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Structures of (Cyclooctadiene)Rh-μ(SPPh$_2$)$_2$-PtS$_2$CNEt$_2$ and (Cyclooctadiene)Ir-μ(SPPh$_2$)$_2$-PtS$_2$CN^{i}Pr$_2$

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Abstract. (1-2:5-6-η-1,5-Cyclooctadiene)[(*N*,*N*-diethyldithiocarbamato)bis(diphenylthiophosphinito-*P*,*P*′)platinum-*S*,*S*′]rhodium (I), [PtRh(C$_{12}$H$_{10}$PS)$_2$(C$_5$H$_{10}$NS$_2$)(C$_8$H$_{12}$)], M_r = 988·94, orthorhombic, *Pccn*, a = 22·533 (13), b = 22·817 (17), c = 14·690 (15) Å, V = 7553 Å^3, Z = 8, D_x = 1·739 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 44·93 mm^{-1}, F(000) = 3904, T = 293 K, R = 0·0486 for 2679 unique observed reflections. (1-2:5-6-η-1,5-Cyclooctadiene)[(*N*,*N*-diisopropyldithiocarbamato)bis(diphenylthiophosphinito-*P*,*P*′)platinum-*S*,*S*′]iridium (II), [PtIr(C$_{12}$H$_{10}$PS)$_2$(C$_7$H$_{14}$NS$_2$)(C$_8$H$_{12}$)], M_r = 1106·3, monoclinic, $P2_1/c$, a = 11·873 (4), b = 23·064 (9), c = 15·609 (9) Å, β = 107·826 (25)°, V = 4069 Å^3, Z = 4, D_x = 1·870 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 7·280 mm^{-1}, F(000) = 2144, T = 293 K, R = 0·0534 for 4612 unique observed reflections. In both structures, the central six-membered chelate ring comprising the two metal atoms, two P and two S atoms is found to adopt a boat conformation: in (I) the Pt···Rh separation is 4·3514 (13) Å, while (II) shows a Pt···Ir separation of 4·2728 (8) Å.

Experimental. Compound (I) prepared by reaction of [Rh(COD)Cl]$_2$ with NH$_2$Et$_2$[Pt(S$_2$CNEt$_2$)(PhPS)$_2$] in CDCl$_3$, crystals obtained from CDCl$_3$/MeOH. Orange lath, 0·56 × 0·20 × 0·08 mm, mounted about **c** on STADI-2 two-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell parameters from angles of 17 *hk*0 and four 00*l* reflections ($3 < 2\theta < 26°$). For data collection, ω scans with scan width $(2{\cdot}0 + 1{\cdot}5\sin\mu/\tan\theta)°$, $2\theta_{\max} = 50°$, h 0→27, k 0→27, l 0→16, 3 standard reflections showed no significant crystal movement or decay, initial absorption correction (min. 0·764, max. 1·310) using ψ scans on (004), 6554 reflections, 6533 unique ($R_{\rm int}$ = 0·006), giving 2679 with $F > 6\sigma(F)$ for structure solution [from a Patterson synthesis (Pt, Rh) followed by iterative cycles of least-squares refinement and difference Fourier synthesis] and refinement [using full-matrix least squares on F (Sheldrick, 1976)]. At isotropic convergence, final absorption corrections (min. 0·755, max. 1·224) applied empirically using *DIFABS* (Walker & Stuart, 1983). Anisotropic thermal parameters for Pt, Rh, S, P and N atoms, phenyl rings refined as rigid, idealized hexagons, H atoms in fixed, calculated positions. At final convergence, R = 0·0486, wR = 0·0366, S = 1·069 for 182 parameters, $(\Delta/\sigma)_{\max}$ in final cycle 0·023, max. and min. residues in final ΔF synthesis 0·80, −0·90 e Å^{-3} respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0001F^2$ gave satisfactory agreement analyses. Atomic coordinates and equiva-

Table 1. *Atomic coordinates and isotropic temperature factors for* (I)

	x	y	z	$U_{\rm iso}$
Rh	0·52217 (5)	0·14818 (5)	0·56533 (9)	0·0347 (7)
S(3)	0·51283 (15)	0·17481 (16)	0·7206 (3)	0·0395 (25)
S(4)	0·42066 (16)	0·14978 (20)	0·5361 (3)	0·043 (3)
Pt	0·39516 (2)	0·05649 (2)	0·73711 (4)	0·0291 (3)
S(1)	0·30672 (15)	0·00245 (17)	0·7186 (3)	0·0391 (25)
S(2)	0·40891 (17)	−0·03197 (18)	0·8199 (3)	0·053 (3)
N(1)	0·3109 (5)	−0·0984 (5)	0·8139 (9)	0·047 (9)
P(1)	0·36818 (16)	0·13209 (18)	0·6467 (3)	0·0304 (24)
P(2)	0·48116 (15)	0·10037 (16)	0·78055 (25)	0·0291 (23)
C(1)	0·6145 (6)	0·1248 (6)	0·5868 (10)	0·041 (4)
C(2)	0·6083 (6)	0·1850 (7)	0·5632 (11)	0·051 (4)
C(3)	0·6205 (7)	0·2115 (7)	0·4705 (11)	0·068 (6)
C(4)	0·5918 (7)	0·1780 (7)	0·3934 (12)	0·076 (6)
C(5)	0·5344 (7)	0·1485 (7)	0·4181 (11)	0·055 (5)
C(6)	0·5334 (6)	0·0919 (6)	0·4489 (10)	0·046 (4)
C(7)	0·5823 (6)	0·0506 (7)	0·4666 (12)	0·062 (5)
C(8)	0·6338 (6)	0·0788 (7)	0·5176 (11)	0·056 (5)
C(1*N*)	0·3384 (5)	−0·0497 (6)	0·7893 (9)	0·033 (4)
C(11)	0·2493 (7)	−0·1099 (6)	0·7920 (10)	0·048 (4)
C(12)	0·2435 (8)	−0·1522 (7)	0·7137 (13)	0·093 (7)
C(21)	0·3424 (7)	−0·1401 (8)	0·8730 (12)	0·061 (5)
C(22)	0·3258 (8)	−0·1300 (8)	0·9712 (14)	0·116 (8)
C(32)	0·3010 (4)	0·0713 (4)	0·5220 (6)	0·044 (5)
C(33)	0·2499 (4)	0·0573 (4)	0·4728 (6)	0·051 (4)
C(34)	0·1972 (4)	0·0876 (4)	0·4890 (6)	0·061 (5)
C(35)	0·1956 (4)	0·1319 (4)	0·5545 (6)	0·061 (5)
C(36)	0·2466 (4)	0·1459 (4)	0·6037 (6)	0·048 (4)
C(31)	0·2993 (4)	0·1155 (4)	0·5874 (6)	0·030 (4)
C(42)	0·3314 (4)	0·1891 (4)	0·8013 (7)	0·068 (6)
C(43)	0·3256 (4)	0·2370 (4)	0·8598 (7)	0·100 (7)
C(44)	0·3436 (4)	0·2925 (4)	0·8315 (7)	0·077 (6)
C(45)	0·3675 (4)	0·3003 (4)	0·7447 (7)	0·068 (5)
C(46)	0·3734 (4)	0·2524 (4)	0·6863 (7)	0·056 (5)
C(41)	0·3554 (4)	0·1969 (4)	0·7146 (7)	0·025 (4)
C(52)	0·5422 (3)	0·0041 (4)	0·7096 (5)	0·040 (4)
C(53)	0·5901 (3)	−0·0342 (4)	0·7014 (5)	0·043 (4)
C(54)	0·6387 (3)	−0·0289 (4)	0·7598 (5)	0·048 (4)
C(55)	0·6393 (3)	0·0148 (4)	0·8263 (5)	0·038 (4)
C(56)	0·5913 (3)	0·0531 (4)	0·8345 (5)	0·037 (4)
C(51)	0·5427 (3)	0·0477 (4)	0·7761 (5)	0·024 (3)
C(62)	0·4843 (4)	0·1779 (4)	0·9317 (6)	0·050 (5)
C(63)	0·4841 (4)	0·1908 (4)	1·0245 (6)	0·062 (5)
C(64)	0·4776 (4)	0·1458 (4)	1·0881 (6)	0·055 (5)
C(65)	0·4712 (4)	0·0880 (4)	1·0588 (6)	0·046 (4)
C(66)	0·4714 (4)	0·0751 (4)	0·9660 (6)	0·043 (4)
C(61)	0·4779 (4)	0·1201 (4)	0·9024 (6)	0·026 (3)

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Table 2. *Bond lengths* (Å), *angles* (°) *and torsion angles* (°) *for the six-membered* RhSPPtPS *ring in* (I)

Pt—S(1)	2·359 (4)	C(4)—C(5)	1·503 (23)
Pt—S(2)	2·377 (4)	C(5)—C(6)	1·368 (22)
Pt—P(1)	2·260 (4)	C(6)—C(7)	1·473 (22)
Pt—P(2)	2·273 (4)	C(7)—C(8)	1·525 (22)
Rh—S(3)	2·370 (4)	S(1)—C(1*N*)	1·733 (14)
Rh—S(4)	2·328 (4)	S(2)—C(1*N*)	1·700 (14)
Rh—C(1)	2·171 (14)	C(1*N*)—N(1)	1·323 (18)
Rh—C(2)	2·115 (15)	N(1)—C(11)	1·448 (19)
Rh—C(5)	2·181 (16)	N(1)—C(21)	1·472 (21)
Rh—C(6)	2·154 (15)	C(11)—C(12)	1·507 (23)
S(3)—P(2)	2·042 (5)	C(21)—C(22)	1·51 (3)
S(4)—P(1)	2·050 (6)	P(1)—C(31)	1·820 (10)
C(1)—C(2)	1·425 (20)	P(1)—C(41)	1·806 (10)
C(1)—C(8)	1·523 (21)	P(2)—C(51)	1·837 (9)
C(2)—C(3)	1·515 (22)	P(2)—C(61)	1·847 (10)
C(3)—C(4)	1·513 (23)		
S(1)—Pt—S(2)	74·05 (14)	C(1)—C(8)—C(7)	113·7 (12)
S(1)—Pt—P(1)	95·97 (14)	S(1)—C(1*N*)—S(2)	112·3 (8)
S(1)—Pt—P(2)	169·39 (13)	S(1)—C(1*N*)—N(1)	123·2 (11)
S(2)—Pt—P(1)	169·68 (14)	S(2)—C(1*N*)—N(1)	124·5 (11)
S(2)—Pt—P(2)	96·85 (14)	C(1*N*)—N(1)—C(11)	122·7 (12)
P(1)—Pt—P(2)	93·33 (13)	C(1*N*)—N(1)—C(21)	118·5 (12)
Pt—S(1)—C(1*N*)	86·7 (5)	C(11)—N(1)—C(21)	118·5 (12)
Pt—S(2)—C(1*N*)	86·8 (5)	N(1)—C(11)—C(12)	111·6 (13)
S(4)—P(1)—Pt	117·44 (21)	N(1)—C(21)—C(22)	110·2 (14)
Pt—P(1)—C(31)	110·6 (3)	S(4)—P(1)—C(31)	98·8 (4)
Pt—P(1)—C(41)	110·1 (4)	S(4)—P(1)—C(41)	111·6 (4)
S(3)—P(2)—Pt	122·91 (20)	C(31)—P(1)—C(41)	107·3 (5)
Pt—P(2)—C(51)	110·2 (3)	P(1)—C(31)—C(32)	117·2 (7)
Pt—P(2)—C(61)	110·2 (3)	P(1)—C(31)—C(36)	122·8 (7)
S(3)—Rh—S(4)	94·94 (14)	P(1)—C(41)—C(42)	117·5 (7)
Rh—S(3)—P(2)	103·46 (19)	P(1)—C(41)—C(46)	122·2 (7)
Rh—S(4)—P(1)	114·69 (21)	S(3)—P(2)—C(51)	105·4 (3)
C(2)—C(1)—C(8)	122·0 (13)	S(3)—P(2)—C(61)	103·2 (3)
C(1)—C(2)—C(3)	125·8 (13)	C(51)—P(2)—C(61)	102·9 (4)
C(2)—C(3)—C(4)	113·2 (13)	P(2)—C(51)—C(52)	119·0 (6)
C(3)—C(4)—C(5)	114·5 (14)	P(2)—C(51)—C(56)	120·9 (6)
C(4)—C(5)—C(6)	121·2 (14)	P(2)—C(61)—C(62)	121·7 (7)
C(5)—C(6)—C(7)	130·5 (14)	P(2)—C(61)—C(66)	118·3 (7)
C(6)—C(7)—C(8)	112·7 (13)		
S(4)—Rh—S(3)—P(2)	73·76 (20)	Rh—S(4)—P(1)—Pt	−44·3 (3)
S(3)—Rh—S(4)—P(1)	−27·94 (24)	P(2)—Pt—P(1)—S(4)	64·40 (24)
Rh—S(3)—P(2)—Pt	−57·89 (25)	P(1)—Pt—P(2)—S(3)	−9·85 (24)

lent isotropic thermal parameters are given in Table 1, while selected bond lengths, angles and torsion angles appear in Table 2.* The atomic numbering scheme for the molecule is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985).

Compound (II) synthesized by reaction of $[Ir(COD)Cl]_2$ with $NH_2{}^iPr_2[Pt(S_2CN{}^iPr_2)(PhPS)_2]$ in $CHCl_3$, crystals obtained from $CDCl_3$/MeOH. Red needle, 0·075 × 0·125 × 0·825 mm, mounted about **b** on STADI-2 two-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell parameters from angles of 14 $h0l$ and four $0k0$ reflections ($3·5 < 2\theta < 43°$). For data collection, ω scans with scan width $(0·8 + 0·4\sin\mu/\tan\theta)°$, $2\theta_{max} = 50°$, h $-14\rightarrow14$, k $0\rightarrow26$, l $0\rightarrow16$, 2 standard reflections showed no significant crystal movement or decay, initial absorption correction (min. 0·447, max. 2·230) using ψ scans, 7132 reflections, 6872 unique ($R_{int} = 0·020$),

Table 3. *Atomic coordinates and isotropic temperature factors for* (II)

	x	*y*	*z*	U_{iso}
Pt	0·80643 (5)	0·56164 (2)	0·21824 (3)	0·0328 (3)
S(3)	1·0605 (3)	0·66620 (14)	0·29508 (23)	0·0407 (20)
S(4)	0·8592 (4)	0·65511 (17)	0·41391 (23)	0·0466 (22)
Ir	1·06046 (5)	0·64096 (2)	0·44279 (4)	0·0390 (3)
S(1)	0·6253 (3)	0·51273 (15)	0·20068 (25)	0·0458 (22)
S(2)	0·8237 (4)	0·47093 (15)	0·1546 (3)	0·054 (3)
P(1)	0·7632 (3)	0·64341 (13)	0·28112 (24)	0·0361 (20)
P(2)	0·9843 (3)	0·59712 (13)	0·21644 (23)	0·0347 (19)
C(1)	1·0727 (19)	0·6483 (8)	0·5845 (13)	0·072 (5)
C(2)	1·0577 (19)	0·5915 (8)	0·5574 (13)	0·075 (5)
C(3)	1·1488 (20)	0·5426 (10)	0·5740 (16)	0·090 (6)
C(4)	1·2621 (19)	0·5591 (8)	0·5490 (14)	0·080 (6)
C(5)	1·2353 (14)	0·6039 (6)	0·4737 (11)	0·052 (4)
C(6)	1·2424 (15)	0·6634 (7)	0·4892 (11)	0·058 (4)
C(7)	1·2723 (18)	0·6934 (8)	0·5818 (12)	0·072 (5)
C(8)	1·1959 (21)	0·6735 (10)	0·6358 (15)	0·093 (7)
C(1*N*)	0·6802 (13)	0·4571 (5)	0·1492 (9)	0·041 (3)
N(1)	0·6180 (12)	0·4114 (5)	0·1125 (9)	0·055 (3)
C(11)	0·4968 (19)	0·4013 (9)	0·1123 (14)	0·078 (5)
C(12)	0·482 (3)	0·3686 (13)	0·1838 (21)	0·150 (12)
C(13)	0·4046 (23)	0·4345 (10)	0·0441 (17)	0·106 (8)
C(21)	0·6730 (20)	0·3692 (8)	0·0639 (14)	0·079 (5)
C(22)	0·707 (3)	0·3148 (12)	0·1024 (20)	0·141 (11)
C(23)	0·660 (3)	0·3824 (12)	−0·0251 (17)	0·120 (9)
C(32)	0·5877 (8)	0·6067 (4)	0·3505 (6)	0·053 (4)
C(33)	0·4761 (8)	0·6072 (4)	0·3626 (6)	0·079 (5)
C(34)	0·3900 (8)	0·6461 (4)	0·3142 (6)	0·066 (5)
C(35)	0·4157 (8)	0·6846 (4)	0·2538 (6)	0·070 (5)
C(36)	0·5273 (8)	0·6841 (4)	0·2417 (6)	0·058 (4)
C(31)	0·6133 (8)	0·6452 (4)	0·2900 (6)	0·037 (3)
C(42)	0·7831 (11)	0·7624 (4)	0·2525 (5)	0·066 (5)
C(43)	0·7752 (11)	0·8111 (4)	0·1979 (5)	0·088 (6)
C(44)	0·7473 (11)	0·8046 (4)	0·1049 (5)	0·081 (6)
C(45)	0·7274 (11)	0·7495 (4)	0·0665 (5)	0·073 (5)
C(46)	0·7352 (11)	0·7008 (4)	0·1211 (5)	0·052 (4)
C(41)	0·7631 (11)	0·7073 (4)	0·2141 (5)	0·041 (3)
C(52)	1·1921 (9)	0·5423 (4)	0·2098 (6)	0·054 (4)
C(53)	1·2858 (9)	0·5035 (4)	0·2412 (6)	0·075 (5)
C(54)	1·2857 (9)	0·4639 (4)	0·3087 (6)	0·073 (5)
C(55)	1·1919 (9)	0·4632 (4)	0·3449 (6)	0·073 (5)
C(56)	1·0982 (9)	0·5020 (4)	0·3136 (6)	0·052 (4)
C(51)	1·0983 (9)	0·5416 (4)	0·2460 (6)	0·040 (3)
C(62)	0·9509 (10)	0·5757 (3)	0·0361 (7)	0·053 (4)
C(63)	0·9472 (10)	0·5900 (3)	−0·0516 (7)	0·067 (5)
C(64)	0·9729 (10)	0·6465 (3)	−0·0719 (7)	0·083 (6)
C(65)	1·0024 (10)	0·6885 (3)	−0·0045 (7)	0·072 (5)
C(66)	1·0061 (10)	0·6742 (3)	0·0832 (7)	0·061 (4)
C(61)	0·9804 (10)	0·6178 (3)	0·1035 (7)	0·040 (3)

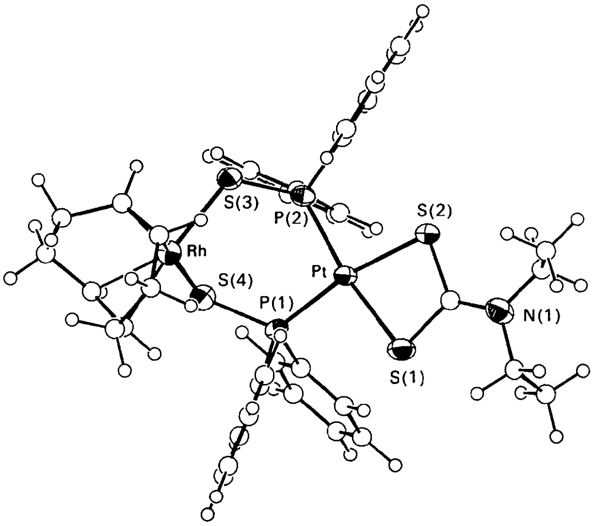

Fig. 1. A general view of a molecule of (I) showing atomic numbering scheme; thermal ellipsoids are drawn at the 30% probability level, except those of C and H which have artificial radii of 0·15 and 0·10 Å respectively for clarity.

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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 52605 (48 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Bond lengths* (Å), *angles* (°) *and torsion angles* (°) *for the six-membered* IrSPPtPS *ring in* (II)

Pt—S(1)	2·368 (4)	C(3)—C(4)	1·56 (3)
Pt—S(2)	2·352 (4)	C(4)—C(5)	1·52 (3)
Pt—P(1)	2·257 (4)	C(5)—C(6)	1·392 (24)
Pt—P(2)	2·273 (4)	C(6)—C(7)	1·54 (3)
S(4)—Ir	2·315 (4)	C(7)—C(8)	1·49 (3)
S(4)—P(1)	2·055 (5)	C(1*N*)—N(1)	1·313 (20)
S(3)—Ir	2·378 (4)	N(1)—C(11)	1·46 (3)
S(3)—P(2)	2·045 (5)	N(1)—C(21)	1·50 (3)
Ir—C(1)	2·178 (20)	C(11)—C(12)	1·40 (4)
Ir—C(2)	2·132 (21)	C(11)—C(13)	1·48 (3)
Ir—C(5)	2·159 (16)	C(21)—C(22)	1·40 (4)
Ir—C(6)	2·122 (17)	C(21)—C(23)	1·38 (4)
S(1)—C(1*N*)	1·742 (15)	P(1)—C(31)	1·827 (10)
S(2)—C(1*N*)	1·711 (15)	P(1)—C(41)	1·807 (11)
C(1)—C(2)	1·37 (3)	P(2)—C(51)	1·817 (10)
C(1)—C(8)	1·55 (3)	P(2)—C(61)	1·814 (10)
C(2)—C(3)	1·53 (3)		

S(1)—Pt—S(2)	73·80 (14)	N(1)—C(11)—C(13)	115·4 (18)
S(1)—Pt—P(1)	97·40 (13)	C(12)—C(11)—C(13)	127·2 (23)
S(1)—Pt—P(2)	169·73 (13)	N(1)—C(21)—C(22)	118·7 (20)
S(2)—Pt—P(1)	171·15 (14)	N(1)—C(21)—C(23)	115·5 (19)
S(2)—Pt—P(2)	96·51 (14)	C(22)—C(21)—C(23)	124·0 (23)
P(1)—Pt—P(2)	92·33 (13)	Pt—P(1)—S(4)	114·74 (19)
Ir—S(4)—P(1)	113·51 (20)	Pt—P(1)—C(31)	114·1 (3)
Ir—S(3)—P(2)	105·05 (18)	Pt—P(1)—C(41)	112·9 (4)
S(3)—Ir—S(4)	94·78 (13)	S(4)—P(1)—C(31)	100·0 (4)
Pt—S(1)—C(1*N*)	87·1 (5)	S(4)—P(1)—C(41)	112·2 (4)
Pt—S(2)—C(1*N*)	88·4 (5)	C(31)—P(1)—C(41)	101·4 (5)
C(2)—C(1)—C(8)	122·0 (18)	P(1)—C(31)—C(32)	117·2 (7)
C(1)—C(2)—C(3)	129·3 (19)	P(1)—C(31)—C(36)	122·8 (7)
C(2)—C(3)—C(4)	113·5 (18)	P(1)—C(41)—C(42)	121·6 (8)
C(3)—C(4)—C(5)	111·5 (17)	P(1)—C(41)—C(46)	118·3 (8)
C(4)—C(5)—C(6)	123·1 (15)	Pt—P(2)—S(3)	121·00 (19)
C(5)—C(6)—C(7)	126·1 (16)	Pt—P(2)—C(51)	111·5 (4)
C(6)—C(7)—C(8)	112·8 (17)	Pt—P(2)—C(61)	111·1 (4)
C(1)—C(8)—C(7)	117·8 (19)	S(3)—P(2)—C(51)	103·8 (4)
S(1)—C(1*N*)—S(2)	110·3 (8)	S(3)—P(2)—C(61)	104·7 (4)
S(1)—C(1*N*)—N(1)	123·5 (11)	C(51)—P(2)—C(61)	103·1 (5)
S(2)—C(1*N*)—N(1)	126·1 (12)	P(2)—C(51)—C(52)	120·9 (8)
C(1*N*)—N(1)—C(11)	123·6 (14)	P(2)—C(51)—C(56)	118·7 (8)
C(1*N*)—N(1)—C(21)	117·9 (14)	P(2)—C(61)—C(62)	118·4 (8)
C(11)—N(1)—C(21)	118·4 (15)	P(2)—C(61)—C(66)	121·6 (8)
N(1)—C(11)—C(12)	116·6 (21)		

P(2)—Pt—P(1)—S(4)	−71·85 (21)	Ir—S(4)—P(1)—Pt	47·9 (3)
P(1)—Pt—P(2)—S(3)	17·89 (23)	P(2)—S(3)—Ir—S(4)	−73·75 (19)
P(1)—S(4)—Ir—S(3)	26·99 (22)	Ir—S(3)—P(2)—Pt	51·98 (24)

giving 4612 with $F > 6\sigma(F)$ for structure solution [from a Patterson synthesis (Pt, Ir) followed by iterative cycles of least-squares refinement and difference Fourier synthesis] and refinement [using full-matrix least squares on F (Sheldrick, 1976)]. At isotropic convergence, final absorption corrections applied empirically using *DIFABS* (Walker & Stuart, 1983). Anisotropic thermal parameters for Pt, Ir, S, P and N atoms, phenyl rings refined as rigid, idealized hexagons, H atoms in fixed, calculated positions. Inter-layer scale factors (on **b** axis) refined. At final convergence, $R = 0{\cdot}0534$, $wR = 0{\cdot}0712$, $S = 0{\cdot}978$ for 210 parameters, $(\Delta/\sigma)_{\max}$ in final cycle 0·050, max. and min. residues in final ΔF synthesis 1·41, −1·42 e Å^{-3} respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}002153F^2$ gave satisfactory agreement analyses. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 3, while selected bond lengths, angles and torsion angles appear in Table 4.* The atomic numbering scheme for the molecule is shown in Fig. 2, which was generated using *ORTEP* (Mallinson & Muir, 1985). Fig. 3 shows the boat conformation adopted by the central (Rh,Ir)SPPtPS ring.

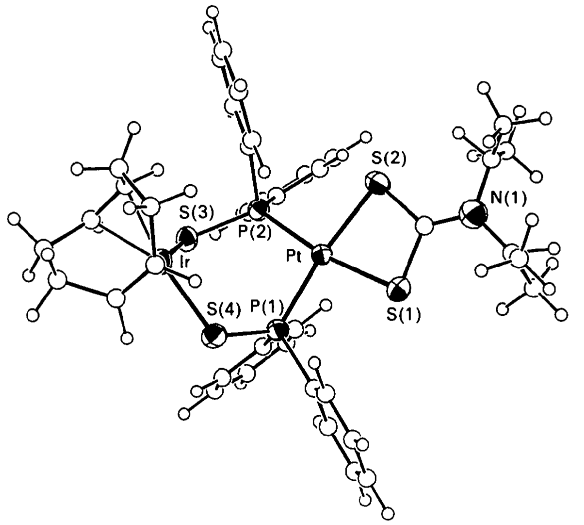

Fig. 2. A general view of a molecule of (II) showing atomic numbering scheme; thermal ellipsoids are drawn at the 30% probability level, except those of C and H which have artificial radii of 0·15 and 0·10 Å respectively for clarity.

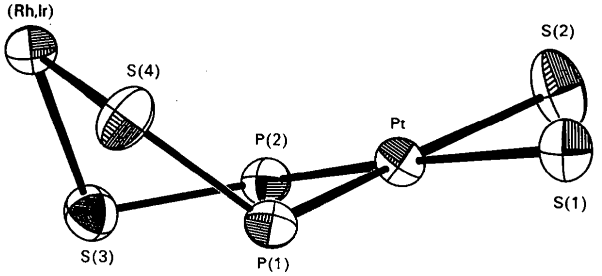

Fig. 3. A view of the central (Rh,Ir)SPPtPS ring showing the boat conformation adopted.

For both structures, molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985) and scattering factors were inlaid (Sheldrick, 1976) except for Rh, Ir and Pt (Cromer & Mann, 1968).

Related literature. Complexes which show similar six-membered heterobimetallic rings include ${}^{i}Pr_2NCS_2Pt\text{-}\mu(Ph_2PS)_2\text{-}PtS_2CNEt_2$ (Anderson, 1982), $\{UO_2(OH_2)[(OPPh_2)_2Pd(S_2CNEt_2)]_2\}$ (Allan, Blake, Schröder & Veitch, 1987) and $[{}^{i}Pr_2NCS_2Pt\text{-}\mu(Ph_2PS)\text{-}\mu(Ph_2PO)]_2Co$ (Anderson, Blake, Fotheringham, Stephenson, Allan & Veitch, 1988).

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* See deposition footnote.

References

ALLAN, J. R., BLAKE, A. J., SCHRÖDER, M. & VEITCH, P. M. (1987). *J. Chem. Soc. Dalton Trans.* pp. 2853–2856.

ANDERSON, D. M. (1982). PhD Thesis. Univ. of Edinburgh, Scotland.

ANDERSON, D. M., BLAKE, A. J., FOTHERINGHAM, J. D., STEPHENSON, T. A., ALLAN, J. R. & VEITCH, P. M. (1988). *Acta Cryst.* **C44**, 1305–1307.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

GOULD, R. O. & TAYLOR, P. (1985). *CALC*. Program for molecular geometry calculations, Fortran77 version. Univ. of Edinburgh, Scotland.

MALLINSON, P. D. & MUIR, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1990). **C46**, 1105–1107

Structure of $[RuI(C_{10}H_8N_2)(C_{15}H_{11}N_3)]PF_6.0{\cdot}5C_7H_8$

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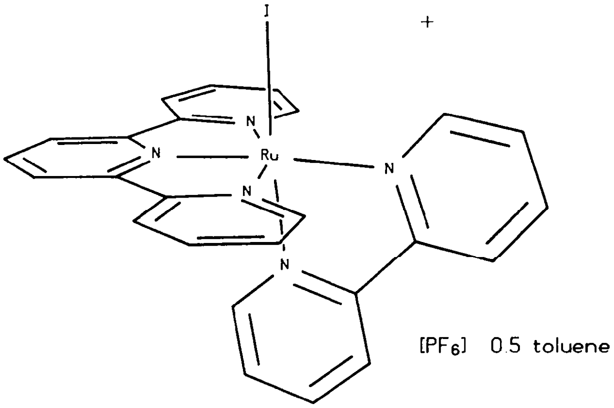
Abstract. 2,2′-Bipyridyl(iodo)(2,2′:6′,2″-terpyridyl)ruthenium(II) hexafluorophosphate 0·5 toluene solvate (1), $C_{28\cdot5}H_{23}F_6IN_5PRu$, M_r = 808·51, monoclinic, $P2_1/n$, a = 14·482 (3), b = 12·841 (3), c = 16·100 (3) Å, β = 94·92 (2)°, V = 2983 (1) Å^3, Z = 4, D_x = 1·80 g cm^{-3}, Mo $K\alpha$, λ = 0·71073 Å, μ = 16·56 cm^{-1}, $F(000)$ = 1580, T = 294 K, R = 0·0367 for 3056 observed reflections. The Ru—I distance is 2·711 (1) Å; Ru—N distances range from 1·944 (6) to 2·092 (5) Å. Bite angles are 79·1 (2) and 80·0 (2)° for the terpyridine ligand, and 77·7 (2)° for the bipyridine ligand. Ligand geometries are indicative of considerable strain associated with metal coordination. In addition to the ruthenium cation and a hexafluorophosphate anion, the asymmetric unit contains a toluene solvent molecule which is disordered about an inversion center ($\frac{1}{2}$,0,0).

Experimental. Title compound prepared by heating an ethanol solution containing one equivalent of $[Ru(C_{15}H_{11}N_3)(CF_3SO_3)_3]$, 0·9 equivalents of 2,2′-bipyridine and two equivalents of NH_4I at reflux for 3 h. Addition of saturated KPF_6 solution resulted in a brown precipitate which was collected and chromatographed on N-alumina using an acetonitrile/toluene solution. Deep-purple parallelepiped crystal of (1) obtained by slow evaporation of an acetonitrile/toluene solution, dimensions 0·35 × 0·47 × 0·52 mm. Data collected at room temperature, graphite-monochromated Mo $K\alpha$ radiation (λ = 0·71073 Å), Nicolet $R3m/V$ diffractometer, $\omega/2\theta$ scans of 2–15° min^{-1}, $2\theta_{max}$ = 45°, h = 0 to 15, k = 0 to 13, l = −17 to 17, 4317 measurements, 3916 unique (R_{int} = 0·013), 3056 observed [$I > 3\sigma(I)$]. Unit-cell dimensions determined by least-squares fit to settings for 32 reflections ($16 < 2\theta < 29°$). Empirical absorption correction (μ = 16·56 cm^{-1}), transmission factors 0·84–1·00; secondary-extinction correction [η = 0·00012 (3)]; three standards monitored (± 1%), 37·7 h of X-ray exposure.



(1)

Solved by direct methods; full-matrix least-squares refinement on F, R = 0·0367, wR = 0·0470, S = 1·33, 374 variables including positional and anisotropic thermal parameters for non-hydrogen atoms, hydrogen atoms of the cation in idealized positions [d(C—H) = 0·96 Å] with isotropic group thermal parameter [U_{iso} = 0·085 (6) Å^2], $(\Delta/\sigma)_{max}$ = 0·01, function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma^2(F_o) + 0{\cdot}0005|F_o|^2]$.

Final difference map peaks ranged from −0·53 to 0·82 e Å^{-3}; largest peaks located near the hexafluorophosphate anion and disordered solvent molecule. Computer programs (Sheldrick, 1986), scattering fac-

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