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The Structure of $[(C_6H_5)CH_2P(C_6H_5)_3][Au(C_6F_5)Cl]$: an Organometallic Gold(I) Pentafluorophenyl Complex Useful as an Ylide Precursor

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Abstract. $[(C_6H_5)CH_2P(C_6H_5)_3][Au(C_6F_5)Cl]$, $M_r = 752.9$, triclinic, $P\bar{1}$, $a = 17.808$ (3), $b = 18.419$ (3), $c = 9.725$ (1) Å, $\alpha = 97.68$ (1), $\beta = 94.32$ (1), $\gamma = 62.36$ (1)° [(0 0 1, -1 0 0, 0 -1 0) is the transform which converts the chosen cell to a standard: $a = 9.725$ (1), $b = 17.808$ (3), $c = 18.419$ (3) Å, $\alpha = 62.36$ (1), $\beta = 82.32$ (1), $\gamma = 85.62$ (1)°], $V = 2800.3$ (8) Å³, $Z = 4$, $D_x = 1.78$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 54.5$ cm⁻¹, $F(000) = 1456$, $T = 295$ K. Convergence to final conventional R values of $R = 0.0535$ and $wR = 0.0501$ was obtained using 283 variable parameters and 3545 reflections with $F_o^2 > 3\sigma(F_o^2)$. The structure of (benzyl)(triphenyl)phosphonium chloro(pentafluorophenyl)gold(I) consists of an anion-cation pair. The anionic $[Au(C_6F_5)Cl]^-$ part is composed of a two-coordinate Au atom bonded to a pentafluorophenyl ring and a Cl atom, and is the first structurally characterized arylgold(I) halide. The counter ion is a benzyltriphenylphosphonium.

Experimental. The title complex (Usón, Laguna & Laguna, 1988; Usón, Laguna, Laguna & Usón, 1983), along with the $Au(C_6F_5)(SC_4H_9)$ starting material (Usón, Laguna & Vicente, 1977; Usón, Laguna, Laguna & Fernandez, 1980), were prepared according to the literature procedure. Benzyltriphenylphosphonium chloride was obtained from Alfa Products and

used as received. Crystals suitable for X-ray analysis were obtained by recrystallization from an ethyl alcohol/hexane solution cooled to 278 K.

A single multi-faceted white crystal of approximate dimensions 0.25 × 0.15 × 0.10 mm was mounted in a random orientation on a glass fiber. Triclinic symmetry was suggested on the basis of interaxial angles and confirmed by a Delaunay reduction. Axial lengths were checked by comparison with the interlayer spacings observed in axial photographs. Refined cell parameters were obtained from the setting angles of 25 reflections with $25 < 2\theta < 31^\circ$. Data collection was carried out at room temperature using the ω scanning technique in bisecting geometry. (Nicolet *R3m/E* diffractometer, graphite-monochromated $Mo K\alpha$ radiation.) Intensities were measured for 3545 unique reflections ($h < 20$, $|k| < 20$, $|l| < 11$) with $0 < 2\theta < 45^\circ$, and no symmetry-equivalent reflections were collected. Scan rate was variable, 3–30° min⁻¹; scan range -0.8 in ω from $K\alpha_1$ to +0.8 from $K\alpha_2$. Backgrounds were estimated from a 96-step peak profile. Three low-angle standards ($\bar{1}21$, $3\bar{1}1$, $25\bar{1}$) measured every 97 data. No decay in these check reflections was noted through completion of data collection. Corrections for absorption were applied empirically on the basis of azimuthal scans of nine reflections spanning a range of 2θ values (minimum transmission, 0.304 and 0.475, respectively). Structure solution and refinement were carried out using the *SHELXTL* collection of crystallographic software (Sheldrick, 1981). The Au position was determined from a sharpened Patterson map; all remaining non-hydrogen atoms located on difference

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>
Au(1)	2435 (1)	283 (1)	3200 (1)	58 (1)*
Au(2)	3804 (1)	4206 (1)	1699 (1)	49 (1)*
Cl(1)	3806 (3)	-138 (3)	2528 (4)	41 (2)*
Cl(2)	5113 (2)	4202 (3)	1928 (4)	29 (2)*
P(1)	7609 (3)	2801 (3)	8773 (5)	43 (2)*
P(2)	3841 (3)	2079 (3)	5205 (4)	36 (2)*
C(1)	6953 (12)	3765 (12)	9834 (17)	55 (10)*
C(2)	4063 (10)	1131 (11)	5921 (17)	41 (9)*
C(11)	9231 (8)	2595 (8)	9337 (10)	60 (6)
C(12)	9974	2618	9046	79 (7)
C(13)	10062	2835	7769	77 (7)
C(14)	9406	3029	6783	80 (7)
C(15)	8663	3006	7074	58 (6)
C(16)	8575	2789	8352	46 (5)
C(21)	8560 (7)	1192 (8)	9279 (11)	60 (6)
C(22)	8738	497	9927	86 (8)
C(23)	8199	544	10941	92 (8)
C(24)	7481	1286	11308	83 (7)
C(25)	7303	1981	10660	61 (6)
C(26)	7842	1934	9645	45 (5)
C(31)	7028 (7)	2042 (6)	6651 (12)	56 (6)
C(32)	6586	2009	5416	69 (6)
C(33)	6125	2721	4759	62 (6)
C(34)	6105	3464	5335	79 (7)
C(35)	6547	3497	6570	50 (5)
C(36)	7008	2785	7228	47 (5)
C(41)	7445 (7)	3709 (7)	12400 (13)	59 (6)
C(42)	7892	3885	13523	64 (6)
C(43)	8374	4284	13345	70 (6)
C(44)	8404	4507	12045	68 (6)
C(45)	7961	4331	10922	64 (6)
C(46)	7479	3932	11100	46 (5)
C(51)	2427 (7)	2296 (7)	3565 (11)	50 (5)
C(52)	1556	2647	3291 (6)	62 (6)
C(53)	995	3283	4216	72 (7)
C(54)	1305	3568	5413	72 (7)
C(55)	2176	3217	5687	61 (6)
C(56)	2736	2581	4763	44 (5)
C(61)	4178 (6)	2243 (6)	2574 (11)	39 (5)
C(62)	4675	2025	1401	57 (6)
C(63)	5466	1330	1336	59 (6)
C(64)	5760	854	2444	59 (6)
C(65)	5263	1072	3617	54 (6)
C(66)	4472	1767	3682	42 (5)
C(71)	3822 (7)	3576 (7)	6060 (10)	59 (6)
C(72)	4084	4094	6902	67 (6)
C(73)	4643	3797	8012	60 (6)
C(74)	4940	2982	8280	59 (6)
C(75)	4678	2465	7437	48 (5)
C(76)	4118	2761	6327	36 (4)
C(81)	2695 (7)	1289 (8)	6852 (10)	51 (5)
C(82)	2226	1294	7943	67 (6)
C(83)	2564	1243	9289	78 (7)
C(84)	3369	1187	9545	78 (7)
C(85)	3837	1182	8453	69 (6)
C(86)	3500	1234	7107	47 (5)
C(101)	515 (10)	1180 (9)	3020 (11)	71 (7)
C(102)	-305	1442	3473	90 (8)
C(103)	-428	1227	4727	83 (7)
C(104)	268	749	5529	82 (7)
C(105)	1088	486	5076	80 (7)
C(106)	1212	702	3821	67 (6)
F(11)	645	1394 (7)	1840 (12)	86 (7)*
F(12)	-941 (9)	1870 (9)	2660 (17)	135 (10)*
F(13)	-1216 (10)	1471 (11)	5104 (20)	165 (12)*
F(14)	155 (10)	559 (10)	6752 (14)	123 (11)*
F(15)	1693 (8)	22 (8)	5851 (12)	95 (8)*
C(201)	2441 (7)	3829 (8)	302 (11)	60 (6)
C(202)	1633	3879	148	78 (7)
C(203)	1037	4312	1188	64 (6)
C(204)	1248	4695	2381	64 (6)
C(205)	2055	4645	2534	44 (5)
C(206)	2651	4212	1495	54 (5)
F(21)	2983 (7)	3415 (8)	-699 (11)	89 (8)*
F(22)	1441 (8)	3510 (10)	-976 (14)	116 (10)*
F(23)	265 (7)	4340 (9)	1032 (15)	109 (9)*
F(24)	672 (8)	5116 (9)	3372 (14)	115 (9)*
F(25)	2219 (7)	5021 (7)	3679 (11)	88 (7)*

Fourier maps (Table 1 contains atomic coordinates).^{*} Phenyl rings were refined as idealized polygons (C—C = 1.395 Å, C—C—C = 120°) using H atoms placed in calculated positions with fixed thermal parameters [$U(\text{H}) = 0.08 \text{ \AA}^2$]. All non-hydrogen atoms were refined anisotropically. (See Figs. 1 and 2. Bond angles and distances are given in Table 2.) Initial refinement was based on F with unit weights. The final refinement employed weights of the form $w^{-1} = [\sigma^2(F) +$

^{*} 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 44875 (46 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

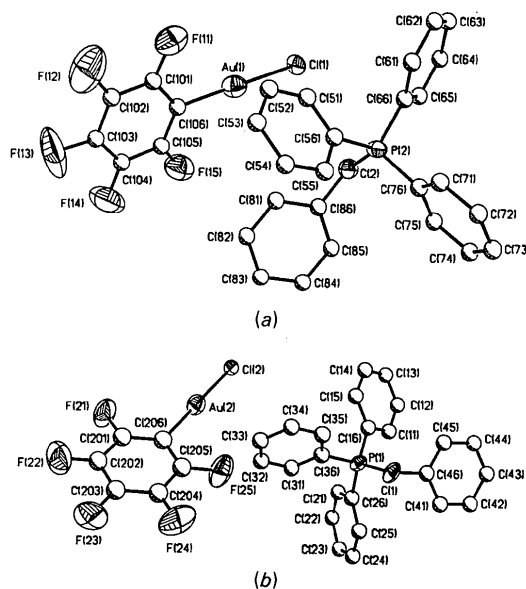


Fig. 1. Perspective views of the two independent molecules. Thermal ellipsoids have been drawn at the 50% probability level. H atoms have not been included. Phenyl group carbons are of arbitrary radii. Differences between the two molecules in the asymmetric unit are not significant. (a) The molecule containing Au(1). (b) The molecule with Au(2).

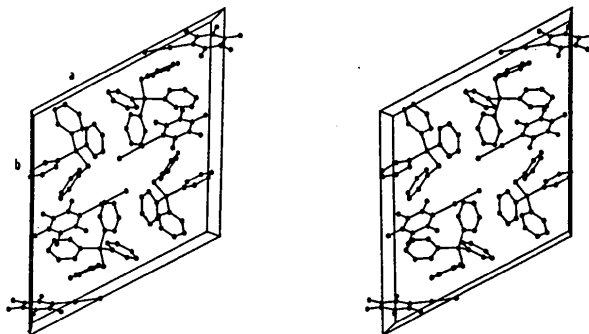


Fig. 2. A stereo packing diagram viewed down the c axis. H atoms have not been included.

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

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

Au(1)—Cl(1)	2.311 (5)	Au(1)—C(106)	2.054 (17)
Au(2)—Cl(2)	2.322 (5)	Au(2)—C(206)	2.042 (15)
P(1)—C(1)	1.830 (16)	P(1)—C(16)	1.787 (17)
P(1)—C(26)	1.772 (16)	P(1)—C(36)	1.783 (13)
P(2)—C(2)	1.826 (21)	P(2)—C(56)	1.789 (12)
P(2)—C(66)	1.792 (12)	P(2)—C(76)	1.772 (14)
C(1)—C(46)	1.574 (23)	C(2)—C(86)	1.520 (22)
F(11)—C(101)	1.331 (20)	F(12)—C(102)	1.316 (19)
F(13)—C(103)	1.323 (24)	F(14)—C(104)	1.338 (21)
F(15)—C(105)	1.286 (17)	F(21)—C(201)	1.312 (15)
F(22)—C(202)	1.317 (20)	F(23)—C(203)	1.348 (21)
F(24)—C(204)	1.339 (17)	F(25)—C(205)	1.318 (17)
Cl(1)—Au(1)—C(106)	177.8 (5)	Cl(2)—Au(2)—C(206)	179.8 (4)
C(1)—P(1)—C(16)	109.9 (9)	C(1)—P(1)—C(26)	111.2 (8)
C(1)—P(1)—C(36)	106.2 (7)	C(2)—P(2)—C(56)	107.8 (8)
C(2)—P(2)—C(66)	106.1 (6)	C(2)—P(2)—C(76)	114.3 (7)
P(1)—C(1)—C(46)	110.9 (10)	P(2)—C(2)—C(86)	115.5 (10)
P(1)—C(16)—C(11)	119.4 (4)	P(1)—C(16)—C(15)	120.6 (4)
P(1)—C(26)—C(21)	119.3 (4)	P(1)—C(26)—C(25)	120.7 (4)
P(1)—C(36)—C(31)	119.0 (4)	P(1)—C(36)—C(35)	120.9 (4)
C(1)—C(46)—C(41)	120.6 (10)	C(1)—C(46)—C(45)	119.4 (10)
P(2)—C(56)—C(51)	120.5 (4)	P(2)—C(56)—C(55)	119.3 (4)
P(2)—C(66)—C(61)	119.3 (3)	P(2)—C(66)—C(65)	120.7 (3)
P(2)—C(76)—C(71)	120.5 (4)	P(2)—C(76)—C(75)	119.4 (4)
C(2)—C(86)—C(81)	121.0 (8)	C(2)—C(86)—C(85)	118.6 (8)
C(102)—C(101)—F(11)	120.8 (8)	C(106)—C(101)—F(11)	119.2 (8)
C(101)—C(102)—F(12)	117.7 (10)	C(103)—C(102)—F(12)	122.2 (11)
C(102)—C(103)—F(13)	118.1 (10)	C(104)—C(103)—F(13)	121.8 (10)
C(103)—C(104)—F(14)	120.4 (9)	C(105)—C(104)—F(14)	119.6 (9)
C(104)—C(105)—F(15)	115.9 (10)	C(106)—C(105)—F(15)	124.0 (10)
Au(1)—C(106)—C(101)	121.9 (3)	Au(1)—C(106)—C(105)	118.1 (3)
C(202)—C(201)—F(21)	118.1 (9)	C(206)—C(201)—F(21)	121.9 (9)
C(201)—C(202)—F(22)	119.6 (9)	C(203)—C(202)—F(22)	120.4 (9)
C(202)—C(203)—F(23)	119.1 (7)	C(204)—C(203)—F(23)	120.9 (7)
C(203)—C(204)—F(24)	119.9 (9)	C(205)—C(204)—F(24)	120.1 (9)
C(204)—C(205)—F(25)	117.5 (8)	C(206)—C(205)—F(25)	122.5 (8)
Au(2)—C(206)—C(201)	120.9 (3)	Au(2)—C(206)—C(205)	119.1 (3)

0.00038(F^2)]. The value of g , 0.00038, was refined by fitting $(F_o - F_c)^2$ to $[\sigma^2(F) + gF^2]/k$ (k = scale factor) to put weights on an approximately absolute scale. Neutral scattering factors, including terms for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974). Convergence to conventional R values of $R = 0.0535$ and $wR = 0.0501$ was obtained using 283 variable param-

eters and 3545 reflections with $F_o^2 > 3\sigma(F_o^2)$. For final cycle, maximum shift/e.s.d. = 0.016 with a goodness-of-fit indicator of 1.370. Residual electron density on final difference Fourier map, using unique data, of +1.45 and -0.69 e Å⁻³ was found in the vicinity of Cl(2).

Related literature. A number of related pentafluorophenyl complexes have been reported and characterized (Usón, Laguna, Laguna & Fernandez, 1982; Lever, 1986; Melnik & Parish, 1986).

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N,N-Diethyl-4-[2,2,4,4-tetrakis(trifluoromethyl)-2*H*,4*H*-1,3,5-dioxazin-6-ylthio]-2,2,5,5-tetrakis(trifluoromethyl)-2,5-dihydro-3-furylamine

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Abstract. C₁₉H₁₀F₂₄N₂O₃S, $M_r = 802.3$, orthorhombic, *Pbca*, $a = 14.425$ (2), $b = 14.469$ (2), $c = 26.660$ (5) Å, $V = 5564$ Å³, $Z = 8$, $D_x = 1.915$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.3$ mm⁻¹, $F(000) = 3152$, $T = 293$ K. The structure was refined

to $R = 0.063$ for 2990 independent reflections. The compound is isostructural with its selenium analogue. The furyl ring is planar (mean deviation 0.02 Å), while five atoms of the dioxazine ring also lie in a plane, to a reasonable approximation [mean deviation 0.06 Å;