R. T. (1990). *Acta Cryst.* **C46**, 1546–1547.
 JOHNSON, C. K. (1971). *ORTEPII*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Structure of *cis*-9,10-Dibenzyl-9,10-dihydroanthracene at 193 K

BY K. A. ABBOUD, S. H. SIMONSEN, R. S. PRASAD AND R. M. ROBERTS

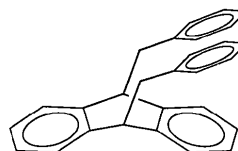
Department of Chemistry, The University of Texas at Austin, Austin, TX 78712, USA

(Received 5 July 1990; accepted 18 September 1990)

Abstract. $C_{28}H_{24}$ (1), $M_r = 360.5$, orthorhombic, $Pbca$, $a = 6.694$ (1), $b = 17.498$ (1), $c = 34.959$ (5) Å, $V = 4094.7$ (9) Å³, $Z = 8$, $D_x = 1.169$ g cm⁻³, Mo $K\alpha$ ($\lambda = 0.71069$ Å), $\mu = 0.61$ cm⁻¹, $F(000) = 1536$, $T = 193$ K, $R = 0.0402$ and $wR = 0.0413$ for 2325 reflections [$I \geq 2\sigma(I)$]. Compound (1) has an approximate molecular mirror plane perpendicular to the anthraceno phenyl rings and bisecting C(2)—C(3) and C(6)—C(7) bonds. This symmetry can also be seen from the torsion angles C(12)—C(11)···C(11')—C(12') = -2.5 (2)° and C(11)—C(10)···C(9)—C(11') = -1.2 (2)°. The dihedral angle between the C(1) and the C(5) phenyl rings, 148.1 (2)°, is considerably larger than the 135.2 (3)° in its counterpart, 9,10-dihydro-10,9-propenoanthracene, recently studied in our laboratory. Flattening of the anthraceno unit is a result of the presence of the bulky benzyl groups at the 9 and 10 positions. The planes of the C(9) and the C(10) benzyl groups make equal angles with the C(1) phenyl ring [39.1 (2) and 38.1 (2)°, respectively] and an angle of 22.1 (2)° with each other.

Experimental. In continuation of our work on Friedel–Crafts intramolecular cyclialkylation reactions using an $AlCl_3$ catalyst, we wanted to study the reaction of *cis*-9,10-dibenzyl-9,10-dihydroanthracene. Dihydroanthracene was dibenzylated using *n*-butyllithium and ammonia to obtain a product which had a melting-point range of 390–392 K (1). The crystal used in the X-ray analysis was obtained by slow evaporation from a benzene solution and was a pale yellow block of dimensions $0.21 \times 0.30 \times 0.55$ mm. Data were collected on a Siemens $R3m/V$ diffractometer equipped with a graphite monochromator utilizing Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å) and an LT-2 low-temperature delivery system (193 K). 50 reflections with $17.89 \leq 2\theta \leq 23.74^\circ$ were used to refine the cell parameters. 8188 reflections were collected using the ω -scan method (h , $0 \rightarrow 7$; k , $0 \rightarrow 20$; l , $-41 \rightarrow 41$), 3617 unique reflections, $R_{int} = 0.0166$; 2θ range $3 \rightarrow 50^\circ$, 1.2° ω scan at $4-8^\circ$ min⁻¹, depending upon intensity. Four reflections ($0\bar{2}\bar{4}$, $1\bar{1}1$, 111 , $1\bar{4}1$) were measured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was $< 1.01\%$). Absorption corrections were applied

based on measured crystal faces using *SHELXTL-Plus* (Sheldrick, 1987); min. and max. transmission 0.9805 and 0.9973.



(1)

The structure was solved by direct methods in *SHELXTL-Plus* from which the locations of all non-H atoms were obtained. The structure was refined (*SHELX76*; Sheldrick, 1976) using full-matrix least squares and the positions of all H atoms were determined from a difference Fourier map. The

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

	x	y	z	U_{eq}^*
C(1)	-10 (3)	-155 (1)	589 (1)	39 (1)
C(2)	317 (3)	-917 (1)	513 (1)	45 (1)
C(3)	339 (3)	-1439 (1)	809 (1)	46 (1)
C(4)	52 (3)	-1191 (1)	1179 (1)	41 (1)
C(4A)	-255 (3)	-418 (1)	1262 (1)	32 (1)
C(5)	-2965 (3)	678 (1)	2022 (1)	42 (1)
C(6)	-4193 (4)	1307 (1)	2049 (1)	46 (1)
C(7)	-4322 (3)	1816 (1)	1747 (1)	45 (1)
C(8)	-3190 (3)	1691 (1)	1422 (1)	38 (1)
C(8A)	-1915 (3)	1064 (1)	1393 (1)	32 (1)
C(9)	-660 (3)	947 (1)	1040 (1)	32 (1)
C(9A)	-299 (3)	106 (1)	962 (1)	31 (1)
C(10)	-528 (3)	-160 (1)	1672 (1)	35 (1)
C(10A)	-1821 (3)	545 (1)	1696 (1)	33 (1)
C(11)	1497 (4)	-37 (1)	1882 (1)	41 (1)
C(11')	1325 (3)	1409 (1)	1071 (1)	38 (1)
C(12)	2665 (3)	-764 (1)	1926 (1)	35 (1)
C(12')	2535 (3)	1396 (1)	707 (1)	35 (1)
C(13)	2154 (3)	-1288 (1)	2208 (1)	38 (1)
C(13')	2058 (3)	1892 (1)	411 (1)	41 (1)
C(14)	3094 (3)	-1992 (1)	2231 (1)	43 (1)
C(14')	3074 (4)	1872 (2)	68 (1)	53 (1)
C(15)	4568 (4)	-2183 (1)	1972 (1)	47 (1)
C(15')	4582 (4)	1349 (2)	13 (1)	57 (1)
C(16)	5127 (3)	-1664 (1)	1697 (1)	49 (1)
C(16')	5081 (4)	853 (2)	303 (1)	54 (1)
C(17)	4190 (3)	-961 (1)	1675 (1)	47 (1)
C(17')	4070 (3)	880 (1)	651 (1)	45 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.