eters. The positional parameters of the H atoms were allowed to refine freely. 6197 reflections were unique (R_{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 $\Delta/\sigma = 0.001$, $\Delta\rho_{max} = 0.43$, $\Delta\rho_{min} = -0.53$ e Å^{-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.

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* 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$_2$BH$_2$)(PMe$_3$)$_3$] (Slater, Wilkinson, Thornton-Pett & Hursthouse, 1984) and 1.50 (4) Å in [(P–N)(η^2-H$_2$)Ru(μ-Cl)$_2$(μ-H)Ru(H)(PPh$_3$)$_2$], where P–N = [Fe{η-C$_5$H$_3$(CHMeNMe$_2$)P(^{i}Pr)$_2$-1,2}-(η^5-C$_5$H$_5$)] (Hampton, Cullen, James & Charland, 1988).

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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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Abstract. Chloro(η^4-1,5-cyclooctadiene)(1,8-diazabicyclo[5.4.0]undec-7-ene)rhodium(I), (I), [RhCl(C$_8$H$_{12}$)(C$_9$H$_{16}$N$_2$)], 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^3, $Z = 2$, $D_x = 1.521$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.12$ mm^{-1}, $F(000) = 412$, $T = 296$ (1) K, final $R = 0.026$, $wR = 0.029$ for 1912 unique observed intensities. Chloro(η^4-1,5-cyclooctadiene)(1,5-diazabicyclo[4.3.0]non-5-ene)rhodium(I) dioxane solvate, (II), [RhCl(C$_8$H$_{12}$)(C$_7$H$_{12}$N$_2$)].$\frac{1}{2}$C$_4$H$_8$O$_2$, 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^3, $Z = 4$, $D_x = 1.533$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.09$ 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]$_2$ 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, $15 \leq 2\theta \leq 30°$; ω–2θ scan; 2222 intensities collected, $3 \leq 2\theta \leq 55°$, $-9 \leq h \leq 9$, $0 \leq k \leq 14$, $0 \leq l \leq 13$; three standards recorded every 400 measurements showed only random deviations; Lp correction; empirical absorption correction *via* ψ scans, minimum/maximum transmission 0.596/0.643; 2111 unique intensities, $R_{int} = 0.019$, 1912 observed with $F > 4\sigma(F)$. Structure solved by Patterson and Fourier methods; full-matrix least-squares refinement based

Table 1. Atomic coordinates (× 10⁴) and equivalent isotropic displacement parameters (Å² × 10³) for (I) and (II)

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

	x	y	z	U_{eq}
(I)				
Rh(1)	968 (1)	0	3090 (1)	30 (1)
Cl(1)	2059 (2)	−1762 (1)	2389 (1)	63 (1)
N(1)	−718 (5)	241 (3)	1176 (3)	31 (2)
N(2)	−3060 (5)	−269 (3)	−713 (3)	36 (2)
C(1)	−2300 (6)	−281 (3)	590 (4)	31 (2)
C(2)	−3318 (7)	−949 (5)	1398 (5)	44 (3)
C(3)	−3564 (9)	−2248 (5)	1052 (6)	58 (4)
C(4)	−5179 (17)	−2550 (9)	−137 (11)	72 (5)
C(5)	−5118 (10)	−2012 (6)	−1400 (6)	65 (4)
C(6)	−4964 (7)	−697 (5)	−1349 (5)	46 (3)
C(7)	−2012 (7)	113 (8)	−1604 (4)	49 (3)
C(8)	44 (7)	211 (7)	−911 (4)	57 (3)
C(9)	265 (7)	837 (5)	360 (4)	44 (3)
C(10)	992 (8)	127 (9)	5940 (4)	54 (3)
C(11)	−811 (8)	718 (6)	5151 (5)	62 (4)
C(12)	−724 (7)	1119 (5)	3827 (5)	43 (3)
C(13)	681 (8)	1769 (5)	3562 (5)	45 (3)
C(14)	2407 (9)	2231 (5)	4598 (6)	61 (4)
C(15)	4038 (8)	1387 (6)	4916 (6)	65 (4)
C(16)	3454 (6)	128 (8)	4685 (4)	45 (3)
C(17)	2085 (7)	−455 (5)	5106 (4)	41 (3)
(II)				
Rh(1)	2184 (1)	1629 (1)	1293 (1)	46 (1)
Cl(1)	3691 (2)	2042 (1)	2683 (1)	70 (1)
N(1)	620 (6)	762 (3)	1880 (3)	62 (3)
N(2)	163 (6)	−497 (3)	2546 (3)	61 (2)
C(1)	−981 (9)	1096 (4)	2238 (5)	92 (5)
C(2A)	−1435 (27)	759 (9)	2921 (12)	96 (13)*
C(2B)	−2346 (19)	530 (9)	2406 (10)	71 (9)*
C(3)	−1464 (9)	−210 (4)	2912 (4)	88 (4)
C(4)	1022 (10)	−1315 (4)	2697 (4)	78 (4)
C(5)	2657 (12)	−1299 (4)	2276 (5)	97 (5)
C(6)	2671 (8)	−470 (3)	1837 (4)	69 (3)
C(7)	1048 (7)	3 (3)	2079 (3)	51 (3)
C(8)	1488 (8)	1065 (4)	81 (3)	65 (3)
C(9)	88 (8)	1627 (4)	202 (4)	70 (4)
C(10)	−262 (9)	2503 (5)	−137 (5)	96 (5)
C(11)	1461 (9)	3045 (4)	−7 (4)	79 (4)
C(12)	2902 (7)	2777 (3)	756 (3)	55 (3)
C(13)	4308 (7)	2198 (3)	731 (3)	55 (3)
C(14)	4554 (9)	1717 (4)	−61 (4)	80 (4)
C(15)	2897 (10)	1252 (5)	−486 (4)	89 (4)
O(20)	3135 (7)	5138 (4)	−372 (4)	102 (4)
C(21)	4382 (12)	4771 (5)	−824 (4)	90 (5)
C(22)	4149 (11)	5696 (4)	252 (4)	85 (5)

* Disordered, s.o.f. = 0.5 .

Table 2. Selected bond lengths (Å) and angles (°) in (I) and (II)

$X(1)$ and $X(2)$ are the centres of C(12)—C(13) and C(16)—C(17) bonds, respectively, in (I); $Y(1)$ and $Y(2)$ are the centres of the C(2)—C(9) and C(12)—C(13) bonds, respectively, in (II).

(I)

Rh(1)—Cl(1)	2.371 (2)	Rh(1)—N(1)	2.097 (3)
Rh(1)—X(1)	1.989 (5)	Rh(1)—X(2)	2.026 (5)
Cl(1)—Rh(1)—N(1)	87.9 (1)	Cl(1)—Rh(1)—X(1)	177.9 (2)
Cl(1)—Rh(1)—X(2)	91.9 (2)	X(1)—Rh(1)—X(2)	88.2 (2)
X(1)—Rh(1)—N(1)	92.2 (2)	X(2)—Rh(1)—N(1)	175.3 (2)

(II)

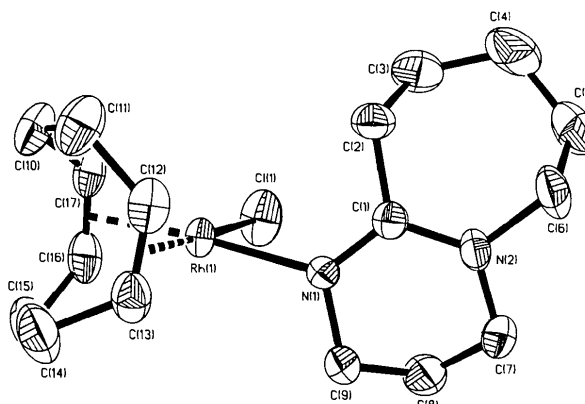|			
Rh(1)—Cl(1)	2.375 (1)	Rh(1)—N(1)	2.103 (4)
Rh(1)—Y(1)	1.978 (5)	Rh(1)—Y(2)	2.001 (5)
Cl(1)—Rh(1)—N(1)	87.7 (1)	Cl(1)—Rh(1)—Y(1)	175.9 (2)
Cl(1)—Rh(1)—Y(2)	91.9 (1)	Y(1)—Rh(1)—Y(2)	88.0 (2)
Y(1)—Rh(1)—N(1)	92.2 (2)	Y(2)—Rh(1)—N(1)	177.8 (2)

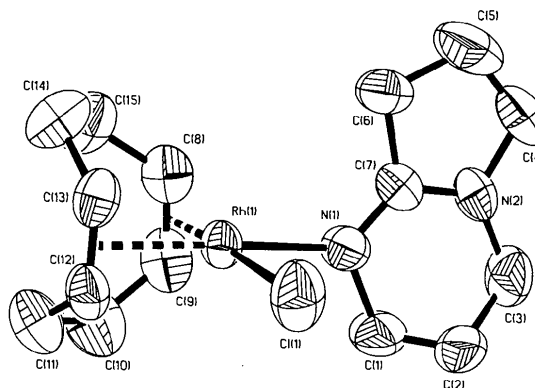
Fig. 1. Molecular structure of complex (I).

Fig. 2. Molecular structure of complex (II).

on F; 189 parameters with anisotropic refinement for all atoms except H atoms which were fixed at idealized positions with common isotropic displacement parameters, $U_{iso} = 0.08$ Å²; y coordinate of Rh was held invariant; although no Friedel equivalents have been measured in general, refinement of Rogers (1981) η parameter indicated that the given configuration is the correct one; refinement converged at $R = 0.026$, $wR = 0.029$, $1/w = \sigma^2(F) + 0.00034F^2$, $S = 0.996$, maximum $(\Delta/\sigma) = 0.001$, minimum/maximum height in final ΔF map −0.43/0.38 e Å⁻³. (II): orange–yellow crystal, 0.40 × 0.51 × 0.65 mm; diffractometer as above; lattice parameters from 40 reflections, $15 \le 2\theta \le 36°$; 4449 intensities collected, data collection as above; data corrections as above; minimum/maximum transmission 0.281/0.356; 4153 unique intensities, $R_{int} = 0.013$, 3203 with $F > 4\sigma(F)$. Structure solution and refinement as above; 208 parameters; H atoms as above; one half-enclosed dioxane solvent molecule per asymmetric unit; disordered C(2) atom of dbn ring refined with split model, two sites with occupation factor 0.5 each; refinement converged at $R = 0.044$, $wR = 0.048$, $1/w = \sigma^2(F) + 0.00039F^2$, $S = 2.068$, maximum $(\Delta/\sigma) = 0.001$, minimum/maximum height in final ΔF map −0.82/1.82 e Å⁻³ near the Rh atom position. Scat-

tering factors and structure refinement for both structures: *SHELXTL-Plus* (Sheldrick, 1990), other programs: *PARST* (Nardelli, 1983). Atomic parameters for (I) and (II) are given in Table 1, selected bond lengths and angles in Table 2.* Figs. 1 and 2 show the molecular structures.

Related literature. Only a few other structures of Rh complexes containing one neutral monodentate

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* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54986 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE0084]

ligand besides cod and Cl have been reported: [{RhCl(cod)}$_2$(μ-C$_4$H$_4$N$_2$)] (Halesha, Reddy, Sudhakar Rao & Manohar, 1983), [RhCl(cod)P(*p*-C$_6$H$_4$F)$_3$] (Iglesias, del Pino, Corma, Garcia-Blanco & Martinez Carrera, 1987).

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Structure of Chlorodioxotetrakis(tetrahydrofuran)uranium(VI) Pentachloro(tetrahydrofuran)uranate(IV)

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Abstract. [UO$_2$Cl(C$_4$H$_8$O)$_4$][UCl$_5$(C$_4$H$_8$O)], M_r = 1081.3, triclinic, $P\bar{1}$, a = 9.272 (2), b = 13.031 (3), c = 15.240 (3) Å, α = 80.04 (3), β = 79.15 (3), γ = 73.46 (3)°, V = 1719.5 (6) Å^3, Z = 2, D_x = 2.088 Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ = 9.422 mm^{-1}, $F(000)$ = 1004, T = 293 K, R = 0.0639, wR = 0.0588 for 2544 unique observed [$F > 4.0\sigma(F)$] reflections. [UO$_2$Cl(OC$_4$H$_8$)$_4$]$^+$ is pentagonal biprismatic with the chloride and the four tetrahydrofuran ligands forming the equatorial plane. The mean U—O distances are 1.72 (1) (uranyl O atoms) and 2.43 (2) Å (thf O atoms) and U—Cl 2.637 (8) Å. Within the octahedral [UCl$_5$(OC$_4$H$_8$)]$^-$ the U—O distance is 2.389 (17) Å and the average U—Cl distance is 2.57 (1) Å.

Experimental. During an attempt to purify a compound which forms on the reaction between UCl$_4$ and (CH$_3$)$_3$SiN=S=NSi(CH$_3$)$_3$, green [UO$_2$Cl(OC$_4$H$_8$)$_4$]$^+$.[UCl$_5$(OC$_4$H$_8$)]$^-$ crystallized overnight from tetrahydrofuran (thf) at 250 K. A green plate of approximate dimensions 0.3 × 0.4 × 0.6 mm was

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used for data collection on a Siemens–Stoe AED2 diffractometer, with graphite-monochromated Mo $K\alpha$ radiation and profile-fitted 2θ–ω scans involving variable scan width and speed (Clegg, 1981). Lattice parameters were determined from a least-squares fit of 36 reflections with $20 < 2\theta < 25°$. 4513 reflections were measured in the range 2θ to 45° and $-9 \le h \le 9$, $-13 \le k \le 14$, and $-5 \le l \le 16$. *XEMP* (Sheldrick, 1985) was used to apply a semiempirical absorption correction to the 4469 unique data in which a minimum to maximum transmission ratio of 0.3824/0.4933 was observed. Three standard reflections measured every 90 min showed about 30% decrease in intensity; an appropriate correction was applied.

The structure was solved with Patterson methods using *SHELXS*86 (Sheldrick, 1985) and 2544 reflections with $F > 4.0\sigma(F)$ were refined with 316 parameters using *SHELX*76 (Sheldrick, 1976); S = 1.6092, R = 0.0639, wR = 0.0588, where $w^{-1} = \sigma^2(F) + 0.0004F^2$. All non-H atoms were refined with anisotropic displacement coefficients and H atoms were included using a riding model with C—H = 0.96 Å and U(H) = 0.08 Å^2. In the final cycle the largest Δ/σ was 0.049 and the maximum and minimum $\Delta\rho$ were