SHORT-FORMAT PAPERS

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Acta Cryst. (1992). C**48**, 1485–1487

[Bis(diphenylphosphino)methane-*P*](*N*,*N*-diethyldithiocarbamato-*S*,*S*′)-(η^5-pentamethylcyclopentadienyl)rhodium(III) Tetraphenylborate

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Abstract. $[Rh(C_5H_{10}NS_2)(C_{10}H_{15})(C_{25}H_{22}P_2)][C_{24}H_{20}B]$, $M_r = 1090$, triclinic, $P\bar{1}$, $a = 10.428$ (4), $b = 15.452$ (8), $c = 19.416$ (9) Å, $\alpha = 107.86$ (4), $\beta = 90.41$ (4), $\gamma = 105.92$ (4)°, $V = 2849$ Å^3, $Z = 2$, $D_x = 1.271$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.427$ mm^{-1}, $F(000) = 1144$, $T = 298$ K, $R = 0.0638$ for 6381 unique observed reflections. In contrast to the analogous complex with the bis(diphenylphosphino)ethane ligand, where the major product is a centrosymmetric complex in which the ligand bridges two metal centres, here the diphosphine is monodentate to one centre with the second P donor 'dangling' and therefore available for coordination to another metal centre.

Experimental. The title compound was prepared by reaction of equimolar quantities of $[(C_5Me_5)Rh(S_2CNEt_2)Cl]$ and bis(diphenylphosphino)methane in $CDCl_3$, followed by addition of $NaBPh_4$ in MeOH. Crystals were obtained by recrystallization from $CDCl_3$/MeOH. An orange lamellar crystal, 1.08 × 0.92 × 0.23 mm, was mounted on a Stoe Stadi-4 four-circle diffractometer, equipped with graphite-monochromated Mo $K\alpha$ radiation. Cell parameters were determined from setting angles of 13 reflections ($27 \le 2\theta \le 28°$). For data collection, $T = 298$ K, ω–2θ scans with ω-scan width $(0.80 + 0.35\tan\theta)°$, $2\theta_{max} = 45°$, h −11→11, k −16→16, l 0→20. Three standard reflections showed less than ±2% variation. 7418 unique reflections were collected, giving 6381 with $F \ge 6\sigma(F)$ for structure solution [from a Patterson synthesis (Rh) followed by iterative cycles of least-squares refinement and difference Fourier synthesis] and refinement [using full-matrix least squares on F (*SHELX*76; Sheldrick, 1976)]. At isotropic convergence, corrections (min. 0.848, max. 1.113) for absorption were applied empirically using *DIFABS* (Walker & Stuart, 1983). Anisotropic thermal parameters for Rh, S and P atoms, H atoms treated as part of rigid methyl groups in C_5Me_5 or in fixed calculated positions. The ordered phenyl rings were treated as idealized planar hexagons, as was the principal disorder component for the C(31)–C(36) phenyl ring. The disorder was modelled in terms of one complete and two partial rings (identified by unprimed, singly primed and doubly primed atoms in Table 1) in which the partially occupied C atoms had fixed U_{iso} values of 0.08 Å^2. At final convergence, $R = 0.0638$, $wR = 0.0855$, $S = 1.322$ for 247 parameters, $(\Delta/\sigma)_{max}$ in final cycle 0.06, maximum and minimum residues in final ΔF synthesis 0.97, −0.81 e Å^{-3} respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0.000023F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for Rh (Cromer & Mann, 1968). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, while selected bond lengths and angles appear in Table 2.† The atom-numbering scheme for the molecule is shown in Fig. 1, which was generated using the version of *ORTEP* included in the *GX* crystallographic program system (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Related literature. With bis(diphenylphosphino)-ethane as the diphosphine ligand the major product is a centrosymmetric complex in which the ligand

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† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55014 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å²)

$U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}/U_{iso}
Rh	0.20277 (5)	0.24465 (3)	0.163980 (20)	0.0409 (3)
P(1)	0.15892 (16)	0.32693 (11)	0.27839 (8)	0.0444 (10)
P(2)	0.08568 (19)	0.32190 (14)	0.43312 (10)	0.0604 (13)
S(1)	0.33443 (16)	0.17612 (12)	0.21898 (9)	0.0543 (11)
S(2)	0.05924 (17)	0.09783 (12)	0.16779 (9)	0.0548 (11)
N(1)	0.1910 (6)	0.0052 (4)	0.2257 (3)	0.060 (4)
C(11)	0.0152 (4)	0.3713 (3)	0.27975 (25)	0.0496 (16)
C(12)	0.0069 (4)	0.4565 (3)	0.32907 (25)	0.0699 (21)
C(13)	−0.1106 (4)	0.4829 (3)	0.32817 (25)	0.095 (3)
C(14)	−0.2198 (4)	0.4242 (3)	0.27794 (25)	0.107 (3)
C(15)	−0.2115 (4)	0.3390 (3)	0.22859 (25)	0.109 (3)
C(16)	−0.0940 (4)	0.3126 (3)	0.22951 (25)	0.0722 (21)
C(21)	0.3031 (4)	0.4258 (3)	0.32610 (24)	0.0487 (16)
C(22)	0.4031 (4)	0.4071 (3)	0.36236 (24)	0.0661 (20)
C(23)	0.5210 (4)	0.4792 (3)	0.39386 (24)	0.086 (3)
C(24)	0.5388 (4)	0.5700 (3)	0.38913 (24)	0.090 (3)
C(25)	0.4388 (4)	0.5886 (3)	0.35288 (24)	0.0758 (22)
C(26)	0.3209 (4)	0.5165 (3)	0.32136 (24)	0.0613 (18)
C(1)	0.1176 (6)	0.2565 (4)	0.3403 (3)	0.0466 (15)
C(31)	0.1120 (7)	0.2426 (5)	0.4816 (4)	0.0735 (22)
C(32)	0.2381 (7)	0.2285 (5)	0.4844 (4)	0.0841 (24)
C(33)	0.2668 (7)	0.1776 (5)	0.5276 (4)	0.0800
C(34)	0.1693 (7)	0.1408 (5)	0.5682 (4)	0.0800
C(35)	0.0431 (7)	0.1550 (5)	0.5655 (4)	0.0800
C(36)	0.0144 (7)	0.2059 (5)	0.5222 (4)	0.0800
C(34′)	0.125 (4)	0.092 (3)	0.5400 (20)	0.0800
C(35′)	0.016 (3)	0.0866 (22)	0.5165 (17)	0.0800
C(36′)	0.001 (3)	0.1536 (23)	0.4843 (17)	0.0800
C(33″)	0.209 (6)	0.126 (4)	0.528 (3)	0.0800
C(34″)	0.205 (3)	0.179 (3)	0.5834 (16)	0.0800
C(35″)	0.0967 (24)	0.2065 (17)	0.5928 (12)	0.0800
C(36″)	0.0584 (24)	0.2508 (18)	0.5479 (13)	0.0800
C(41)	−0.0951 (5)	0.2963 (4)	0.4270 (3)	0.0627 (19)
C(42)	−0.1442 (5)	0.3651 (4)	0.4745 (3)	0.092 (3)
C(43)	−0.2821 (5)	0.3503 (4)	0.4762 (3)	0.119 (4)
C(44)	−0.3709 (5)	0.2667 (4)	0.4304 (3)	0.132 (4)
C(45)	−0.3219 (5)	0.1979 (4)	0.3828 (3)	0.169 (6)
C(46)	−0.1840 (5)	0.2127 (4)	0.3812 (3)	0.122 (4)
C(1N)	0.1940 (6)	0.0801 (4)	0.2067 (3)	0.0446 (15)
C(11*D*)	0.3109 (8)	−0.0012 (6)	0.2625 (4)	0.0719 (21)
C(12*D*)	0.3227 (9)	0.0419 (7)	0.3426 (5)	0.093 (3)
C(21*D*)	0.0663 (8)	−0.0731 (6)	0.2139 (4)	0.0763 (22)
C(22*D*)	−0.0178 (10)	−0.0647 (7)	0.2750 (5)	0.096 (3)
C(1*R*)	0.3179 (6)	0.3514 (4)	0.1164 (4)	0.0491 (16)
C(2*R*)	0.1780 (6)	0.3412 (4)	0.1039 (4)	0.0494 (16)
C(3*R*)	0.1149 (7)	0.2453 (5)	0.0613 (4)	0.0549 (17)
C(4*R*)	0.2140 (7)	0.1957 (5)	0.0457 (4)	0.0535 (17)
C(5*R*)	0.3408 (6)	0.2630 (4)	0.0787 (4)	0.0501 (16)
C(1*M*)	0.4266 (7)	0.4427 (5)	0.1517 (4)	0.0639 (19)
C(2*M*)	0.1174 (7)	0.4215 (5)	0.1245 (4)	0.0651 (19)
C(3*M*)	−0.0315 (8)	0.2016 (6)	0.0322 (4)	0.0758 (22)
C(4*M*)	0.1957 (8)	0.0957 (5)	−0.0019 (4)	0.0717 (21)
C(5*M*)	0.4747 (8)	0.2440 (5)	0.0736 (4)	0.0695 (21)
B	0.4785 (8)	0.7648 (6)	0.1689 (4)	0.0549 (20)
C(51)	0.3697 (4)	0.6819 (3)	0.10052 (20)	0.0486 (16)
C(52)	0.2414 (4)	0.6908 (3)	0.09226 (20)	0.0618 (19)
C(53)	0.1503 (4)	0.6263 (3)	0.03410 (20)	0.0677 (20)
C(54)	0.1874 (4)	0.5530 (3)	−0.01581 (20)	0.0633 (19)
C(55)	0.3158 (4)	0.5441 (3)	−0.00756 (20)	0.0634 (19)
C(56)	0.4069 (4)	0.6085 (3)	0.05061 (20)	0.0543 (17)
C(61)	0.6468 (4)	0.6923 (4)	0.22629 (22)	0.0652 (19)
C(62)	0.7609 (4)	0.6618 (4)	0.22687 (22)	0.0788 (23)
C(63)	0.8497 (4)	0.6698 (4)	0.17424 (22)	0.0724 (21)
C(64)	0.8245 (4)	0.7081 (4)	0.12102 (22)	0.0649 (19)
C(65)	0.7104 (4)	0.7386 (4)	0.12041 (22)	0.0553 (17)
C(66)	0.6216 (4)	0.7306 (4)	0.17305 (22)	0.0476 (15)
C(71)	0.4151 (5)	0.7784 (4)	0.24932 (22)	0.0573 (17)
C(72)	0.4893 (5)	0.8494 (4)	0.31080 (22)	0.0741 (22)
C(73)	0.4381 (5)	0.8628 (4)	0.37821 (22)	0.094 (3)
C(74)	0.3126 (5)	0.8051 (4)	0.38415 (22)	0.099 (3)
C(75)	0.2383 (5)	0.7340 (4)	0.32268 (22)	0.108 (3)
C(76)	0.2896 (5)	0.7207 (4)	0.25527 (22)	0.0799 (23)
C(81)	0.5258 (4)	0.87067 (24)	0.15277 (24)	0.0484 (16)
C(82)	0.4466 (4)	0.88953 (24)	0.10419 (24)	0.0560 (17)
C(83)	0.4837 (4)	0.97774 (24)	0.09314 (24)	0.0680 (20)
C(84)	0.6000 (4)	1.04710 (24)	0.13065 (24)	0.0751 (22)
C(85)	0.6792 (4)	1.02822 (24)	0.17922 (24)	0.0760 (22)
C(86)	0.6421 (4)	0.94002 (24)	0.19027 (24)	0.0640 (19)

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

Rh—P(1)	2.3154 (17)	N(1)—C(1N)	1.313 (9)
Rh—S(1)	2.3700 (18)	N(1)—C(11*D*)	1.477 (10)
Rh—S(2)	2.3737 (19)	N(1)—C(21*D*)	1.473 (11)
Rh—C(1*R*)	2.209 (7)	C(11*D*)—C(12*D*)	1.482 (13)
Rh—C(2*R*)	2.221 (7)	C(21*D*)—C(22*D*)	1.475 (14)
Rh—C(3*R*)	2.193 (7)	C(1*R*)—C(2*R*)	1.434 (10)
Rh—C(4*R*)	2.202 (7)	C(1*R*)—C(5*R*)	1.424 (10)
Rh—C(5*R*)	2.235 (7)	C(1*R*)—C(1*M*)	1.505 (10)
P(1)—C(11)	1.809 (5)	C(2*R*)—C(3*R*)	1.420 (10)
P(1)—C(21)	1.818 (5)	C(2*R*)—C(2*M*)	1.493 (10)
P(1)—C(1)	1.836 (7)	C(3*R*)—C(4*R*)	1.429 (10)
C(1)—P(2)	1.858 (7)	C(3*R*)—C(3*M*)	1.514 (11)
P(2)—C(31)	1.831 (8)	C(4*R*)—C(5*R*)	1.437 (10)
P(2)—C(41)	1.812 (6)	C(4*R*)—C(4*M*)	1.496 (11)
S(1)—C(1N)	1.729 (7)	C(5*R*)—C(5*M*)	1.501 (11)
S(2)—C(1N)	1.715 (7)		

P(1)—Rh—S(1)	89.24 (6)	C(1N)—N(1)—C(11*D*)	121.0 (6)
P(1)—Rh—S(2)	92.68 (6)	C(1N)—N(1)—C(21*D*)	120.8 (6)
S(1)—Rh—S(2)	73.22 (6)	C(11*D*)—N(1)—C(21*D*)	118.1 (6)
Rh—P(1)—C(11)	115.02 (17)	S(1)—C(1N)—S(2)	110.5 (4)
Rh—P(1)—C(21)	111.72 (15)	S(1)—C(1N)—N(1)	124.5 (5)
Rh—P(1)—C(1)	114.79 (22)	S(2)—C(1N)—N(1)	125.0 (5)
C(11)—P(1)—C(21)	108.31 (22)	N(1)—C(11*D*)—C(12*D*)	113.1 (7)
C(11)—P(1)—C(1)	101.6 (3)	N(1)—C(21*D*)—C(22*D*)	115.2 (7)
C(21)—P(1)—C(1)	104.4 (3)	C(2*R*)—C(1*R*)—C(5*R*)	108.2 (6)
P(1)—C(11)—C(12)	124.0 (4)	C(2*R*)—C(1*R*)—C(1*M*)	126.5 (6)
P(1)—C(11)—C(16)	115.9 (4)	C(5*R*)—C(1*R*)—C(1*M*)	124.6 (6)
P(1)—C(21)—C(22)	118.2 (3)	C(1*R*)—C(2*R*)—C(3*R*)	107.5 (6)
P(1)—C(21)—C(26)	121.5 (3)	C(1*R*)—C(2*R*)—C(2*M*)	124.5 (6)
P(1)—C(1)—P(2)	115.5 (4)	C(3*R*)—C(2*R*)—C(2*M*)	127.5 (6)
C(1)—P(2)—C(31)	99.5 (3)	C(2*R*)—C(3*R*)—C(4*R*)	109.0 (6)
C(1)—P(2)—C(41)	103.4 (3)	C(2*R*)—C(3*R*)—C(3*M*)	126.9 (7)
C(31)—P(2)—C(41)	101.6 (3)	C(4*R*)—C(3*R*)—C(3*M*)	124.0 (7)
P(2)—C(31)—C(32)	118.3 (6)	C(3*R*)—C(4*R*)—C(5*R*)	107.1 (6)
P(2)—C(31)—C(36)	121.3 (6)	C(3*R*)—C(4*R*)—C(4*M*)	127.8 (7)
P(2)—C(41)—C(42)	115.6 (4)	C(5*R*)—C(4*R*)—C(4*M*)	124.8 (6)
P(2)—C(41)—C(46)	124.4 (5)	C(1*R*)—C(5*R*)—C(4*R*)	108.1 (6)
Rh—S(1)—C(1N)	88.04 (23)	C(1*R*)—C(5*R*)—C(5*M*)	125.8 (6)
Rh—S(2)—C(1N)	88.24 (23)	C(4*R*)—C(5*R*)—C(5*M*)	126.1 (6)

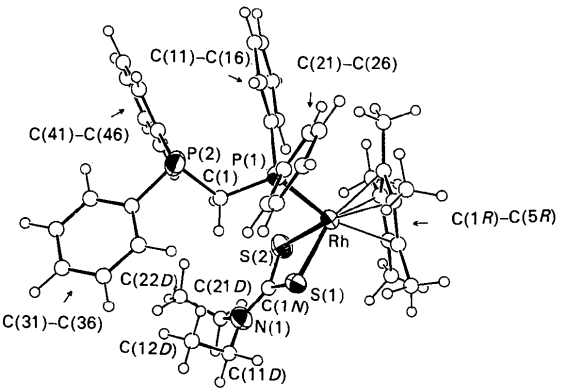

Fig. 1. A general view of the cation showing the atom-numbering scheme. Thermal ellipsoids are drawn at the 30% probability level, except those of C and H which have artificial radii of 0.15 and 0.10 Å, respectively, for clarity. The disorder affecting the ring C(31)–C(36) is not shown.

bridges two metal centres (Blake, Fotheringham, Stephenson, Hambling & Sawyer, 1991).

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Tetrakis(triphenylstibine)gold(I) Perchlorate

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Abstract. $[Au(C_{18}H_{15}Sb)_4]ClO_4$, $M_r = 1708.6$, rhombohedral, $R\bar{3}$, $a = 14.5860$ (8), $c = 52.351$ (4) Å (hexagonal axes), $V = 9646$ Å^3, $Z = 6$, $D_x = 1.765$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 4.0$ mm^{-1}, $F(000) = 4944$, $T = 293$ K, $R = 0.038$ for 3404 reflections. The Au atom and one Sb atom lie on the crystallographic threefold axis $\frac{2}{3}, \frac{1}{3}, z$; the coordination at the Au is almost exactly tetrahedral, with Au—Sb bond lengths of 2.658 (2) and 2.656 (2) Å, and Sb—Au—Sb bond angles of 108.8 (1) and 110.1 (1)°. The Cl atoms of the two independent perchlorate anions lie on special positions $\frac{1}{3}, \frac{2}{3}, \frac{1}{6}$ and 0,0,0 respectively; the site symmetry $\bar{3}$ is impossible for ordered perchlorate, and the O atoms are severely disordered.

Experimental. A colourless prism $0.55 \times 0.3 \times 0.2$ mm was mounted in a glass capillary. Intensities were registered to $2\theta_{max} = 50°$ on a Stoe–Siemens four-circle diffractometer using monochromated Mo $K\alpha$ radiation; ω-scan technique. Of 7016 measured reflections, 3760 were unique ($R_{int} = 0.014$, index ranges h −17 to 8, k 0 to 17, l 0 to 62) and 3404 with $F > 4\sigma(F)$ were considered observed. The cell constants were refined from $\pm\omega$ angles of 60 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 0.62–0.85.

The structure was solved by the heavy-atom method and subjected to full-matrix least-squares refinement on F. The O atoms of the perchlorates were very badly resolved and are probably disordered over spheres with the Cl atoms as centres. Au, Sb and Cl atoms were refined anisotropically; idealized phenyl groups isotropically. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.00015F^2$; final $R = 0.038$, with $wR = 0.042$; 76 parameters; S 2.5; maximum $\Delta/\sigma = 0.1$ for the disordered O atoms, otherwise $\Delta/\sigma = 0.04$; maximum, minimum $\Delta\rho = 1.5$, -0.8 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). Final atom coordinates are given in Table 1,* with derived bond lengths and angles in

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

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

	x	y	z	U_{eq}
Au	6667	3333	5868.9 (1)	35 (1)
Sb(1)	6667	3333	5361.1 (1)	42 (1)
Sb(2)	5293.8 (3)	3895.0 (3)	6032.4 (1)	41 (1)
C(12)	8356 (4)	3650 (3)	4967 (1)	71 (2)
C(13)	9088	3445	4846	93 (3)
C(14)	9235	2623	4933	81 (2)
C(15)	8649	2006	5140	75 (2)
C(16)	7916	2210	5260	66 (2)
C(11)	7769	3032	5174	46 (1)
C(22)	4220 (3)	3878 (5)	6537 (1)	89 (3)
C(23)	4175	4066	6797	121 (4)
C(24)	5083	4450	6947	117 (4)
C(25)	6037	4647	6838	96 (3)
C(26)	6082	4459	6578	73 (2)
C(21)	5174	4075	6428	57 (2)
C(32)	3381 (4)	1688 (4)	5965 (1)	77 (2)
C(33)	2395	919	5873	96 (3)
C(34)	1739	1222	5750	81 (2)
C(35)	2070	2293	5719	75 (2)
C(36)	3056	3061	5811	60 (2)
C(31)	3712	2759	5934	47 (1)
C(42)	5708 (4)	5659 (4)	5653 (1)	66 (2)
C(43)	5667	6524	5553	79 (2)
C(44)	5239	7022	5698	70 (2)
C(45)	4852	6654	5943	78 (2)
C(46)	4893	5789	6043	56 (2)
C(41)	5321	5291	5898	43 (1)
Cl(1)	3333	6667	1667	77 (2)
Cl(2)	0	0	0	98 (2)
O(1)	4293 (14)	7169 (16)	1516 (3)	210 (7)
O(2)	9242 (20)	9213 (18)	174 (4)	303 (13)

* 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 55012 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0090]

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