Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

	x	y	z	U_{eq}*
Rh	2500 (1)	1877 (1)	1122 (1)	30 (1)
N(1)	1610 (3)	2206 (3)	1182 (2)	32 (2)
C(1)	1028 (4)	2021 (4)	1126 (3)	44 (4)
C(2)	864 (5)	1406 (5)	953 (3)	56 (4)
C(3)	655 (6)	2500	1250	53 (5)
C(4)	3336 (5)	1617 (4)	1060 (3)	39 (3)
N(2)	3843 (4)	1480 (4)	996 (3)	47 (3)
C(5)	4456 (4)	1255 (5)	891 (4)	54 (4)
C(6)	4623 (4)	750 (5)	1198 (4)	72 (5)
C(7)	4901 (5)	1776 (5)	923 (5)	97 (6)
C(8)	4427 (6)	1027 (6)	422 (4)	82 (5)
P(1)	2331 (1)	3515 (1)	670 (1)	34 (1)
C(9)	1745 (4)	4107 (4)	703 (3)	30 (3)
C(10)	1212 (5)	4102 (5)	457 (3)	48 (4)
C(11)	791 (5)	4568 (6)	507 (4)	65 (5)
C(12)	894 (6)	5048 (6)	790 (4)	66 (5)
C(13)	1432 (5)	5060 (6)	1028 (3)	63 (4)
C(14)	1857 (5)	4593 (4)	989 (3)	45 (4)
C(15)	2910 (5)	3891 (4)	333 (3)	45 (4)
C(16)	3343 (5)	4268 (5)	535 (4)	53 (4)
C(17)	3779 (6)	4553 (6)	289 (6)	84 (6)
C(18)	3793 (7)	4514 (6)	−151 (6)	92 (7)
C(19)	3362 (7)	4141 (6)	−364 (5)	82 (6)
C(20)	2924 (5)	3852 (5)	−119 (4)	58 (4)
C(21)	1998 (4)	2901 (4)	342 (3)	37 (3)
P(2)	2439 (1)	2189 (1)	408 (1)	35 (1)
C(22)	3122 (5)	2320 (4)	88 (3)	50 (4)
C(23)	3627 (5)	2599 (5)	277 (4)	53 (4)
C(24)	4129 (6)	2749 (5)	17 (5)	75 (5)
C(25)	4140 (7)	2647 (6)	−419 (5)	91 (7)
C(26)	3637 (8)	2375 (7)	−604 (4)	101 (7)
C(27)	3123 (6)	2201 (5)	−361 (3)	69 (5)
C(28)	2010 (5)	1608 (4)	112 (3)	45 (4)
C(29)	1495 (6)	1728 (5)	−138 (3)	65 (4)
C(30)	1168 (7)	1261 (6)	−319 (4)	93 (6)
C(31)	1367 (8)	670 (7)	−266 (5)	116 (8)
C(32)	1892 (7)	528 (5)	−35 (5)	106 (7)
C(33)	2199 (6)	1002 (5)	152 (4)	72 (5)
P(3)	633 (2)	1719 (1)	3233 (1)	59 (1)
F(1)	738 (4)	2392 (3)	3412 (2)	104 (3)
F(2)	526 (3)	1054 (3)	3060 (3)	94 (3)
F(3)	712 (4)	1447 (4)	3698 (3)	125 (4)
F(4)	558 (4)	1983 (3)	2759 (2)	101 (3)
F(5)	−79 (3)	1788 (4)	3317 (3)	109 (4)
F(6)	1338 (3)	1655 (5)	3140 (3)	122 (4)

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

Table 2. *Selected bond distances* (Å) *and angles* (°)

Rh—N(1)	2.078 (6)	Rh—C(4)	1.920 (10)
Rh—P(2)	2.298 (2)	C(4)—N(2)	1.161 (13)
N(2)—C(5)	1.462 (13)	P(1)—C(21)	1.825 (9)
C(21)—P(2)	1.838 (9)		
N(1)—Rh—C(4)	177.0 (3)	N(1)—Rh—P(2)	85.9 (2)
C(4)—Rh—P(2)	92.7 (3)	Rh—N(1)—C(1)	140.2 (6)
N(1)—C(1)—C(2)	122.9 (8)	Rh—C(4)—N(2)	175.5 (8)
C(4)—N(2)—C(5)	174.1 (10)	P(1)—C(21)—P(2)	110.5 (5)
Rh—P(2)—C(21)	112.7 (3)		

dicarbonyl complex is 3.060 Å compared to 2.828 (1) Å for the title complex. The shorter distance for the title complex is consistent with an Rh—Rh bond order of $\frac{1}{2}$ for the Rh_2^{3+} core. The increase in bond order was predicted from the electronic model developed by Hoffman and co-workers (Hoffmann & Hoffman, 1981) for *A*-frame complexes since oxidation of the parent Rh^I—Rh^I complex results in removal of an electron from an Rh—Rh antibonding orbital. The small bite size of the pyrazolate ligands results in a dihedral angle of 38.4° between the two least-squares planes formed by the square-plane core atoms of the two Rh atoms.

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μ-[Bis(diphenylphosphino)methane]-bis[pentafluorophenylgold(I)] at 178 K

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Abstract. $[Au_2(C_{25}H_{22}P_2)(C_6F_5)_2]$, $M_r = 1112.4$, monoclinic, $P2_1/c$, $a = 14.141$ (4), $b = 20.209$ (5), $c = 14.207$ (4) Å, $\beta = 95.85$ (3)°, $V = 4039$ (2) Å^3, $Z = 4$, $D_x = 1.829$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 7.4$ mm^{-1}, $F(000) = 2088$, $T = 178$ K, $R = 0.046$ for 5181 reflections. The molecule crystallizes in a *cis*

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^4$)

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

	x	y	z	U_{eq}
Au(1)	5601.3 (3)	2063.8 (2)	7220.7 (3)	313 (2)
C(11)	6881 (8)	2561 (6)	7408 (8)	315 (28)
C(12)	6928 (9)	3250 (6)	7426 (8)	312 (28)
C(13)	7767 (8)	3592 (6)	7547 (8)	312 (28)
C(14)	8624 (9)	3242 (6)	7625 (9)	380 (31)
C(15)	8592 (10)	2567 (7)	7616 (9)	422 (33)
C(16)	7741 (9)	2248 (6)	7531 (8)	334 (29)
F(12)	6123 (6)	3615 (4)	7317 (6)	534 (30)
F(13)	7798 (6)	4251 (4)	7579 (6)	625 (33)
F(14)	9465 (5)	3569 (4)	7698 (7)	700 (36)
F(15)	9419 (6)	2219 (5)	7707 (7)	767 (40)
F(16)	7762 (6)	1576 (4)	7543 (6)	612 (33)
Au(2)	5641.9 (3)	1821.2 (3)	5025.8 (3)	306 (2)
C(21)	6965 (8)	2244 (6)	5022 (8)	294 (28)
C(22)	7112 (9)	2912 (6)	5108 (8)	350 (29)
C(23)	8006 (9)	3210 (6)	5166 (9)	363 (30)
C(24)	8790 (10)	2819 (7)	5098 (9)	411 (33)
C(25)	8669 (10)	2153 (7)	4972 (9)	427 (33)
C(26)	7768 (8)	1895 (6)	4941 (8)	301 (28)
F(22)	6361 (5)	3337 (4)	5219 (5)	470 (27)
F(23)	8113 (6)	3872 (4)	5252 (6)	583 (32)
F(24)	9643 (5)	3097 (4)	5144 (7)	654 (35)
F(25)	9423 (5)	1762 (4)	4904 (7)	677 (37)
F(26)	7701 (6)	1225 (4)	4831 (6)	537 (30)
P(1)	4165 (2)	1533 (2)	7100 (2)	290 (10)
C(1)	3973 (8)	941 (6)	6119 (7)	281 (27)
C(112)	3272 (5)	2467 (4)	4629 (6)	374 (31)
C(113)	2478	2860	4366	449 (34)
C(114)	1602	2566	4086	498 (36)
C(115)	1519	1879	4068	570 (40)
C(116)	2313	1485	4331	420 (33)
C(111)	3189	1779	4612	310 (28)
C(122)	4516 (5)	701 (3)	3298 (5)	366 (30)
C(123)	4357	242	2565	420 (33)
C(124)	3815	−323	2687	387 (31)
C(125)	3433	−429	3542	342 (29)
C(126)	3592	30	4275	361 (30)
C(121)	4134	595	4153	276 (26)
P(2)	4220 (2)	1286 (2)	4980 (2)	275 (10)
C(212)	3363 (5)	2781 (4)	7052 (6)	382 (32)
C(213)	2620	3236	6923	532 (38)
C(214)	1692	3017	6679	565 (40)
C(215)	1507	2342	6564	443 (34)
C(216)	2250	1887	6693	371 (30)
C(211)	3178	2106	6937	336 (29)
C(222)	4560 (5)	1190 (4)	8972 (6)	330 (29)
C(223)	4425	851	9804	471 (36)
C(224)	3719	369	9801	521 (38)
C(225)	3148	225	8966	537 (39)
C(226)	3284	563	8135	392 (32)
C(221)	3990	1046	8138	279 (27)

configuration with a short intramolecular Au···Au contact of 3.163 (1) Å. The linear coordination at Au is scarcely distorted: P—Au—C 176.6, 175.8 (4)°. The *ipso* angles of the C_6F_5 rings are narrow: 114.8, 113.6 (11)°.

Experimental. Single crystals were obtained by diffusion of petrol ether into a solution of the title compound in dichloromethane. A colourless prism 0.6 × 0.25 × 0.2 mm was mounted on a glass fibre with inert oil and transferred to the cold gas stream of a Siemens *R3m/V* diffractometer with LT-2 low-temperature attachment. Intensities were measured to $2\theta_{max}$ 50° using graphite-monochromated Mo $K\alpha$ radiation; ω-scan mode, rate 7.32° min^{-1}, range 1.10°. Of 8826 measured reflections, 7078 were unique ($R_{int} = 0.019$, index ranges h −16 to 16, k 0 to 24, l 0 to 16) and 5181 with $F > 4\sigma(F)$ were considered observed. The cell constants were refined from setting angles of 50 reflections in the 2θ range 20–23°. Three check reflections measured every 147 reflections showed no significant intensity variation. An absorption correction based on ψ scans was applied, with transmission factors 0.60–0.78.

The structure was solved by the heavy-atom method and subjected to full-matrix least-squares refinement on F; $\sum w(F_o - F_c)^2$ minimized. Au, P and F were refined anisotropically, phenyl (but not pentafluorophenyl) groups with idealized geometry. H atoms were included in the refinement using a riding model and with fixed isotropic U values. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.0003F^2$; final $R = 0.046$, with $wR = 0.060$; 227 parameters (data to parameter ratio 22.8:1); $S = 1.9$; maximum $\Delta/\sigma = 0.001$; maximum, minimum $\Delta\rho = 2.9$, -1.1 e $Å^{-3}$. The largest difference peaks occurred in 'holes' in the structure and may correspond to small regions of highly disordered solvent, but no attempt was made to refine this. Atomic scattering factors and f', f'' values were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The program system used was Siemens *SHELXTL-Plus* (Sheldrick, 1989) on a MicroVAX II computer. Final atomic coordinates are given in Table 1, with derived bond

Table 2. *Selected interatomic distances* (Å) *and angles* (°)

Au(1)—C(11)	2.063 (12)	Au(1)···Au(2)	3.163 (1)
Au(1)—P(1)	2.288 (3)	Au(2)—C(21)	2.058 (12)
Au(2)—P(2)	2.279 (3)	P(1)—C(1)	1.834 (11)
P(1)—C(211)	1.811 (8)	P(1)—C(211)	1.811 (8)
C(1)—P(2)	1.828 (11)	C(111)—P(2)	1.799 (7)
C(121)—P(2)	1.821 (7)		

C(11)—Au(1)—P(1)	176.6 (3)	C(12)—C(11)—C(16)	114.8 (11)
C(21)—Au(2)—P(2)	175.8 (3)	C(22)—C(21)—C(26)	113.6 (11)
Au(1)—P(1)—C(1)	115.1 (4)	Au(1)—P(1)—C(211)	112.1 (3)
C(1)—P(1)—C(211)	105.5 (4)	Au(1)—P(1)—C(221)	112.8 (3)
C(1)—P(1)—C(221)	104.0 (4)	C(211)—P(1)—C(221)	106.5 (4)
P(1)—C(1)—P(2)	113.4 (6)	Au(2)—P(2)—C(1)	113.8 (4)
Au(2)—P(2)—C(111)	115.6 (3)	C(1)—P(2)—C(111)	104.2 (5)
Au(2)—P(2)—C(121)	112.5 (3)	C(1)—P(2)—C(121)	105.9 (4)
C(111)—P(2)—C(121)	103.8 (4)		

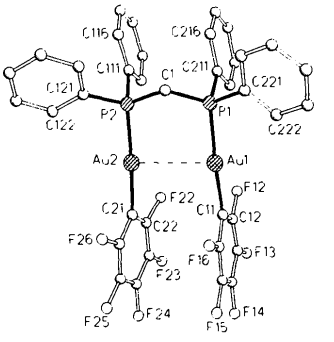

Fig. 1. The title compound in the crystal, showing the atom-numbering scheme. Radii are arbitrary.

lengths and angles in Table 2.* The atom-numbering is shown in Fig. 1.

Related literature. The fortuitous preparation of the title compound is described by Thöne (1991). Narrow *ipso* angles in C_6F_5 groups are discussed by Jones (1988). Some Au—C_6F_5 bond lengths are presented by Jones (1992). The tendency of Au atoms in Au^I derivatives to aggregate has been termed 'aurophilicity' by Schmidbaur (1990). A non-parallel arrangement of Au—Cl groups, leading to intermolecular Au···Au contacts, is observed in (dppm)$(AuCl)_2$ (Schmidbaur, Wohlleben, Wagner, Orama & Huttner, 1977), whereas a *cis* arrangement similar to that of the title compound is seen in (dppm)$(AuSiMe_3)_2$ (Piana, Wagner & Schubert, 1991).

* Lists of structure factors, anisotropic thermal parameters, complete bond distances and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54937 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0092]

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Bis[1,1-di(2-pyridyl)ethyl methyl ether-*N,N',O*]copper(II) Di-μ-iodo-bis[iodoargentate(I)]

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Abstract. $[Cu(C_{13}H_{14}N_2O)_2][Ag_2I_4]$, $M_r = 1215.4$, triclinic, $P\bar{1}$, $a = 8.5402$ (14), $b = 10.0286$ (16), $c = 10.584$ (2) Å, $\alpha = 110.221$ (10), $\beta = 95.476$ (10), $\gamma = 93.420$ (12)°, $V = 842.6$ (2) Å^3, $Z = 1$, $D_x = 2.395$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 5.424$ mm^{-1}, $F(000) = 563$, $T = 293$ K, $R = 0.034$ for 3381 reflections. The Cu atom of the cation lies on a centre of symmetry and is octahedrally coordinated by ligand donor atoms N (at 2.020, 2.000 Å) and O (at 2.393 Å). The centrosymmetric anion displays Ag—I (bridging) 2.780, 2.800, (terminal) 2.679 Å.

Experimental. A pale brown tablet 0.6 × 0.35 × 0.12 mm was mounted in a glass capillary. Using a Stoe four-circle diffractometer, 4493 intensities were registered to $2\theta_{max} = 55°$ with graphite-monochromated Mo $K\alpha$ radiation; 2θ–θ scan mode. Of 3839 unique reflections ($R_{int} = 0.021$, index ranges h −11 to 11, k −13 to 12, l 0 to 13) 3381 with $F > 4\sigma(F)$ were considered observed. The cell constants were refined from $\pm\omega$ angles of 46 reflections in the 2θ range 20–23°. Three check reflections measured every 100 reflections showed no significant intensity variation. An absorption correction based on ψ scans was applied, with transmission factors 0.53–0.91.

The structure was solved by the heavy-atom method and subjected to anisotropic full-matrix least-squares refinement on F; $\sum w(F_o - F_c)^2$ minimized. H atoms were included in the refinement using a riding model and with fixed isotropic U. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.0002F^2$; final $R = 0.034$, with $wR = 0.048$ (for all data $R = 0.039$, $wR = 0.053$); 184 parameters (data to parameter ratio 18.4:1); $S = 2.4$; maximum $\Delta/\sigma = 0.01$; maximum, minimum $\Delta\rho = 1.53$, -1.43 e Å^{-3}. Atomic scattering factors and f',f'' values were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The program system used was Siemens *SHELXTL-Plus* (Sheldrick, 1989) on a MicroVAX

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