

O(3) atoms, 1.633 (2) and 1.613 (3) Å (see Table 2). The O—V—O angles are in the range 106.95 (11) to 111.85 (10)°, indicating a rather undistorted tetrahedral environment of the V atom.

Interatomic distances and bond angles in the VO₄ tetrahedra are in good agreement with those found by Bensch, Hug, Reller & Oswald (1987). They are within the range observed in other metavanadates. The doubly protonated molecules of the hexanediamine are located between the (VO₃)_∞ chains. As indicated by the dotted lines in Fig. 1, a hydrogen-bond network is formed between H(7), H(8), H(9) and O(2^{iv}), O(2^v), O(3^{vi}) oxygen atoms, which are terminal O atoms of VO₄ tetrahedra. The geometries for the strongest hydrogen bonds are also given in Table 2.

References

- ANDRETTI, G. D., CALESTANI, G., MONTENERO, A. & BETTINELLI, M. (1984). *Z. Kristallogr.* **168**, 53–58.
 ANGENAULT, J. (1970). *Rev. Chim. Minér.* **7**, 651–699.
 ANGENAULT, J. & RIMSKY, A. (1968). *CR Acad. Sci. Sér. C*, **267**, 227–230.
 BENSCH, W., HUG, P., RELLER, A. & OSWALD, H. R. (1987). *Mater. Res. Bull.* **22**, 577–583.
 BOULOUX, J. C. & GALY, J. (1969). *Bull. Soc. Chim. Fr.* pp. 736–740.
 BOULOUX, J. C., PEREZ, G. & GALY, J. (1972). *Bull. Soc. Fr. Minéral. Cristallogr.* pp. 130–133.
 CALESTANI, G., ANDRETTI, G. D., MONTENERO, A. & BETTINELLI, M. (1985). *Acta Cryst.* **C41**, 177–179.
 CALESTANI, G., ANDRETTI, G. D., MONTENERO, A., BETTINELLI, M. & REBIZANT, J. (1985). *Acta Cryst.* **C41**, 179–182.
 CALVO, C. & MANOLESCU, D. (1973). *Acta Cryst.* **B29**, 1743–1745.
 GANNE, M., PIFFARD, Y. & TOURNOUX, M. (1974). *Can. J. Chem.* **52**, 3539–3543.
 JASPER-TÖNNIES, B. & MÜLLER-BUSCHBAUM, Hk. (1984). *Z. Anorg. Allg. Chem.* **508**, 7–11.
 JORDAN, B. D. & CALVO, C. (1974). *Can. J. Chem.* **52**, 2701–2704.
 KARPOV, O. G., SIMONOV, H. A., KRASNENKO, T. I. & ZABARA, O. A. (1989). *Kristallografiya*, **34**(6), 1392.
 LAUNAY, S. & THORET, J. (1973). *CR Acad. Sci. Sér. D*, **277**, 541–543.
 MARUMO, F., ISOBE, M. & IWAI, S. (1974). *Acta Cryst.* **B30**, 1628–1630.
 NG, H. N. & CALVO, C. (1972). *Can. J. Chem.* **50**, 3619–3624.
 PAVELČÍK, F. & SIVÝ, J. (1989). *XFPS89*. A computer program for automatic structure determination by Fourier, Patterson and superposition methods. J. A. Komenský Univ., Bratislava, Czechoslovakia.
 PETRÁŠOVÁ, M., MADAR, J. & HANIC, F. (1958). *Chem. Zvesti*, **12**, 410–418.
 SAUERBREI, E. E. (1972). MSc Thesis, McMaster Univ., Canada.
 SHANNON, R. D. & CALVO, C. (1973). *Can. J. Chem.* **51**, 265–273.
 SYNEČEK, V. & HANIC, F. (1954). *Czech. J. Phys.* **4**, 120–130.

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[(Methyldiphenylphosphonio)methanide]tris(pentafluorophenyl)gold(III) Acetone Solvate

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Abstract. [Au(C₁₄H₁₅P)(C₁₈F₁₅)]₃.C₃H₆O, $M_r = 970.45$, triclinic, $P\bar{1}$, $a = 11.258$ (3), $b = 13.586$ (3), $c = 13.969$ (3) Å, $\alpha = 108.77$ (2), $\beta = 101.69$ (2), $\gamma = 112.07$ (1)°, $V = 1742$ (1) Å³, $Z = 2$, $D_x = 1.850$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 4.4$ mm⁻¹, $F(000) = 936$, $T = 293$ K, $R = 0.036$ for 6980 reflections. The coordination at the Au atom is square planar, with Au—C(ylide) 2.109 (6), Au—C₆F₅ 2.055–2.069 (4–6) Å and P—C(ylide) 1.773 (6) Å. No *trans* effect is observed.

Introduction. Organogold chemistry is one of the main topics of research in the groups of Professors R. Usón and A. Laguna, University of Zaragoza, Spain; for recent review articles see Usón (1986), Laguna & Laguna (1990). In particular, they have

reported the synthesis of the [tris(pentafluorophenyl)](ylide)gold(III) complexes (C₆F₅)₃Au(CH₂-PPh₃) (Usón, Laguna, Laguna & Usón, 1983) and, in a short communication, (C₆F₅)₃Au(CH₂PPh₂Me) (Usón, Laguna, Laguna, Usón & Gimeno, 1987). Here we present the crystal structure of the latter compound.

Experimental. Colourless tablets were obtained from acetone/hexane. A crystal 0.6 × 0.2 × 0.1 mm was mounted in a glass capillary. Using a Stoe four-circle diffractometer, 9141 intensities were registered to $2\theta_{\text{max}} = 55^\circ$ with monochromated Mo $K\alpha$ radiation; θ -scan technique. Of 7968 unique reflections ($R_{\text{int}} = 0.033$, index ranges $h - 14$ to 14 , $k - 16$ to 17 , $l 0$ to 18), 6980 $> 4\sigma(F)$ were considered observed. The cell

constants were refined from $\pm \omega$ angles of 48 reflections in the 2θ range 20–23°. Three check reflections showed no significant intensity variation. An absorption correction based on ψ scans was applied, with transmission factors 0.67–1.00.

The structure was solved by the heavy-atom method and subjected to anisotropic full-matrix least-squares refinement on F . A poorly resolved acetone molecule was refined isotropically. H atoms (except those of solvent) were included in the refinement using a riding model. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.00015F^2$; final $R = 0.035$, with $wR = 0.038$. 458 parameters; $S = 1.7$; maximum $\Delta/\sigma = 0.2$ for solvent, otherwise < 0.04 ; maximum, minimum $\Delta\rho = 1.0, -0.7 \text{ e } \text{Å}^{-3}$. Program system: *SHELXTL-Plus* (Sheldrick, 1987). Atomic scattering factors and f' , f'' values from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atom coordinates are given in Table 1, with selected bond lengths and angles in Table 2.*

Discussion. Fig. 1 shows a plot of the title compound. As would be expected for a gold(III) complex, the coordination geometry is square planar, with the Au atom 0.05 Å out of the plane of the four C donor atoms. The C₆F₅ groups are approximately perpendicular to the ligand plane (dihedral angles 89, 77, 75°).

A pitfall in the structure determination of metal C₆F₅ complexes is the use of rigid idealized aromatic rings in structure refinement (Jones, 1988). Because the *ipso* ring angles are often much less than 120° in such complexes, idealized rings lead to Au—C bonds that are systematically too long, by several hundredths of an ångström. For structures involving third-row transition metals, or for datasets that are imprecise because of solvent or other disorder, the systematic errors thus introduced may be small compared to the e.s.d.'s of metal—carbon bond lengths, and the use of rigid groups can be recommended to increase the stability of refinement. However, the refinement of accurate data should not involve idealized C₆F₅ groups.

In the current structure, the Au—C₆F₅ bond lengths of 2.055 (4)–2.069 (4) Å show no *trans* effect and may be considered normal; cf. 2.037 (3) (*trans*), 2.067, 2.076 (4) Å in (C₆F₅)₃Au(S₂CPEt₃) (Usón, Laguna, Laguna, Castilla, Jones & Fittschen, 1987). Less precise determinations of (C₆F₅)₃AuL complexes gave Au—C 2.104 (12) (*trans*), 2.065,

* Lists of structure factors, anisotropic thermal parameters, bond distances and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54885 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0089]

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

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Au	5110.7 (2)	1774.6 (1)	3686.5 (1)	44 (1)
P	3685 (1)	2494 (1)	5443 (1)	44 (1)
C(11)	5756 (4)	1842 (4)	2414 (4)	54 (2)
C(12)	6066 (5)	990 (5)	1873 (4)	62 (3)
C(13)	6556 (6)	1003 (6)	1037 (5)	82 (3)
C(14)	6740 (6)	1886 (7)	726 (5)	83 (4)
C(15)	6438 (6)	2744 (5)	1248 (4)	73 (3)
C(16)	5958 (5)	2719 (4)	2073 (4)	59 (2)
F(12)	5883 (4)	91 (3)	2144 (3)	86 (2)
F(13)	6852 (5)	136 (4)	540 (3)	122 (3)
F(14)	7206 (5)	1927 (5)	-73 (3)	132 (3)
F(15)	6612 (4)	3624 (4)	936 (3)	109 (2)
F(16)	5646 (4)	3590 (3)	2541 (3)	81 (2)
C(21)	3097 (4)	721 (4)	2622 (4)	56 (2)
C(22)	2446 (5)	-479 (5)	2313 (4)	68 (3)
C(23)	1064 (7)	-1205 (5)	1621 (5)	86 (3)
C(24)	321 (6)	-722 (7)	1247 (5)	88 (4)
C(25)	912 (6)	447 (7)	1519 (5)	82 (4)
C(26)	2296 (5)	1149 (5)	2198 (4)	68 (3)
F(22)	3139 (4)	-977 (3)	2704 (3)	91 (2)
F(23)	481 (4)	-2378 (3)	1371 (4)	124 (2)
F(24)	-1029 (4)	-1413 (4)	611 (3)	128 (3)
F(25)	175 (4)	904 (5)	1150 (3)	113 (3)
F(26)	2841 (3)	2315 (3)	2469 (3)	86 (2)
C(31)	7144 (4)	2739 (4)	4751 (3)	46 (2)
C(32)	7778 (4)	2203 (4)	5163 (4)	52 (2)
C(33)	9102 (5)	2800 (4)	5901 (4)	61 (3)
C(34)	9852 (5)	3993 (5)	6269 (5)	66 (3)
C(35)	9256 (5)	4560 (4)	5882 (5)	63 (2)
C(36)	7937 (5)	3941 (4)	5146 (4)	54 (2)
F(32)	7070 (3)	1007 (2)	4816 (3)	71 (2)
F(33)	9671 (3)	2209 (3)	6277 (3)	95 (2)
F(34)	11132 (3)	4591 (3)	7017 (3)	100 (2)
F(35)	9993 (3)	5750 (3)	6252 (3)	94 (2)
F(36)	7398 (3)	4567 (3)	4803 (3)	80 (2)
C(41)	1898 (4)	1744 (4)	4583 (3)	45 (2)
C(42)	1128 (4)	564 (4)	4276 (4)	52 (2)
C(43)	-272 (5)	-51 (4)	3617 (4)	58 (2)
C(44)	-876 (5)	553 (5)	3284 (4)	65 (3)
C(45)	-125 (5)	1739 (5)	3603 (4)	69 (3)
C(46)	1260 (5)	2341 (4)	4244 (4)	60 (2)
C(51)	3744 (4)	2735 (4)	6800 (4)	49 (2)
C(52)	3260 (6)	3469 (6)	7310 (5)	76 (4)
C(53)	3262 (8)	3642 (7)	8347 (6)	96 (5)
C(54)	3761 (7)	3119 (6)	8883 (5)	82 (4)
C(55)	4236 (7)	2389 (5)	8373 (5)	82 (4)
C(56)	4220 (6)	2176 (4)	7335 (4)	65 (3)
C(1)	4540 (4)	1673 (4)	5014 (4)	50 (2)
C(2)	4502 (5)	3913 (4)	5446 (4)	59 (2)
O(100)	2821 (7)	5775 (7)	2743 (6)	151 (2)
C(101)	2059 (15)	4974 (13)	1788 (12)	184 (5)
C(102)	2936 (25)	4405 (22)	1274 (19)	349 (12)
C(103)	1814 (22)	5593 (19)	1030 (17)	309 (10)

2.085 (11) Å [$L = \text{diars}$ (Usón, Laguna, Laguna, Fernandez, Jones & Sheldrick, 1982)]; with rigid groups 2.028 (24) (*trans*), 2.064 (20), 2.071 (25) Å [$L = \text{Cl}^-$ (Usón, Laguna, Laguna, Fernandez, Villacampa, Jones & Sheldrick, 1983)]. The structure of the $[\text{Au}(\text{C}_6\text{F}_5)_4]^-$ anion has also been determined (Murray, Fackler, Porter, Briggs, Guerra & Lagow, 1987), but unfortunately with rigid groups: Au—C 2.075–2.098 Å (e.s.d.'s 0.010–0.013 Å).*

Bond lengths at the ylidic C atom are C(1)—Au 2.109 (6), C(1)—P 1.773 (6) Å. The former may be compared with 2.111 (17) Å in (PPh₃CH₂)Au(SCN)₃

* Note added in proof: We have recently redetermined this structure with non-idealized C₆F₅ groups (P. G. Jones & E. Bembenek, unpublished) and obtained Au—C bond lengths of 2.053–2.060 (8) Å.

Table 2. Selected bond lengths (Å) and angles (°)

Au—C(11)	2.067 (6)	Au—C(21)	2.055 (4)
Au—C(31)	2.069 (4)	Au—C(1)	2.109 (6)
P—C(41)	1.794 (4)	P—C(51)	1.800 (5)
P—C(1)	1.773 (6)	P—C(2)	1.794 (5)
C(11)—Au—C(21)	90.5 (2)	C(11)—Au—C(31)	89.8 (2)
C(21)—Au—C(31)	176.4 (2)	C(11)—Au—C(1)	176.5 (2)
C(21)—Au—C(1)	91.6 (2)	C(31)—Au—C(1)	87.9 (2)
C(41)—P—C(51)	106.9 (2)	C(41)—P—C(1)	112.0 (2)
C(51)—P—C(1)	111.0 (3)	C(41)—P—C(2)	108.8 (3)
C(51)—P—C(2)	107.9 (2)	C(1)—P—C(2)	110.1 (3)
Au—C(11)—C(12)	119.4 (5)	Au—C(11)—C(16)	125.0 (4)
C(12)—C(11)—C(16)	115.5 (5)	Au—C(21)—C(26)	123.5 (4)
Au—C(21)—C(22)	120.9 (5)	Au—C(31)—C(36)	124.7 (4)
C(22)—C(21)—C(26)	115.6 (4)	Au—C(1)—P	115.3 (3)
Au—C(31)—C(32)	120.5 (3)		
C(32)—C(31)—C(36)	114.7 (4)		

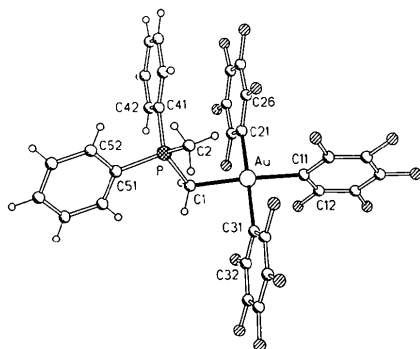


Fig. 1. The title compound in the crystal, showing the atom-numbering scheme. Radii are arbitrary.

(Usón, Laguna, Laguna, Gimeno, de Pablo, Jones, Meyer-Bäse & Freire Erdbrügger, 1987), 2.149 (13) Å in $(\text{PPh}_3\text{CH}_2)\text{AuMe}_3$ (Stein, Fackler, Papparizos & Chen, 1981) and 2.079, 2.099 (13) Å in $[(\text{Ph}_3\text{PCH}_2)_2\text{Au}.\text{AgClO}_4]_2$ (Usón, Laguna, Laguna, Usón, Jones & Freire Erdbrügger, 1987).

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Structure of *trans*-Tetraaquabis(trimethylammonioacetato)cobalt(II) Dichloride Tetrahydrate*

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Abstract. A new cobalt(II) complex of trimethylammonioacetate (betaine, BET) has been prepared

* Metal–Betaine Interactions. XVI. Part III: Chen & Mak (1991a).

The shortest nonbonded contact between non-H atoms is F(15)⋯F(34) ($2-x, 1-y, 1-z$) 2.91 Å.

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References

- JONES, P. G. (1988). *J. Organomet. Chem.* **345**, 405–411.
LAGUNA, A. & LAGUNA, M. (1990). *J. Organomet. Chem.* **394**, 743–756.
MURRAY, H. H., FACKLER, J. P., PORTER, L. C., BRIGGS, D. A., GUERRA, M. A. & LAGOW, R. J. (1987). *Inorg. Chem.* **26**, 357–363.
SHELDRICK, G. M. (1987). *SHELXTL-Plus*. Release 4.1 for Siemens R3m/V crystallographic system. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.
STEIN, J., FACKLER, J. P., PAPPARIZOS, C. & CHEN, H.-W. (1981). *J. Am. Chem. Soc.* **103**, 2192–2198.
USÓN, R. (1986). *Coord. Chem. Rev.* **70**, 1–50.
USÓN, R., LAGUNA, A., LAGUNA, M., CASTILLA, M. L., JONES, P. G. & FITTSCHEN, C. (1987). *J. Chem. Soc. Dalton Trans.* pp. 3017–3022.
USÓN, R., LAGUNA, A., LAGUNA, M., FERNANDEZ, E., JONES, P. G. & SHELDRICK, G. M. (1982). *J. Chem. Soc. Dalton Trans.*, pp. 1971–1976.
USÓN, R., LAGUNA, A., LAGUNA, M., FERNANDEZ, E., VILLACAMPA, M. D., JONES, P. G. & SHELDRICK, G. M. (1983). *J. Chem. Soc. Dalton Trans.* pp. 1679–1685.
USÓN, R., LAGUNA, A., LAGUNA, M., GIMENO, M. C., DE PABLO, A., JONES, P. G., MEYER-BÄSE, K. & FREIRE ERDBRÜGGER, C. (1987). *J. Organomet. Chem.* **336**, 461–468.
USÓN, R., LAGUNA, A., LAGUNA, M. & USÓN, A. (1983). *Inorg. Chim. Acta*, **73**, 63–66.
USÓN, R., LAGUNA, A., LAGUNA, M., USÓN, A. & GIMENO, M. C. (1987). *Organometallics*, **6**, 682–683.
USÓN, R., LAGUNA, A., LAGUNA, M., USÓN, A., JONES, P. G. & FREIRE ERDBRÜGGER, C. (1987). *Organometallics*, **6**, 1778–1880.