of organic and inorganic entities of the *ABABA*... type of packing. Intermolecular interactions are observed through short S···S (≤ 3·60 Å) and S···O (≤ 3·20 Å) contacts (see Fig. 2).

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$[(C_5Me_5)(Et_2NCS_2)Rh(\mu\text{-}Ph_2PCH_2CH_2PPh_2)Rh(Et_2NCS_2)(C_5Me_5)]^{2+}.2BPh_4^-$

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(*Received* 24 *April* 1990; *accepted* 28 *August* 1990)

Abstract. [μ-Ethylenebis(diphenylphosphine)-1κP:-2κP]-bis[(*N*,*N*-diethyldithiocarbamato)(η^5-pentamethylcyclopentadienyl)rhodium(III)] bis(tetraphenylborate), $[Rh_2(C_{10}H_{15})_2(C_5H_{10}NS_2)_2(C_{26}H_{24}P_2)]^{2+}.2C_{24}H_{20}B^-$, $M_r = 1809{\cdot}7$, orthorhombic, *Pbca*, $a = 28{\cdot}497\ (9)$, $b = 19{\cdot}236\ (8)$, $c = 16{\cdot}700\ (9)$ Å, $V = 9154$ Å^3, $Z = 4$, $D_x = 1{\cdot}31$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}492$ mm^{-1}, $F(000) = 3784$, $T = 295$ K, $R = 0{\cdot}0598$ for 5198 unique observed reflections. The dication is centrosymmetric, the two $[C_5Me_5Rh(S_2CNEt_2)]$ units being linked by a Ph_2PCH_2—CH_2PPh_2 ligand whose midpoint lies on a crystallographic inversion centre.

Experimental. Title compound prepared by reaction of $[C_5Me_5Rh(S_2CNEt_2)Cl]$ with $Ph_2PCH_2CH_2PPh_2$ (dppe) in CH_2Cl_2 followed by the addition of $NaBPh_4$ in MeOH, crystals obtained by crystallization from MeOH. Orange sphenoid, 0·2 × 0·3 × 1·4 mm, mounted to rotate about **c** on Stoe STADI-2 two-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell parameters from nine *hk*0 and four 00*l* reflections. For data collection, ω scans with ω-scan width $(1{\cdot}0 + 0{\cdot}50\tan\theta)°$, $2\theta_{max} = 50°$, h 0→32, k 0→22, l 0→19, no significant crystal movement or decay, 8127 reflections collected, 7936 unique ($R_{int} = 0{\cdot}039$), giving 5198 with $F \geq 6\sigma(F)$ for

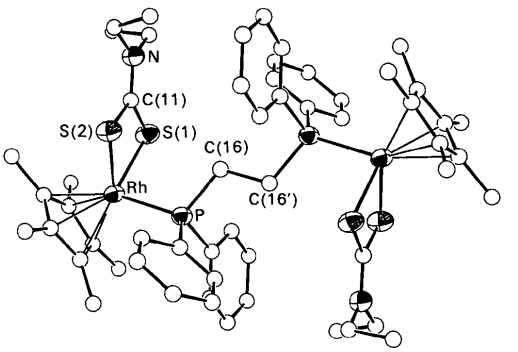

Fig. 1. A general view of the dication showing atom-numbering scheme: H atoms have been removed for clarity. Thermal ellipsoids are drawn at the 30% probability level, except those of C atoms which have artificial radii of 0·15 Å. The dication is centrosymmetric; the midpoint of the CH_2—CH_2 bond in the dppe ligand lies on an inversion centre.

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0108-2701/91/030648-03$03.00

Table 1. *Atomic coordinates and isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq} or U_{iso}
Rh	0·05308 (2)	0·18832 (2)	0·08300 (3)	0·0368 (3)
S(1)	−0·02598 (6)	0·18018 (9)	0·12175 (10)	0·0466 (10)
S(2)	0·00819 (6)	0·21574 (10)	−0·03176 (9)	0·0490 (10)
N	−0·08274 (18)	0·2304 (3)	0·0075 (3)	0·051 (4)
P	0·05552 (6)	0·07250 (8)	0·04892 (9)	0·0356 (8)
C(1)	0·12733 (22)	0·2041 (3)	0·1142 (4)	0·0427 (16)
C(2)	0·09923 (23)	0·2048 (3)	0·1853 (4)	0·0466 (17)
C(3)	0·0698 (3)	0·2633 (4)	0·1806 (4)	0·0593 (20)
C(4)	0·0771 (3)	0·2952 (4)	0·1061 (4)	0·0563 (19)
C(5)	0·11250 (24)	0·2587 (4)	0·0649 (4)	0·0485 (17)
C(6)	0·1680 (3)	0·1573 (4)	0·0998 (4)	0·0606 (20)
C(7)	0·1065 (3)	0·1607 (4)	0·2579 (5)	0·0709 (22)
C(8)	0·0358 (3)	0·2853 (5)	0·2455 (5)	0·083 (3)
C(9)	0·0534 (3)	0·3611 (4)	0·0763 (5)	0·086 (3)
C(10)	0·1337 (3)	0·2804 (4)	−0·0126 (5)	0·0733 (23)
C(11)	−0·04009 (22)	0·2111 (3)	0·0282 (4)	0·0437 (16)
C(12)	−0·1209 (3)	0·2291 (4)	0·0638 (4)	0·0631 (21)
C(13)	−0·1209 (3)	0·2950 (4)	0·1148 (5)	0·0654 (21)
C(14)	−0·0911 (3)	0·2625 (4)	−0·0719 (4)	0·0676 (22)
C(15)	−0·1032 (3)	0·2081 (4)	−0·1341 (5)	0·083 (3)
C(16)	0·00013 (22)	0·0397 (3)	0·0107 (4)	0·0435 (15)
C(17)	0·06911 (14)	0·01780 (25)	0·1336 (3)	0·0432 (16)
C(18)	0·11481 (14)	−0·00409 (25)	0·1498 (3)	0·0557 (18)
C(19)	0·12485 (14)	−0·03840 (25)	0·2214 (3)	0·0792 (25)
C(20)	0·08918 (14)	−0·05082 (25)	0·2768 (3)	0·089 (3)
C(21)	0·04349 (14)	−0·02893 (25)	0·2606 (3)	0·086 (3)
C(22)	0·03345 (14)	0·00538 (25)	0·1890 (3)	0·0659 (21)
C(24)	0·10825 (15)	0·10219 (17)	−0·0834 (3)	0·0514 (17)
C(25)	0·13860 (15)	0·08791 (17)	−0·1468 (3)	0·0670 (21)
C(26)	0·15760 (15)	0·02150 (17)	−0·1555 (3)	0·0723 (23)
C(27)	0·14624 (15)	−0·03063 (17)	−0·1008 (3)	0·0624 (20)
C(28)	0·11589 (15)	−0·01635 (17)	−0·0373 (3)	0·0543 (18)
C(23)	0·09689 (15)	0·05006 (17)	−0·0286 (3)	0·0416 (15)
B	0·3096 (3)	0·4524 (4)	0·5910 (4)	0·0453 (18)
C(29)	0·29070 (16)	0·41034 (17)	0·5099 (3)	0·0444 (16)
C(30)	0·28769 (16)	0·44581 (17)	0·4372 (3)	0·0571 (19)
C(31)	0·27108 (16)	0·41173 (17)	0·3691 (3)	0·0634 (20)
C(32)	0·25748 (16)	0·34218 (17)	0·3737 (3)	0·0603 (19)
C(33)	0·26049 (16)	0·30671 (17)	0·4464 (3)	0·0578 (19)
C(34)	0·27710 (16)	0·34079 (17)	0·5145 (3)	0·0474 (16)
C(35)	0·36838 (15)	0·44813 (24)	0·59386 (20)	0·0471 (16)
C(36)	0·39298 (15)	0·43665 (24)	0·66485 (20)	0·0575 (19)
C(37)	0·44180 (15)	0·43144 (24)	0·66362 (20)	0·0673 (21)
C(38)	0·46602 (15)	0·43772 (24)	0·59139 (20)	0·0709 (22)
C(39)	0·44142 (15)	0·44920 (24)	0·52039 (20)	0·0666 (21)
C(40)	0·39260 (15)	0·45441 (24)	0·52163 (20)	0·0512 (17)
C(41)	0·29284 (16)	0·53430 (24)	0·5937 (3)	0·0448 (16)
C(42)	0·24989 (16)	0·55342 (24)	0·5602 (3)	0·0670 (21)
C(43)	0·23200 (16)	0·61996 (24)	0·5733 (3)	0·079 (3)
C(44)	0·25705 (16)	0·66739 (24)	0·6198 (3)	0·0748 (23)
C(45)	0·30000 (16)	0·64827 (24)	0·6533 (3)	0·0663 (21)
C(46)	0·31790 (16)	0·58173 (24)	0·6402 (3)	0·0535 (18)
C(47)	0·28678 (14)	0·41465 (22)	0·6745 (3)	0·0419 (15)
C(48)	0·30488 (14)	0·35184 (22)	0·7026 (3)	0·0584 (19)
C(49)	0·28521 (14)	0·32021 (22)	0·7699 (3)	0·0704 (22)
C(50)	0·24744 (14)	0·35140 (22)	0·8090 (3)	0·0684 (21)
C(51)	0·22934 (14)	0·41421 (22)	0·7809 (3)	0·0606 (20)
C(52)	0·24901 (14)	0·44584 (22)	0·7137 (3)	0·0470 (16)

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

Rh—S(1)	2·3493 (18)	C(3)—C(8)	1·514 (12)
Rh—S(2)	2·3638 (18)	C(4)—C(5)	1·409 (10)
Rh—C(1)	2·200 (6)	C(4)—C(9)	1·519 (12)
Rh—C(2)	2·179 (6)	C(5)—C(10)	1·488 (11)
Rh—C(3)	2·228 (7)	C(11)—N	1·317 (8)
Rh—C(4)	2·201 (7)	N—C(12)	1·437 (9)
Rh—C(5)	2·189 (7)	N—C(14)	1·481 (10)
Rh—P	2·3005 (17)	C(12)—C(13)	1·528 (11)
S(1)—C(11)	1·720 (6)	C(14)—C(15)	1·514 (12)
S(2)—C(11)	1·704 (6)	P—C(16)	1·816 (6)
C(1)—C(2)	1·432 (9)	P—C(17)	1·805 (5)
C(1)—C(5)	1·400 (9)	P—C(23)	1·804 (4)
C(1)—C(6)	1·488 (10)	B—C(29)	1·667 (9)
C(2)—C(3)	1·404 (10)	B—C(35)	1·678 (9)
C(2)—C(7)	1·496 (10)	B—C(41)	1·648 (9)
C(3)—C(4)	1·403 (10)	B—C(47)	1·701 (9)
C(16)—C(16′)	1·570 (9)*		

S(1)—Rh—S(2)	73·69 (6)	N—C(12)—C(13)	110·5 (6)
S(1)—Rh—P	91·87 (6)	N—C(14)—C(15)	111·3 (6)
S(2)—Rh—P	91·83 (6)	Rh—P—C(16)	113·39 (21)
Rh—S(1)—C(11)	87·19 (22)	Rh—P—C(17)	112·16 (16)
Rh—S(2)—C(11)	87·07 (22)	Rh—P—C(23)	115·41 (15)
C(2)—C(1)—C(5)	108·1 (5)	C(16)—P—C(17)	105·0 (3)
C(2)—C(1)—C(6)	125·2 (6)	C(16)—P—C(23)	103·46 (24)
C(5)—C(1)—C(6)	126·5 (6)	C(17)—P—C(23)	106·43 (20)
C(1)—C(2)—C(3)	107·2 (6)	P—C(17)—C(18)	121·9 (3)
C(1)—C(2)—C(7)	126·1 (6)	P—C(17)—C(22)	117·6 (3)
C(3)—C(2)—C(7)	125·6 (6)	P—C(23)—C(24)	116·8 (3)
C(2)—C(3)—C(4)	108·1 (6)	P—C(23)—C(28)	123·2 (3)
C(2)—C(3)—C(8)	124·5 (7)	C(29)—B—C(35)	108·8 (5)
C(4)—C(3)—C(8)	127·3 (7)	C(29)—B—C(41)	113·1 (5)
C(3)—C(4)—C(5)	108·7 (6)	C(29)—B—C(47)	109·7 (5)
C(3)—C(4)—C(9)	126·2 (7)	C(35)—B—C(41)	109·5 (5)
C(5)—C(4)—C(9)	125·0 (7)	C(35)—B—C(47)	109·7 (5)
C(1)—C(5)—C(4)	107·7 (6)	C(41)—B—C(47)	105·9 (5)
C(1)—C(5)—C(10)	126·8 (6)	B—C(29)—C(30)	119·3 (4)
C(4)—C(5)—C(10)	125·2 (6)	B—C(29)—C(34)	120·6 (4)
S(1)—C(11)—S(2)	111·3 (4)	B—C(35)—C(36)	122·3 (4)
S(1)—C(11)—N	123·5 (5)	B—C(35)—C(40)	117·7 (4)
S(2)—C(11)—N	125·2 (5)	B—C(41)—C(42)	119·7 (4)
C(11)—N—C(12)	121·4 (6)	B—C(41)—C(46)	119·5 (4)
C(11)—N—C(14)	120·0 (6)	B—C(47)—C(48)	120·3 (4)
C(12)—N—C(14)	118·1 (6)	B—C(47)—C(52)	119·7 (4)
C(16)—P—C(16′)	115·0 (4)*		

* C(16′) is generated from C(16) by inversion through (0,0,0).

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). Anisotropic thermal parameters for Rh, S, P and N, H atoms in fixed, calculated positions or as part of rigid CH_3— groups. At final convergence, $R = 0{\cdot}0598$, $wR = 0{\cdot}0736$, $S = 1{\cdot}405$ for 219 parameters, $(\Delta/\sigma)_{max}$ in final cycle 0·19, max. and min. residues in final ΔF synthesis 0·79, −0·47 e Å^{-3}. The weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}000307F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (*SHELX*76; Sheldrick, 1976) except for Rh (Cromer & Mann, 1968).

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, selected bond lengths and angles appear in Table 2.* The atom-numbering scheme for the molecule shown in Fig. 1 was generated using *ORTEP* (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Related literature. ^{31}P NMR spectra indicate that the initial product of the reaction with dppe is similar to that with dppm ($Ph_2PCH_2PPh_2$) in that it affords a mono-rhodium product with a 'dangling' —PPh_2 group available for further coordination (Blake, Fotheringham & Stephenson, 1991). However,

* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53523 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

crystallization yields exclusively the dication with the bridging dppe as described here.

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Acta Cryst. (1991). C**47**, 650–651

A Vanadyl Complex of a Tetradentate Ligand Featuring a *cis*-N_2S_2 Donor Ligand Set

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(*Received* 16 *July* 1990; *accepted* 20 *August* 1990)

Abstract. [*N*,*N*′-Dimethyl-2,2′-(ethylenediimino-κ^2-*N*,*N*′)diethanethiolato-κ^2*S*,*S*′]oxovanadium(IV), [VO($C_8H_{18}N_2S_2$)], $M_r = 273{\cdot}3$, monoclinic, $P2_1/c$, $a = 7{\cdot}620$ (1), $b = 12{\cdot}702$ (2), $c = 12{\cdot}782$ (1) Å, $\beta = 103{\cdot}75$ (1)°, $V = 1202$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}510$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 1{\cdot}074$ mm^{-1}, $F(000) = 572$, $T = 293$ (1) K, $R = 0{\cdot}038$ for 1759 observed reflections. The V atom in [VO($C_8H_{18}N_2S_2$)] exists in a distorted square-pyramidal geometry with the square plane defined by a *cis*-N_2S_2 donor set provided by the dianion L^{2-} [where LH_2 is *N*,*N*′-dimethyl-2,2′-(ethylenediimino)diethanethiol]; V—N 2·151 (3), 2·164 (3); V—S 2·361 (1), 2·335 (1) Å. The V atom lies 0·6986 (5) Å out of the N_2S_2 plane in the direction of the apical O atom [V—O 1·596 (2) Å].

Experimental. [VO(acac)$_2$] (acac = acetylacetonate) and the free ligand, LH_2 (Wilson, Kony, Tiekink, Pilbrow, Spence & Wedd, 1988), were refluxed in methanol to produce a mixture of [V(acac)$_3$] and [VO($C_8H_{18}N_2S_2$)]. Recrystallization from hot methanol produced a low yield (6%) of well formed crystals of [VO($C_8H_{18}N_2S_2$)]. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\alpha$ radiation; ω:2θ scan technique. Cell parameters by least squares on 25 reflections ($2 \le \theta \le 11°$) (de Boer & Duisenberg, 1984) on a spherical crystal of 0·33 mm diameter; no absorption correction applied. 3447 reflections ($1{\cdot}5 \le \theta \le 27{\cdot}5°$) measured in the range $-9 \le h \le 9$, $0 \le k \le 16$, $-16 \le l \le 1$. No significant variation in the net intensities of three reflections ($\bar{2}\bar{1}0$, $0\bar{2}0$, $\bar{2}\bar{4}1$) measured every 7200 s. 2762 unique reflections (R_{int} 0·024) and 1759 satisfied $I \ge 2{\cdot}5\sigma(I)$. Structure solved by Patterson method, full-matrix least-squares refinement on 128 parameters based on *F* (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms included at their calculated positions. Evidence of disorder in the structure is seen in the high thermal motion associated with the C(4) and C(5) atoms and the short C(4)—C(5) bond distance of 1·262 (7) Å. At convergence $R = 0{\cdot}038$, $wR = 0{\cdot}046$, $w = 0{\cdot}57/[\sigma^2(F) + 0{\cdot}0015|F|^2]$, $S = 0{\cdot}96$, $(\Delta/\sigma)_{max} \le 0{\cdot}002$, $\Delta\rho_{max} = 0{\cdot}40$, $\Delta\rho_{min} = -0{\cdot}59$ e Å^{-3}; no extinction correction applied. Scat-

Table 1. *Atomic coordinates and B_{eq} values* (Å^2)

$B_{eq} = (8\pi^2/3)$ trace **U**.

	x	*y*	*z*	B_{eq}
V	0·23878 (7)	0·16363 (4)	0·18823 (4)	2·46
S(1)	0·3692 (2)	0·3332 (1)	0·2013 (1)	4·48
S(2)	−0·0256 (1)	0·2247 (1)	0·0717 (1)	4·83
O(1)	0·3542 (4)	0·0900 (2)	0·1286 (2)	4·39
N(1)	0·3575 (4)	0·1602 (2)	0·3587 (2)	3·03
N(2)	0·0589 (4)	0·0526 (2)	0·2379 (2)	3·79
C(1)	0·4460 (6)	0·3401 (3)	0·3453 (3)	4·57
C(2)	0·3692 (5)	0·2681 (3)	0·4053 (3)	3·62
C(3)	0·5427 (7)	0·1166 (4)	0·3807 (4)	5·84
C(4)	0·2406 (10)	0·0958 (5)	0·4110 (3)	9·29
C(5)	0·1194 (9)	0·0383 (8)	0·3544 (4)	12·36
C(6)	0·0639 (8)	−0·0507 (4)	0·1835 (6)	8·31
C(7)	−0·1287 (6)	0·0910 (4)	0·2114 (4)	5·63
C(8)	−0·1864 (6)	0·1296 (5)	0·0992 (4)	6·40

0108-2701/91/030650-02$03.00