

lengths can be attributed to increased (*p-d*) π S–N bonding effected by the high electron density of the deprotonated negative nitrogen atom.

The mean S=O bond length of 1.432 (6) Å is not significantly different from the values found in (I) [mean 1.422 (2) Å] and (II) [mean 1.427 (1) Å]. However, a comparison of the $\bar{\nu}$ SO₂ values of [Ph₄SbN]((SO₂-CH₃)₂).CHCl₃ (1194 cm⁻¹) and of (I) and (II) (*ca* 1250 cm⁻¹) infers a decrease of the S=O bond order. A very weak H bond is indicated by the short N...H–CCl₃ distance [2.306 (9) Å]. The orientation of the chloroform molecule agrees with this assumption (Fig. 2).

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Structure of Nitrato(triphenylphosphine)gold(I), Au(PPh₃)(NO₃)

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Abstract. [AuNO₃(C₁₈H₁₅P)], *M_r* = 2081.1, monoclinic, *P*2₁/*c*, *a* = 8.922 (6), *b* = 10.131 (8), *c* = 19.592 (15) Å, β = 97.33 (6)°, *V* = 1756.4 (22) Å³, *Z* = 4, *D_x* = 1.97 g cm⁻³, λ (Mo *K* α) = 0.71073 Å, μ = 84.1 cm⁻¹, *F*(000) = 992, *T* = 295 K. Final *R* = 0.0336 for 1603 observed reflections [*F_o*² > 3σ(*F_o*²)]. The coordination of Au^I is linear [P–Au–O = 179.2 (2)°]. The short Au–P bond, 2.208 (3) Å, is attributed to the *trans* influence of the covalently bonded oxygen atom from the nitrate ligand with Au–O(1) = 2.074 (8) Å. Short, identical Au...O(3) and Au...N intramolecular distances [2.84 (1) Å] are observed.

Introduction. Au(PPh₃)(NO₃) has been widely used as a starting material for the synthesis of binuclear and polynuclear gold compounds (Khan, Wang, Heinrich & Fackler, 1988; Heinrich, Khan, Fackler & Porter, 1988; Steggerda, Bour & van der Velden, 1982). Only a few gold(I)–oxygen compounds have been structurally characterized (Jones, 1984, 1985; Hohbein, Jones, Meyer-Base, Schwarzmann & Sheldrick, 1985). The structure of the title compound was reported (Barron, Engelhardt, Healy, Oddy & White, 1987) along with structures of Au(PPh₃)*X*, where *X* = Br, I, and SCN. The abstract of this paper contains a reported Au–O distance for the title compound of 2.02 (1) Å while the text reports 2.199 (5) Å with *R* = 0.077. Examination of the coordinates suggests textual error and gross

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structural features in agreement with the present study. However, we have obtained improved crystal quality and better structural results which clearly address the question of the Au—O and Au—P distances.

Experimental. Au(PPh₃)(NO₃) was synthesized according to a literature procedure (Khan, Wang, Heinrich & Fackler, 1988). Crystals suitable for X-ray analysis were obtained by crystallization from a CH₂Cl₂/diethyl ether solution. A colorless rectangular crystal of dimensions 0.25 × 0.25 × 0.4 mm was selected for study. Cell constants were derived from least-squares refinement of 25 reflections having $20 < 2\theta < 25^\circ$. Intensity data were collected using ω -scan technique with $0 < 2\theta < 45^\circ$ ($h = -10$ to 10 , $k = 0$ to 11 , $l = 0$ to 22) on a Nicolet R3m/E diffractometer using graphite-monochromated Mo K α radiation. Scan rates varied from 2.93 to 29.30° min⁻¹ with scan width 2.0°. Three standard reflections (111, $\bar{2}15$, $\bar{2}\bar{1}5$), measured every 97 reflections, showed small (< 4%) random variations. Data were corrected for Lorentz and polarization effects. An empirical absorption correction was applied; $T_{\min} = 0.428$, $T_{\max} = 0.927$. The 2541 data collected were averaged to 1603 unique observed reflections [$F_o^2 > 3\sigma(F_o^2)$; $R_{\text{int}}(F_o) = 0.0219$]. The space group was $P2_1/c$ (systematic absences: $h0l$, $00l$, $l = \text{odd}$; $0k0$, $k = \text{odd}$). Scattering factors, including anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974). All computations were carried out using the *SHELXTL* crystallographic package (Sheldrick, 1986).

Structure solved by Patterson method which provided position for Au. All other non-hydrogen atoms positioned by Fourier methods. Phenyl rings refined as rigid groups with C—C = 1.395 Å and C—C—C = 120°; hydrogen atoms placed in calculated position (C—H = 0.970 Å) with fixed displacement parameters ($U_{\text{iso}} = 0.08 \text{ \AA}^2$). $\sum w(|F_o| - |F_c|)^2$ minimized, where $w = [\sigma^2(F_o) + 0.00052F_o^2]^{-1}$; final $R = 0.0336$, $wR = 0.0402$ and $S = 1.108$ obtained using 181 variables. Largest shift/e.s.d. in the final least-squares cycle 0.007; maximum and minimum residual electron densities in the difference Fourier map were 0.74 and -0.75 e \AA^{-3} .

Discussion. Final positional and thermal parameters are presented in Table 1.* Bond lengths and angles (including short intramolecular contacts) are given in Table 2. The molecule and the atomic labeling scheme are shown in Fig. 1. A stereoscopic view of the unit cell is shown in Fig. 2.

* Lists of structure factors, anisotropic thermal parameters, and hydrogen parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51704 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) with e.s.d.'s in parentheses

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

	x	y	z	U_{eq}
Au	3207 (1)	639 (1)	1376 (1)	45 (1)
N	1474 (10)	-703 (11)	2281 (4)	46 (4)
O(1)	2477 (10)	-955 (7)	1911 (5)	69 (4)
O(2)	1019 (10)	-1587 (10)	2624 (5)	83 (4)
O(3)	926 (10)	407 (10)	2258 (5)	76 (4)
P	4013 (3)	2333 (3)	814 (1)	34 (1)
C(16)	2847 (6)	4046 (7)	1706 (4)	55 (5)
C(15)	2805 (6)	5209 (7)	2084 (4)	59 (5)
C(14)	3977 (6)	6124 (7)	2098 (4)	58 (5)
C(13)	5191 (6)	5876 (7)	1733 (4)	56 (5)
C(12)	5233 (6)	4713 (7)	1355 (4)	46 (4)
C(11)	4061 (6)	3798 (7)	1341 (4)	36 (4)
C(26)	6189 (7)	2368 (7)	-99 (3)	47 (4)
C(25)	7658 (6)	2238 (7)	-262 (3)	56 (5)
C(24)	8832 (6)	1901 (7)	249 (3)	62 (5)
C(23)	8536 (6)	1694 (7)	922 (3)	54 (5)
C(22)	7066 (6)	1824 (7)	1086 (3)	50 (4)
C(21)	5893 (6)	2161 (7)	575 (3)	34 (4)
C(36)	2188 (8)	3941 (6)	-120 (3)	41 (4)
C(35)	1258 (8)	4162 (6)	-737 (3)	61 (5)
C(34)	952 (8)	3136 (6)	-1209 (3)	61 (5)
C(33)	1577 (8)	1889 (6)	-1064 (3)	59 (5)
C(32)	2506 (8)	1669 (6)	-446 (3)	48 (4)
C(31)	2812 (8)	2695 (6)	26 (3)	35 (4)

Table 2. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

Au—O(1)	2.074 (8)	Au—P	2.208 (3)
N—O(1)	1.248 (14)	N—O(2)	1.220 (14)
N—O(3)	1.225 (15)	P—C(11)	1.805 (8)
P—C(21)	1.806 (6)	P—C(31)	1.802 (7)
O(1)—Au—P	179.2 (2)	O(1)—N—O(2)	119.0 (11)
O(1)—N—O(3)	118.6 (10)	O(2)—N—O(3)	122.3 (10)
Au—O(1)—N	115.4 (7)	Au—P—C(11)	109.9 (3)
Au—P—C(21)	115.2 (3)	C(11)—P—C(21)	105.8 (3)
Au—P—C(31)	112.9 (2)	C(11)—P—C(31)	106.9 (3)
C(21)—P—C(31)	105.5 (3)	P—C(11)—C(16)	118.5 (2)
P—C(11)—C(12)	121.3 (2)	P—C(21)—C(26)	121.7 (2)
P—C(21)—C(22)	118.3 (2)	P—C(31)—C(36)	122.5 (2)
P—C(31)—C(32)	117.5 (2)		

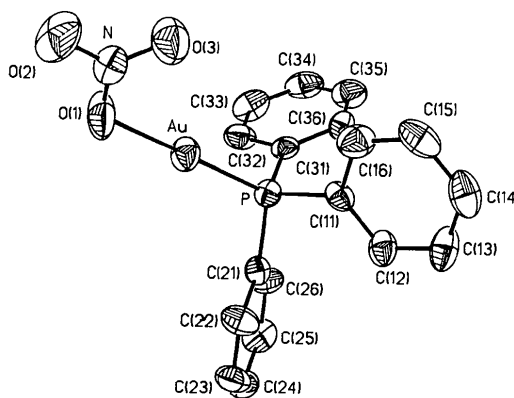


Fig. 1. A perspective view of Au(PPh₃)(NO₃). Thermal ellipsoids have been drawn at the 50% probability level.

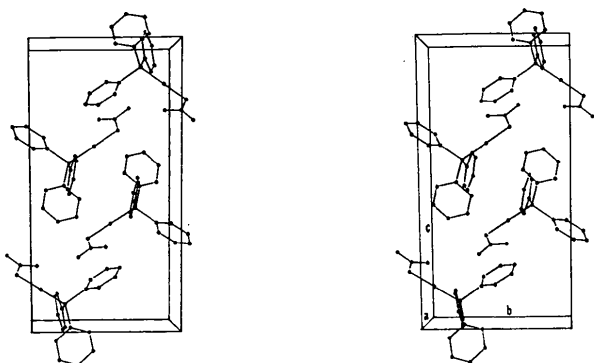


Fig. 2. A stereoview packing diagram viewed down the *a* axis.

The coordination of Au is essentially linear with $O(1)-Au-P = 179.2(2)^\circ$. The Au—P bond distance, 2.208 (3) Å, is short compared to other $AuPPh_3X$ compounds (Jones, 1984, and references therein). However, this short distance is quite common in Au^I compounds with oxygen *trans* to the Au—P bond (Jones, 1984, 1985; Hohbein, Jones, Meyer-Base, Schwarzmann & Sheldrick, 1985; Jones & Schelbach, 1988).

The nitrate is covalently bonded to gold through one oxygen atom. The Au—O(1) bond distance, 2.074 (8) Å, is comparable to other Au^I -oxygen bond distances in $AuPPh_3X$ compounds. The distortion of the Au—O(1)—N angle [$115.4(7)^\circ$] from trigonal is small but significant. The Au...O(3) [2.84 (1) Å] and Au...N [2.84 (1) Å] distances are short but non-bonding. The monodentate coordination mode of Au to the nitrate ligand in the present compound [one bonding interaction, Au—O(1), and two nonbonding inter-

actions, Au...O(3) and Au...N] is different from that observed in the metal cluster compound $[AuIr_3(NO_3)-(dppe)_3]BF_4$ (Casalnuovo, Pignolet, van der Velden, Bour & Steggerda, 1983), wherein the nitrate ligand chelates to the gold with two long Au—O distances (2.36, 2.55 Å).

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Structure of Tetramethyltin, $Sn(CH_3)_4$

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Abstract. $[Sn(CH_3)_4]$, $M_r = 178.84$, cubic, $Pa\bar{3}$, $a = 11.198(3)$ Å, $V = 1404.2$ Å³, $Z = 8$, $D_x = 1.69$ Mg m⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu(Mo K\alpha) = 3.54$ mm⁻¹, $F(000) = 688$, $T = 158$ K, $R = 0.050$, $wR = 0.058$ for 505 unique observed reflections [$I > 1.96\sigma(I)$]. The compound crystallizes in the SnI_4 structure type with the tetrahedral molecule on a

threefold axis with three equivalent Sn—C bond lengths of 2.138 (6) Å, and the fourth of 2.102 (8) Å. No significant angular distortion is observed. The slight nonequivalence of the bond lengths is in accordance with inelastic neutron scattering and NMR data.

Introduction. The molecular structures of the tetramethyl derivatives of the heavy elements of the fourth main group are well documented. Electron diffraction

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