

Fig. 1. Vue de la structure en perspective et noms des atomes de l'unité asymétrique. Les traits les plus fins représentent les liaisons hydrogène.

diammine-*trans*-dihydroxoplatine (IV) (Faggiani, Howard-Lock, Lock, Lippert & Rosenberg, 1982). Structure du *cis*-dichloro-*cis*-diammine-*trans*-dihydroxoplatine(IV) peroxyde d'hydrogène (Vollano, Blatter & Dabrowiak, 1984). Etude par diffraction des rayons X et par RMN du *trans*-dihydroxo-*cis*-diamminemalonatoplatine(IV) dihydrate, du *cis*-di-

chloro-*trans*-dihydroxo-*cis*-bis(méthylamine)platine(IV) tétrahydrate et du *cis*-dichloro-*trans*-dihydroxo-*cis*-bis(méthoxy-3 *n*-propylamine)platine(IV) (Kuroda, Ismail & Sadler, 1984). Structure du *trans*-diammine-*trans*-dichloro-*trans*-dihydroxoplatine(IV) di(peroxyde d'hydrogène) (Khodadad & Rodier, 1987).

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1,3;1,3;2,3;2,3-Tetra- μ -thio-1,2-bis(triphenylphosphine)digold(I)tungsten(VI)

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Abstract. $[\text{AuW}\{(\text{C}_6\text{H}_5)_3\text{P}\}_2\text{S}_4]$, $M_r = 1230.6$, triclinic, $P\bar{1}$, $a = 9.545(2)$, $b = 10.590(2)$, $c = 19.674(3)$ Å, $\alpha = 89.07(2)$, $\beta = 80.86(2)$, $\gamma = 67.36(2)^\circ$, $V = 1810.0$ Å³, $Z = 2$, $D_x = 2.258$ Mg m⁻³, $F(000) = 1144$, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu(\text{Mo K}\alpha) = 11.14$ mm⁻¹, $T = 293$ K, $R = 0.037$ for 5002 unique reflexions [$F \geq 3\sigma(F)$]. The compound is isostructural with 1,3;1,3;2,3;2,3-tetra- μ -thio-1,2-bis(triphenylphosphine)digold(I)molybdenum(VI) [Charnock, Bristow, Nicholson, Garner & Clegg (1987). *J. Chem. Soc. Dalton Trans.* pp. 303–306] and is composed of an approximately linear P–Au–W–Au–P chain with a tetrahedral arrangement of four S atoms around the central W atom, bridging it to the adjacent Au atoms.

Experimental. Sample preparation involved the initial synthesis of triphenylethylphosphonium (tetrathio)-

tungstate monohydrate by adding triphenylethylphosphonium bromide to an aqueous sodium tungstate/hydrogen sulfide solution. The yellow solid thus formed was reacted with (chlorotriphenylphosphine)gold(I) in dichloromethane, yielding the title compound after removal of an off-white precipitate and evaporating under reduced pressure. Crystals suitable for X-ray work were obtained by diffusing diethyl ether into a solution of the compound in nitrobenzene. Crystal dimensions 0.17 × 0.23 × 0.31 mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromatized Mo K α radiation, unit cell and crystal orientation by least-squares refinement on setting angles of 25 randomly measured reflexions ($9.6 < \theta < 12.7^\circ$), ω - 2θ scan mode used to measure 6474 reflexions (724 flagged < 0), ω -scan widths $(0.70 + 0.35 \tan \theta)^\circ$ and scan speed ranging from 1 to 5° min^{-1} depending on pre-scan intensity, $0 \leq h \leq 11$, $-12 \leq k \leq 12$, $-23 \leq$

Table 1. Fractional atomic coordinates ($\times 10^4$) [$(\times 10^5)$ W, Au] and vibrational parameters ($\text{\AA}^2 \times 10^4$) for non-hydrogen atoms

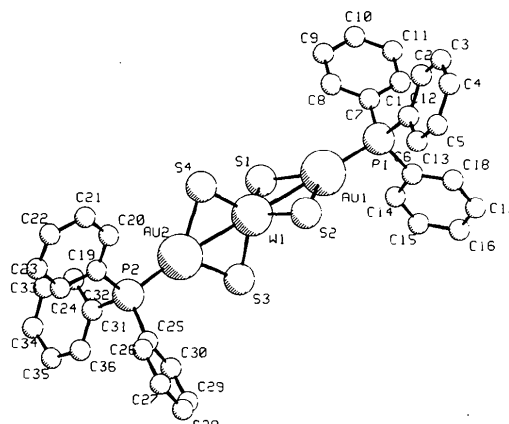
	x	y	z	U_{eq}^*
W(1)	9432 (3)	25549 (3)	25767 (1)	394
Au(1)	2090 (4)	50268 (3)	33287 (2)	521
Au(2)	17765 (4)	388 (3)	18299 (2)	543
S(1)	-1214 (2)	3575 (2)	3308 (1)	564
S(2)	2420 (3)	3731 (2)	2494 (1)	668
S(3)	349 (2)	2379 (2)	1553 (1)	570
S(4)	2204 (3)	526 (2)	2957 (1)	638
P(1)	-601 (2)	7023 (2)	3945 (1)	457
C(1)	954 (9)	7410 (7)	4199 (4)	483
C(2)	957 (11)	7785 (9)	4867 (5)	755
C(3)	2231 (13)	7957 (11)	5029 (5)	962
C(4)	3450 (12)	7800 (10)	4543 (6)	904
C(5)	3461 (10)	7433 (10)	3866 (5)	778
C(6)	2206 (9)	7236 (8)	3697 (4)	613
C(7)	-1983 (9)	7183 (8)	4709 (4)	542
C(8)	-2193 (10)	6063 (8)	4971 (4)	596
C(9)	-3256 (12)	6183 (10)	5553 (5)	829
C(10)	-4090 (11)	7442 (11)	5892 (5)	795
C(11)	-3888 (11)	8556 (10)	5638 (5)	721
C(12)	-2858 (10)	8451 (9)	5049 (4)	632
C(13)	-1628 (8)	8431 (7)	3429 (4)	423
C(14)	-2801 (9)	8308 (9)	3144 (5)	668
C(15)	-3632 (10)	9326 (10)	2751 (5)	735
C(16)	-3248 (10)	10466 (9)	2616 (4)	656
C(17)	-2083 (10)	10552 (9)	2893 (5)	669
C(18)	-1256 (10)	9560 (8)	3299 (4)	602
P(2)	2621 (2)	-1870 (2)	1137 (1)	457
C(19)	4068 (8)	-3373 (8)	1435 (4)	483
C(20)	4771 (10)	-3239 (9)	1969 (5)	715
C(21)	5880 (11)	-4371 (11)	2191 (6)	927
C(22)	6288 (10)	-5607 (9)	1878 (5)	746
C(23)	5601 (11)	-5770 (9)	1358 (5)	794
C(24)	4492 (11)	-4650 (8)	1120 (5)	782
C(25)	3502 (9)	-1546 (8)	297 (4)	541
C(26)	5049 (10)	-2234 (10)	46 (4)	710
C(27)	5671 (12)	-1885 (13)	-579 (5)	987
C(28)	4795 (13)	-885 (11)	-944 (5)	895
C(29)	3266 (13)	-199 (10)	-703 (5)	850
C(30)	2612 (11)	-514 (9)	-81 (5)	722
C(31)	1185 (9)	-2517 (7)	992 (4)	469
C(32)	327 (10)	2792 (9)	1579 (4)	726
C(33)	-690 (11)	-3383 (9)	1509 (5)	761
C(34)	-941 (11)	-3628 (10)	868 (5)	833
C(35)	-143 (12)	-3314 (10)	297 (5)	868
C(36)	914 (11)	-2772 (9)	348 (4)	724

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

$l \leq 23$, $0 \leq \theta \leq 25^\circ$, 5645 unique structure amplitudes on merging [5002 with $F \geq 3\sigma(F)$], $R_{int} = 0.009$. Fluctuations in three intensity standards [152 ($\pm 3.2\%$); 163 ($\pm 2.3\%$); 513 ($\pm 1.4\%$)] measured repeatedly at 3.3 hourly intervals, showed no significant drift. Lp and absorption corrections applied (max., min. transmissions 0.264, 0.162), Au and W atoms found from Patterson, remaining non-hydrogen atoms from difference Fourier synthesis, H atoms constrained to chemically reasonable positions, blocked-matrix least squares (F magnitudes) using *SHELX76* (Sheldrick, 1976) with non-hydrogen atoms treated anisotropically and hydrogens isotropically with vibrational parameter common to each ligand. Final $R = 0.037$, $wR = 0.044$ [$w = 1.2576/(\sigma^2(F) + 0.0004F^2)$], maximum fluctuation in final difference Fourier map in range -2.75 to 1.78 e \AA^{-3} . Maximum LS shift to e.s.d. ratio 0.038 [P(1)z]. Scattering factors from *International Tables for X-ray Crystallography* (1974), computation carried

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

W(1)—Au(1)	2.814 (1)	W(1)—Au(2)	2.831 (1)
W(1)—S(1)	2.208 (2)	W(1)—S(2)	2.199 (3)
W(1)—S(3)	2.206 (2)	W(1)—S(4)	2.206 (2)
Au(1)—S(1)	2.416 (3)	Au(1)—S(2)	2.403 (2)
Au(1)—P(1)	2.251 (2)	Au(2)—S(3)	2.426 (2)
Au(2)—S(4)	2.416 (2)	Au(2)—P(2)	2.254 (2)
P(1)—C(1)	1.824 (10)	P(1)—C(7)	1.799 (8)
P(1)—C(13)	1.832 (7)	P(2)—C(19)	1.822 (7)
P(2)—C(25)	1.822 (8)	P(2)—C(31)	1.812 (10)
S(2)—W(1)—S(1)	111.6 (1)	S(3)—W(1)—S(2)	108.7 (1)
S(3)—W(1)—S(1)	108.3 (1)	S(4)—W(1)—S(2)	108.4 (1)
S(4)—W(1)—S(1)	108.3 (1)	S(2)—Au(1)—S(1)	98.3 (1)
S(4)—W(1)—S(3)	111.6 (1)	P(1)—Au(1)—S(2)	135.7 (1)
P(1)—Au(1)—S(1)	125.9 (1)	P(2)—Au(2)—S(3)	127.7 (1)
S(4)—Au(2)—S(3)	97.8 (1)		
P(2)—Au(2)—S(4)	134.0 (1)		

Fig. 1. The title molecule including atomic labelling scheme produced using *PLUTO* (Motherwell & Clegg, 1978). H atoms have been omitted for clarity.

out on the joint CDC 7600/Amdahl 470 system of the University of Manchester Regional Computing Centre.

Atomic parameters are listed in Table 1,* bond lengths and angles in Table 2. A view of the title molecule is shown in Fig. 1.

Related literature. Details of a related structure may be found in a paper of Huffmann, Roth & Siedle (1976).

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* Lists of structure factors, hydrogen coordinates, anisotropic vibrational parameters and full molecular geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51112 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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ortho-(Benzoyl)phenylarsonic Acid

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Abstract. C₁₃H₁₁AsO₄, *M_r* = 306.15, orthorhombic, *P*2₁2₁2₁, *a* = 6.1611 (3), *b* = 13.6596 (7), *c* = 14.7371 (11) Å, *V* = 1240.26 Å³, *Z* = 4, *D_x* = 1.639 Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 2.73 mm⁻¹, *F*(000) = 616, *T* = 298 K, *R* = 0.0178 for 2125 observed reflections. There is a short non-bonded interaction [2.741 (2) Å] between the carbonyl oxygen [O(4)] and As, and both OH groups make hydrogen bonds [O(2)–O(1') 2.619 (3) and O(3)–O(1') 2.607 (3) Å] to the relatively charged terminal oxygens of symmetry-related arsonate groups.

Experimental. Crystal size 0.5 × 0.3 × 0.3 mm. Stoe-Siemens four-circle diffractometer, monochromated Mo *K*α radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 3654 reflections measured, 2θ_{max} 50°, three check reflections with no significant intensity variation. 2174 unique reflections (*R*_{int} = 0.0168), of which 2125 with *F* > 3σ(*F*) were used for all calculations (*SHELXS86*, Sheldrick, 1985; *SHELX76*, Sheldrick, 1976). Cell constants refined from ±2θ values of 54 reflections in the range 20–25°. 283 azimuthal scan reflections were used to correct the data for absorption (range of transmission factors: 0.736–0.869). An extinction correction was not necessary. Structure solution by Patterson interpretation. Refinement on *F* to *R* = 0.0178, *wR* = 0.0231; all non-H atoms anisotropic, H atoms were included using a riding model, except for those attached to O which were refined with restrained O–H bond lengths [C–H 0.96, O–H 0.90 Å, *U*(H) = 0.08 Å²]. 169 parameters refined, *S* = 1.37, weighting scheme *w*⁻¹ = σ²(*F*) + 0.00015*F*² which led to a featureless analysis of variance in terms of sinθ and *F_o*, max. Δ/σ = 0.001, max. and min. height in final Δρ map 0.39 and

Table 1. Atomic coordinates (×10⁴) and equivalent isotropic displacement parameters (Å² × 10³)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} [*]
As(1)	664 (1)	8384 (1)	692 (1)	27 (1)
O(1)	740 (2)	7177 (1)	634 (1)	31 (1)
O(2)	3078 (3)	8875 (1)	351 (1)	40 (1)
O(3)	-1123 (3)	8929 (1)	-36 (1)	35 (1)
O(4)	-3214 (3)	7775 (1)	1408 (1)	44 (1)
C(1)	403 (4)	8944 (1)	1888 (1)	30 (1)
C(2)	1982 (4)	9610 (2)	2145 (2)	40 (1)
C(3)	1826 (5)	10079 (2)	2984 (2)	50 (1)
C(4)	64 (5)	9900 (2)	3535 (2)	51 (1)
C(5)	-1458 (5)	9217 (2)	3294 (2)	43 (1)
C(6)	-1311 (4)	8710 (1)	2472 (1)	31 (1)
C(7)	-2915 (4)	7945 (2)	2211 (1)	34 (1)
C(8)	-4072 (4)	7366 (1)	2913 (1)	33 (1)
C(9)	-3179 (5)	7166 (2)	3765 (2)	44 (1)
C(10)	-4275 (5)	6561 (2)	4368 (2)	58 (1)
C(11)	-6243 (6)	6163 (2)	4139 (2)	60 (1)
C(12)	-7130 (5)	6355 (2)	3302 (2)	54 (1)
C(13)	-6069 (4)	6944 (2)	2688 (2)	42 (1)

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

–0.31 e Å⁻³ respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The absolute structure of the crystal actually chosen was determined by Rogers' (1981) η-refinement [η = 1.03 (2)].

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows a thermal ellipsoid plot with the atom numbering.

* 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 51173 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.