

Table 1. Atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$B_{eq}$
U	0.000	0.05533 (3)	0.250	3.780 (7)
F(1)	0.3111 (3)	0.1681 (7)	0.3457 (7)	15.3 (2)
F(2)	0.3768 (4)	0.0437 (5)	0.3439 (7)	12.4 (3)
F(3)	0.4122 (3)	0.1984 (7)	0.3440 (9)	14.9 (3)
F(4)	0.3313 (4)	0.1365 (8)	0.1808 (6)	13.5 (3)
O(1)	0.0406 (2)	0.0556 (5)	0.1415 (4)	5.6 (1)
O(2)	0.000	0.2309 (7)	0.250	7.0 (3)
O(3)	0.0647 (3)	-0.0881 (4)	0.3514 (5)	5.4 (1)
O(4)	-0.1051 (3)	0.1092 (4)	0.0922 (5)	5.7 (1)
N(2)	0.000	0.3973 (7)	0.250	6.5 (2)
N(3)	0.1022 (3)	-0.2168 (5)	0.4864 (5)	4.6 (1)
N(4)	-0.1917 (3)	0.0942 (5)	-0.0962 (6)	5.6 (2)
C(2)	0.0285 (8)	0.311 (1)	0.308 (1)	6.2 (4)
C(3)	0.0540 (4)	-0.1663 (6)	0.4012 (7)	5.0 (2)
C(4)	-0.1258 (4)	0.0982 (7)	-0.0248 (8)	5.8 (2)
C(21)	0.0428 (7)	0.455 (2)	0.350 (1)	16.1 (6)
C(31)	0.0875 (5)	-0.3113 (7)	0.5378 (9)	8.0 (3)
C(32)	0.1738 (4)	-0.1822 (8)	0.534 (1)	7.9 (3)
C(41)	-0.2100 (5)	0.082 (1)	-0.2329 (8)	8.4 (3)
C(42)	-0.2458 (4)	0.105 (1)	-0.048 (1)	8.7 (3)
B	0.3587 (5)	0.140 (1)	0.305 (1)	6.6 (3)

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

Table 2. Selected bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ )

Uranium coordination sphere			
U—O(1)	1.762 (3)	U—O(2)	2.334 (5)
U—O(3)	2.378 (3)	U—O(4)	2.383 (3)
O(1)—U—O(2)	89.9 (1)	O(1)—U—O(3)	90.5 (1)
O(1)—U—O(4)	91.1 (1)	O(1)—U—O(4)	88.8 (1)
dmf			
O(2)—C(2)	1.28 (1)	N(3)—C(31)	1.471 (6)
C(2)—N(2)	1.35 (1)	N(3)—C(32)	1.471 (6)
N(2)—C(21)	1.391 (8)	O(4)—C(4)	1.259 (5)
O(3)—C(3)	1.249 (5)	C(4)—N(4)	1.320 (5)
C(3)—N(3)	1.301 (5)	N(4)—C(41)	1.482 (6)
		N(4)—C(42)	1.457 (6)
U—O(2)—C(2)	146.3 (5)	U—O(3)—C(3)	136.3 (3)
U—O(4)—C(4)	128.8 (3)		
BF <sub>4</sub>			
B—F(1)	1.311 (7)	B—F(2)	1.359 (7)
B—F(3)	1.303 (7)	B—F(4)	1.329 (7)
F(1)—B—F(2)	108.2 (6)	F(1)—B—F(3)	112.7 (7)
F(1)—B—F(4)	108.4 (6)	F(2)—B—F(3)	110.5 (6)
F(2)—B—F(4)	106.0 (6)	F(3)—B—F(4)	110.8 (6)

Symmetry code: (i)  $-x, y, \frac{1}{2} - z$ .

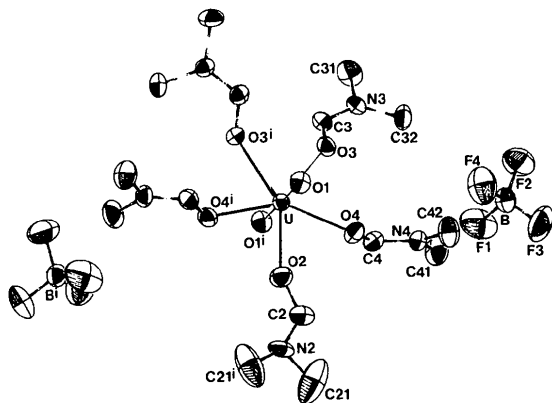


Fig. 1. Perspective view drawn using ORTEP (Johnson, 1976). Atoms labelled with (i) are related by the diad axis.

**Related literature.** This work is part of structural studies of uranyl with poorly coordinating anions in various solvents (Alcock & Esperas, 1977).

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*Acta Cryst.* (1992). **C48**, 1661–1663

## Structure of [1,3-Bis(diphenylphosphino)propane]( $\eta^5$ -cyclopentadienyl)hydridoruthenium(II)

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(Received 25 November 1991; accepted 28 January 1992)

**Abstract.**  $[\text{RuH}(\text{C}_5\text{H}_5)(\text{C}_{27}\text{H}_{26}\text{P}_2)]$ ,  $M_r = 579.6$ , monoclinic,  $P2_1/n$ ,  $a = 9.188$  (2),  $b = 19.307$  (3),  $c = 15.545$  (3)  $\text{\AA}$ ,  $\beta = 102.98$  (1) $^\circ$ ,  $V = 2687.0$  (8)  $\text{\AA}^3$ ,  $Z =$

4,  $D_x = 1.43$   $\text{Mg m}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.7107$   $\text{\AA}$ ,  $\mu = 0.707$   $\text{mm}^{-1}$ ,  $F(000) = 1192$ ,  $T = 213$  K,  $R = 0.039$  for 3412 observed reflections. The compound was

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

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

	x	y	z	$U_{eq}$
Ru(1)	1253 (1)	1312 (1)	2951 (1)	27 (1)
C(6)	-512 (6)	38 (3)	1097 (3)	32 (2)
C(7)	30 (6)	-277 (3)	2023 (3)	30 (2)
C(8)	-1319 (5)	723 (2)	1085 (3)	32 (2)
P(1)	-99 (1)	1431 (1)	1586 (1)	27 (1)
C(11)	-1381 (5)	2172 (2)	1494 (3)	31 (2)
C(12)	-2800 (6)	2104 (3)	1642 (4)	48 (2)
C(13)	-3704 (7)	2674 (4)	1682 (4)	62 (3)
C(14)	-3155 (8)	3325 (3)	1598 (4)	60 (3)
C(15)	-1754 (7)	3413 (3)	1457 (4)	50 (2)
C(16)	-876 (6)	2841 (3)	1399 (3)	41 (2)
C(21)	896 (5)	1598 (2)	703 (3)	25 (1)
C(22)	184 (6)	1824 (2)	-137 (3)	30 (2)
C(23)	968 (6)	1908 (2)	-789 (3)	37 (2)
C(24)	2483 (7)	1773 (3)	-614 (3)	41 (2)
C(25)	3209 (6)	1549 (3)	212 (4)	41 (2)
C(26)	2421 (6)	1464 (3)	865 (3)	34 (2)
P(2)	1590 (1)	182 (1)	2749 (1)	26 (1)
C(31)	1888 (5)	-382 (2)	3734 (3)	28 (2)
C(32)	2329 (6)	-1065 (3)	3711 (3)	42 (2)
C(33)	2498 (7)	-1492 (3)	4449 (4)	51 (2)
C(34)	2224 (7)	-1233 (3)	5212 (4)	55 (2)
C(35)	1787 (8)	-553 (3)	5249 (4)	61 (3)
C(36)	1639 (7)	-129 (3)	4513 (3)	47 (2)
C(41)	3195 (5)	-84 (2)	2301 (3)	28 (2)
C(42)	3110 (6)	-527 (3)	1585 (3)	36 (2)
C(43)	4367 (6)	-660 (3)	1259 (3)	45 (2)
C(44)	5724 (6)	-373 (3)	1643 (4)	43 (2)
C(45)	5833 (6)	53 (3)	2357 (4)	43 (2)
C(46)	4584 (6)	193 (3)	2685 (3)	34 (2)
C(1)	1804 (8)	1907 (3)	4228 (4)	56 (3)
C(2)	1375 (7)	2375 (3)	3514 (4)	53 (2)
C(3)	2436 (7)	2325 (3)	2997 (4)	49 (2)
C(4)	3501 (7)	1837 (3)	3360 (4)	48 (2)
C(5)	3111 (7)	1570 (3)	4130 (3)	47 (2)

prepared in the course of synthetic and structural studies of ruthenium(II) dihydrogen complexes. The Ru atom has pseudo-octahedral geometry: P(1)—Ru(1)—P(2) 91.9 (1), P(1)—Ru(1)—H(10) 87 (2) and P(2)—Ru(1)—H(10) 83 (2)°. The metal-hydride distance Ru(1)—H(10) is 1.55 (5) Å and the two metal-phosphorus distances, Ru(1)—P(1) and Ru(1)—P(2), are 2.217 (1) and 2.236 (1) Å, respectively.

**Experimental.** A yellow irregular-shaped crystal 0.30  $\times$  0.25  $\times$  0.13 mm was used for data collection on a Siemens R3m/V diffractometer with graphite monochromator. Cell dimensions were determined from 25 centred reflections with  $2\theta$  values of 13–28°. A total of 6773 reflections were measured in the  $2\theta$  range 3–50° using  $\theta$ – $2\theta$  scans with index range  $0 \leq h \leq 11$ ,  $0 \leq k \leq 25$ ,  $-20 \leq l \leq 20$ . Three reference reflections monitored periodically showed no significant change. A semi-empirical absorption correction based on azimuthal scans was applied; minimum/maximum transmission 0.78/0.83. Structure solution, refinement and graphical representation used *SHELXTL-Plus* (Sheldrick, 1988). Structure determination was by direct methods and refinement by full-matrix least squares on  $F$  with all non-H atoms with anisotropic thermal parameters. All H atoms were found from difference maps and were included in the refinement with fixed isotropic thermal param-

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

Ru(1)—H(10)	1.546 (47)	Ru(1)—P(1)	2.217 (1)
Ru(1)—P(2)	2.236 (1)	Ru(1)—C(1)	2.251 (6)
Ru(1)—C(2)	2.225 (5)	Ru(1)—C(3)	2.230 (6)
Ru(1)—C(4)	2.259 (6)	Ru(1)—C(5)	2.262 (5)
C(6)—C(7)	1.539 (6)	C(6)—C(8)	1.515 (7)
C(7)—P(2)	1.840 (5)	C(8)—P(1)	1.826 (5)
P(1)—C(11)	1.838 (5)	P(1)—C(21)	1.840 (5)
C(11)—C(12)	1.380 (8)	C(11)—C(16)	1.391 (7)
C(12)—C(13)	1.390 (9)	C(13)—C(14)	1.371 (10)
C(14)—C(15)	1.364 (10)	C(15)—C(16)	1.383 (8)
C(21)—C(22)	1.393 (6)	C(21)—C(26)	1.391 (7)
C(22)—C(23)	1.379 (8)	C(23)—C(24)	1.382 (8)
C(24)—C(25)	1.377 (7)	C(25)—C(26)	1.382 (8)
P(2)—C(31)	1.849 (4)	P(2)—C(41)	1.839 (5)
C(31)—C(32)	1.382 (7)	C(31)—C(36)	1.372 (7)
C(32)—C(33)	1.392 (8)	C(33)—C(34)	1.362 (9)
C(34)—C(35)	1.377 (9)	C(35)—C(36)	1.389 (8)
C(41)—C(42)	1.392 (7)	C(41)—C(46)	1.389 (6)
C(42)—C(43)	1.385 (8)	C(43)—C(44)	1.372 (8)
C(44)—C(45)	1.368 (8)	C(45)—C(46)	1.382 (8)
C(1)—C(2)	1.417 (8)	C(1)—C(5)	1.404 (9)
C(2)—C(3)	1.400 (10)	C(3)—C(4)	1.385 (8)
C(4)—C(5)	1.420 (9)		
H(10)—Ru(1)—P(1)	87.0 (15)	H(10)—Ru(1)—P(2)	82.8 (16)
P(1)—Ru(1)—P(2)	91.9 (1)	H(10)—Ru(1)—C(1)	88.7 (16)
P(1)—Ru(1)—C(1)	139.3 (2)	P(2)—Ru(1)—C(1)	127.6 (2)
H(10)—Ru(1)—C(2)	99.7 (16)	P(1)—Ru(1)—C(2)	104.5 (1)
P(2)—Ru(1)—C(2)	163.6 (1)	C(1)—Ru(1)—C(2)	36.9 (2)
H(10)—Ru(1)—C(3)	135.5 (16)	P(1)—Ru(1)—C(3)	96.0 (1)
P(2)—Ru(1)—C(3)	141.1 (2)	C(1)—Ru(1)—C(3)	60.9 (2)
C(2)—Ru(1)—C(3)	36.6 (2)	H(10)—Ru(1)—C(4)	148.7 (15)
P(1)—Ru(1)—C(4)	120.3 (2)	P(2)—Ru(1)—C(4)	109.3 (1)
C(1)—Ru(1)—C(4)	60.8 (2)	C(2)—Ru(1)—C(4)	60.9 (2)
C(3)—Ru(1)—C(4)	35.9 (2)	H(10)—Ru(1)—C(5)	113.6 (15)
P(1)—Ru(1)—C(5)	155.7 (2)	P(2)—Ru(1)—C(5)	103.0 (1)
C(1)—Ru(1)—C(5)	36.3 (2)	C(2)—Ru(1)—C(5)	61.0 (2)
C(3)—Ru(1)—C(5)	60.5 (2)	C(4)—Ru(1)—C(5)	36.6 (2)
C(7)—C(6)—C(8)	114.2 (4)	C(6)—C(7)—P(2)	115.3 (3)
C(6)—C(8)—P(1)	113.3 (4)	Ru(1)—P(1)—C(8)	119.2 (2)
Ru(1)—P(1)—C(11)	111.5 (1)	C(8)—P(1)—C(11)	103.2 (2)
Ru(1)—P(1)—C(21)	117.8 (1)	C(8)—P(1)—C(21)	100.0 (2)
C(11)—P(1)—C(21)	102.9 (2)	P(1)—C(11)—C(12)	121.7 (4)
P(1)—C(11)—C(16)	120.5 (4)	C(12)—C(11)—C(16)	117.2 (5)
C(11)—C(12)—C(13)	121.9 (5)	C(12)—C(13)—C(14)	119.0 (6)
C(13)—C(14)—C(15)	120.7 (6)	C(14)—C(15)—C(16)	119.7 (6)
C(11)—C(16)—C(15)	121.5 (5)	P(1)—C(21)—C(22)	123.1 (4)
P(1)—C(21)—C(26)	118.8 (3)	C(22)—C(21)—C(26)	118.1 (5)
C(21)—C(22)—C(23)	120.9 (5)	C(22)—C(23)—C(24)	120.1 (4)
C(23)—C(24)—C(25)	119.9 (6)	C(24)—C(25)—C(26)	119.9 (5)
C(21)—C(26)—C(25)	121.1 (4)	Ru(1)—P(2)—C(7)	116.2 (2)
Ru(1)—P(2)—C(31)	117.3 (1)	C(7)—P(2)—C(31)	100.0 (2)
Ru(1)—P(2)—C(41)	118.3 (1)	C(7)—P(2)—C(41)	102.3 (2)
C(31)—P(2)—C(41)	99.6 (2)	P(2)—C(31)—C(32)	122.1 (4)
P(2)—C(31)—C(36)	119.8 (4)	C(32)—C(31)—C(36)	118.1 (4)
C(31)—C(32)—C(33)	121.5 (5)	C(32)—C(33)—C(34)	119.4 (5)
C(33)—C(34)—C(35)	120.0 (5)	C(34)—C(35)—C(36)	120.2 (6)
C(31)—C(36)—C(35)	120.8 (5)	P(2)—C(41)—C(42)	124.7 (4)
P(2)—C(41)—C(46)	117.9 (3)	C(42)—C(41)—C(46)	117.4 (5)
C(41)—C(42)—C(43)	120.3 (4)	C(42)—C(43)—C(44)	121.1 (5)
C(43)—C(44)—C(45)	119.3 (6)	C(44)—C(45)—C(46)	120.1 (5)
C(41)—C(46)—C(45)	121.7 (5)	Ru(1)—C(1)—C(2)	70.5 (3)
Ru(1)—C(1)—C(5)	72.3 (3)	C(2)—C(1)—C(5)	107.7 (6)
Ru(1)—C(2)—C(1)	72.6 (3)	Ru(1)—C(2)—C(3)	71.9 (3)
C(1)—C(2)—C(3)	107.4 (5)	Ru(1)—C(3)—C(2)	71.5 (3)
Ru(1)—C(3)—C(4)	73.2 (3)	C(2)—C(3)—C(4)	109.4 (5)
Ru(1)—C(4)—C(3)	70.9 (3)	Ru(1)—C(4)—C(5)	71.8 (3)
C(3)—C(4)—C(5)	107.6 (6)	Ru(1)—C(5)—C(1)	71.4 (3)
Ru(1)—C(5)—C(4)	71.6 (3)	C(1)—C(5)—C(4)	107.9 (5)

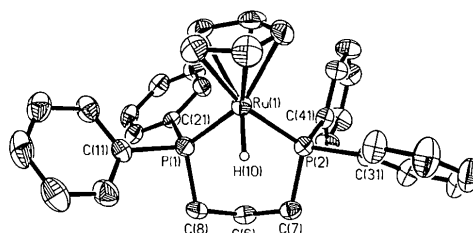


Fig. 1. Molecular structure of the title complex.

eters. The positional parameters of the H atoms were allowed to refine freely. 6197 reflections were unique ( $R_{\text{int}} = 0.024$ ) of which 3412 were observed with  $I > 3\sigma(I)$ . At convergence  $R = 0.039$ ,  $wR = 0.036$  (on all data  $R = 0.091$ ,  $wR = 0.046$ ),  $w^{-1} = [\sigma^2(F) + 0.0002F^2]$ ,  $S = 1.20$  for 412 parameters, maximum  $\Delta/\sigma = 0.001$ ,  $\Delta\rho_{\text{max}} = 0.43$ ,  $\Delta\rho_{\text{min}} = -0.53 \text{ e } \text{\AA}^{-3}$ . Scattering factors for all atoms were obtained from *SHELXTL-Plus*. Atomic parameters are given in Table 1,\* bond distances and angles in Table 2, and Fig. 1 shows the molecule together with the numbering scheme used.

\* 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 55103 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0120]

**Related literature.** The Ru—H(10) distance [1.55 (5) Å] is comparable with those reported for other ruthenium(II)–hydride bond lengths: 1.49 (4) Å in [Ru(H)(H<sub>2</sub>BH<sub>2</sub>)(PMe<sub>3</sub>)<sub>3</sub>] (Slater, Wilkinson, Thornton-Pett & Hursthouse, 1984) and 1.50 (4) Å in [(P–N)( $\eta^2$ -H<sub>2</sub>)Ru( $\mu$ -Cl)<sub>2</sub>( $\mu$ -H)Ru(H)(PPh<sub>3</sub>)<sub>2</sub>], where P–N = [Fe{ $\eta$ -C<sub>5</sub>H<sub>3</sub>(CHMeNMe<sub>2</sub>)P(Pr)<sub>2</sub>-1,2}-( $\eta^5$ -C<sub>5</sub>H<sub>5</sub>)] (Hampton, Cullen, James & Charland, 1988).

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*Acta Cryst.* (1992). **C48**, 1663–1665

## Rhodium(I)–Cyclooctadiene (cod) Complexes with the *N*-Donor Ligands 1,8-Diazabicyclo[5.4.0]undec-7-ene (dbu) and 1,5-Diazabicyclo[4.3.0]non-5-ene (dbn)

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(Received 21 November 1991; accepted 20 January 1992)

**Abstract.** Chloro( $\eta^4$ -1,5-cyclooctadiene)(1,8-diazabicyclo[5.4.0]undec-7-ene)rhodium(I), (I), [RhCl(C<sub>8</sub>H<sub>12</sub>)(C<sub>9</sub>H<sub>16</sub>N<sub>2</sub>)],  $M_r = 398.8$ , monoclinic,  $P2_1$ ,  $a = 7.398$  (1),  $b = 11.439$  (2),  $c = 10.727$  (2) Å,  $\beta = 106.42$  (1)°,  $V = 870.7$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.521 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 1.12 \text{ mm}^{-1}$ ,  $F(000) = 412$ ,  $T = 296$  (1) K, final  $R = 0.026$ ,  $wR = 0.029$  for 1912 unique observed intensities. Chloro( $\eta^4$ -1,5-cyclooctadiene)(1,5-diazabicyclo[4.3.0]non-5-ene)rhodium(I) dioxane solvate, (II), [RhCl(C<sub>8</sub>H<sub>12</sub>)(C<sub>7</sub>H<sub>12</sub>N<sub>2</sub>)] $\cdot\frac{1}{2}$ C<sub>4</sub>H<sub>8</sub>O<sub>2</sub>,  $M_r = 414.8$ , monoclinic,  $P2_1/c$ ,  $a = 7.231$  (1),  $b = 15.870$  (3),  $c = 15.918$  (3) Å,  $\beta = 100.40$  (1)°,  $V = 1796.7$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.533 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 1.09 \text{ mm}^{-1}$ ,  $F(000) = 856$ ,  $T = 296$  (1) K, final  $R = 0.044$ ,  $wR = 0.048$  for 3203 unique observed intensities. In the two complexes each Rh atom is coordinated by one Cl, two double bonds of cod and one N of the corresponding ligand realizing a slightly distorted square planar geometry. Substitution of dbu vs dbn leaves the rhodium–

ligand bond lengths nearly unchanged, the deviation from planarity, however, being significantly greater for the dbu complex.

**Experimental.** Compounds (I) and (II) were prepared by reaction of [Rh(cod)Cl]<sub>2</sub> with dbu and dbn respectively (ratio 1:2) in dioxane solution at 298 K followed by precipitation by adding *n*-pentane. The complexes were recrystallized from dioxane/pentane. (I): Yellow crystal, 0.20 × 0.20 × 0.38 mm; Siemens *R3m/V* diffractometer, Mo *K* $\alpha$  radiation, graphite monochromator; lattice parameters refined from 25 reflections,  $15 \leq 2\theta \leq 30^\circ$ ;  $\omega$ - $2\theta$  scan; 2222 intensities collected,  $3 \leq 2\theta \leq 55^\circ$ ,  $-9 \leq h \leq 9$ ,  $0 \leq k \leq 14$ ,  $0 \leq l \leq 13$ ; three standards recorded every 400 measurements showed only random deviations; Lp correction; empirical absorption correction via  $\psi$  scans, minimum/maximum transmission 0.596/0.643; 2111 unique intensities,  $R_{\text{int}} = 0.019$ , 1912 observed with  $F > 4\sigma(F)$ . Structure solved by Patterson and Fourier methods; full-matrix least-squares refinement based