based on counting statistics. $(\Delta/\sigma)_{max} = 0{\cdot}78$. In final difference electron density synthesis max. height less than 2·1 e Å^{-3}. All calculations were performed on a VAX 785 computer using *SDP* (Frenz, 1978), the scattering factors were taken from Cromer & Waber (1974). *ORTEP*II (Johnson, 1976) was used to produce the molecular configuration shown in Fig. 1. The atom coordinates and equivalent isotropic thermal parameters are listed in Table 1, important bond lengths and bond angles are given in Table 2.*

Related literature. A derivative of the title compound, $[(W_3CuS_4)(I)\{S_2P(OC_2H_5)_2\}_3(\mu_2\text{-}OOCCH_3)(C_5H_5N)]$, has been reported (Zhan, Zheng, Wu & Lu, 1989).

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* Lists of H-atom coordinates, full bond lengths and angles, structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51891 (44 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Rhodium–Allene Complex, Chloro(1,2-cyclononadiene)bis(triphenylphosphine)rhodium(I)

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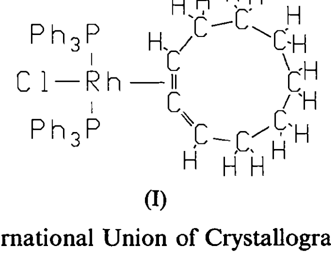
Abstract. $[RhCl(C_9H_{14})(C_{18}H_{15}P)_2]$, $M_r = 785{\cdot}2$, monoclinic, $P2_1/n$, $a = 12{\cdot}066\ (5)$, $b = 17{\cdot}280\ (8)$, $c = 18{\cdot}330\ (8)$ Å, $\beta = 97{\cdot}59\ (5)^\circ$, $V = 3788\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}38$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 6{\cdot}27$ cm^{-1}, $F(000) = 1624$, $T = 293$ K, $R = 0{\cdot}059$ for 3287 reflections with $F_o^2 > 3\sigma(F_o^2)$. The Rh atom is bonded to the cyclic allene by one of the two adjacent C═C units. The coordinated C═C group makes a 74 (2)° angle with the Cl—P—P—Rh plane and is lengthened to 1·45 (4) Å while the other C═C bond is 1·26 (1) Å. The C═C═C bond angle is 146 (2)°, and the Rh—C distances are 2·15 (2) and 2·041 (9) Å where the shorter distance is to the central C of the allene unit.

Experimental. Title compound (I) obtained by the reaction of $[RhCl(PPh_3)_3]$ with 1,2-cyclononadiene in benzene. Data crystal obtained by slow diffusion of *n*-hexane into a benzene solution. Orange crystal (0·2 × 0·2 × 0·3 mm) mounted under argon in a capillary tube. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using θ–2θ scans of 4–16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $28 < 2\theta < 32^\circ$. Absorption correction based on ψ scans varied from 0·94 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·59 Å^{-1}, $0 < h < 14$, $0 < k < 20$, $-21 < l < 21$. Three standard reflections $(4,\bar{4},\overline{12}\ \ \bar{4},\bar{6},10,\ 2,2,12)$ indicated no crystal decomposition over 64 h



(I)

Table 1. *Fractional coordinates and isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Rh	0·90887 (6)	0·01860 (4)	0·26656 (4)	3·18 (1)
Cl	0·8476 (2)	−0·0745 (1)	0·1761 (1)	4·56 (6)
P(1)	1·0896 (2)	−0·0214 (2)	0·2550 (1)	3·47 (5)
P(2)	0·7225 (2)	0·0305 (1)	0·2860 (1)	3·37 (5)
C(1)†	0·922 (1)	0·137 (1)	0·2991 (9)	3·0 (3)*
C(1′)†	0·987 (1)	0·128 (1)	0·2992 (9)	2·9 (3)*
C(2)	0·9613 (8)	0·0819 (5)	0·3587 (5)	4·3 (2)
C(3)	0·9842 (9)	0.0700 (6)	0·4271 (5)	4·7 (2)
C(4)	1·023 (1)	0·1290 (7)	0·4867 (6)	7·3 (4)
C(5)†	1·012 (2)	0·210 (1)	0·462 (1)	4·4 (4)*
C(5′)†	1·064 (2)	0·208 (1)	0·451 (1)	5·3 (5)*
C(6)†	1·113 (2)	0·227 (1)	0·419 (1)	4·8 (4)*
C(6′)†	0·961 (2)	0·250 (1)	0·434 (1)	4·9 (4)*
C(7)†	1·096 (2)	0·288 (1)	0·364 (1)	5·8 (5)*
C(7′)†	0·960 (2)	0·305 (1)	0·372 (1)	7·0 (6)*
C(8)	0·9952 (9)	0·2736 (6)	0·2972 (5)	4·8 (2)
C(9)†	0·998 (1)	0·187 (1)	0·266 (1)	4·0 (4)*
C(9′)†	0·924 (2)	0·201(1)	0·273 (1)	5·0 (4)*
C(10)	1·1187 (7)	−0·0200 (6)	0·1586 (5)	4·0 (2)
C(11)	1·0982 (9)	0·0477 (6)	0·1198 (5)	5·1 (2)
C(12)	1·1234 (9)	0·0474 (7)	0·0472 (6)	6·3 (3)
C(13)	1·1587 (9)	−0·0150 (8)	0·0140 (6)	6·7 (3)
C(14)	1·173 (1)	−0·0842 (9)	0·0507 (6)	7·8 (4)
C(15)	1·150 (1)	−0·0932 (7)	0·1260 (6)	6·2 (3)
C(16)	1·1246 (7)	−0·1200 (5)	0·2902 (5)	3·7 (2)
C(17)	1·0406 (8)	−0·1657 (6)	0·3138 (5)	4·5 (2)
C(18)	1·0687 (9)	−0·2387 (6)	0·3449 (5)	5·0 (3)
C(19)	1·1746 (9)	−0·2631 (6)	0·3522 (5)	5·1 (3)
C(20)	1·2554 (9)	−0·2199 (7)	0·3293 (6)	5·4 (3)
C(21)	1·2322 (8)	−0·1454 (6)	0·2970 (5)	4·4 (2)
C(22)	1·2076 (7)	0·0294 (5)	0·3057 (4)	3·4 (2)
C(23)	1·2723 (8)	0·0841 (6)	0·2736 (5)	4·6 (2)
C(24)	1·3521 (8)	0·1273 (6)	0·3189 (6)	4·9 (2)
C(25)	1·3726 (8)	0·1149 (6)	0·3930 (5)	4·7 (2)
C(26)	1·3103 (9)	0·0613 (6)	0·4242 (5)	5·0 (2)
C(27)	1·2270 (8)	0·0207 (6)	0·3836 (5)	4·5 (2)
C(28)	0·6854 (6)	0·1091 (5)	0·3467 (5)	3·5 (2)
C(29)	0·6340 (8)	0·1758 (5)	0·3178 (6)	5·1 (2)
C(30)	0·6168 (8)	0·2340 (6)	0·3681 (7)	5·9 (3)
C(31)	0·648 (1)	0·2246 (7)	0·4438 (7)	7·0 (3)
C(32)	0·6946 (9)	0·1585 (7)	0·4682 (5)	6·0 (3)
C(33)	0·7139 (8)	0·0967 (6)	0·4240 (5)	4·6 (2)
C(34)	0·6698 (8)	−0·0536 (5)	0·3319 (5)	3·6 (2)
C(35)	0·5682 (8)	−0·0490 (6)	0·3633 (5)	4·3 (2)
C(36)	0·5360 (9)	−0·1129 (7)	0·3985 (5)	5·7 (3)
C(37)	0·597 (1)	−0·1818 (6)	0·4051 (5)	5·8 (3)
C(38)	0·692 (1)	−0·1846 (6)	0·3756 (6)	6·2 (3)
C(39)	0·7301 (8)	−0·1229 (6)	0·3374 (6)	5·1 (3)
C(40)	0·6249 (8)	0·0435 (5)	0·2018 (4)	3·7 (2)
C(41)	0·5165 (8)	0·0161 (6)	0·1906 (5)	4·8 (2)
C(42)	0·449 (1)	0·0305 (6)	0·1241 (6)	5·9 (3)
C(43)	0·488 (1)	0·0711 (6)	0·0675 (5)	5·4 (3)
C(44)	0·5977 (9)	0·0989 (6)	0·0782 (5)	5·3 (3)
C(45)	0·6704 (9)	0·0861 (6)	0·1436 (5)	5·1 (2)

* Isotropic temperature factor, B_{iso}.

† Occupancy factor was fixed at 0·5 (see text).

of data collection. 7056 reflections measured, 6684 unique ($R_{int} = 0·03$), 3288 reflections with $I < 3\sigma(I)$ where $\sigma^2(I) = \sigma_{cs}^2(I) + (0·08I)^2$; $\sigma_{cs}(I)$ is the standard deviation of I based on counting statistics. The 101 reflection was later removed because of evidence of extinction. Structure solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(F_o - F_c)^2$. H atoms were constrained to idealized positions (C—H = 0·95 Å) with fixed isotropic B values of 1·2 times the B value of the attached C atoms. Five of the C atoms of the cyclononadiene ring are disordered; one disorder component very nearly corresponds to the enantiomer of the other. The dis-

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

Rh—Cl	2·358 (2)	C(1)—C(2)	1·48 (2)
Rh—P(1)	2·323 (2)	C(1)—C(9)	1·45 (2)
Rh—P(2)	2·333 (2)	C(1)—C(1′)	0·79 (2)
Rh—C(1)	2·13 (2)	C(2)—C(3)	1·264 (12)
Rh—C(2)	2·041 (9)	C(1′)—C(9′)	1·51 (3)
Rh—C(1′)	2·16 (2)	C(1′)—C(2)	1·41 (2)
P(1)—C(10)	1·847 (8)	C(3)—C(4)	1·522 (13)
P(1)—C(16)	1·851 (9)	C(5)—C(5′)	0·69 (3)
P(1)—C(22)	1·820 (8)	C(6)—C(6′)	1·93 (3)
P(2)—C(28)	1·847 (8)	C(7)—C(7′)	1·70 (3)
P(2)—C(34)	1·834 (8)	C(9)—C(9′)	0·94 (2)
P(2)—C(40)	1·827 (8)		

Cl—Rh—P(1)	86·78 (8)	C(10)—P(1)—C(16)	106·4 (4)
Cl—Rh—P(2)	87·05 (8)	C(10)—P(1)—C(22)	104·4 (4)
Cl—Rh—C(1)	149·0 (4)	C(16)—P(1)—C(22)	97·9 (4)
Cl—Rh—C(2)	169·0 (3)	Rh—P(2)—C(28)	118·2 (3)
Cl—Rh—C(1′)	151·2 (4)	Rh—P(2)—C(34)	113·6 (3)
P(1)—Rh—P(2)	167·34 (9)	Rh—P(2)—C(40)	114·1 (3)
P(1)—Rh—C(1)	105·9 (5)	C(28)—P(2)—C(34)	100·4 (4)
P(1)—Rh—C(2)	92·5 (3)	C(28)—P(2)—C(40)	103·8 (4)
P(1)—Rh—C(1′)	84·6 (5)	C(34)—P(2)—C(40)	105·1 (4)
P(2)—Rh—C(1)	84·9 (5)	C(2)—C(1)—C(9)	122· (2)
P(2)—Rh—C(2)	91·5 (3)	C(1)—C(2)—C(3)	148· (1)
P(2)—Rh—C(1′)	105·9 (5)	C(3)—C(2)—C(1′)	144· (1)
Rh—P(1)—C(10)	112·5 (3)	C(2)—C(1′)—C(9′)	124· (2)
Rh—P(1)—C(16)	114·5 (3)	C(2)—C(3)—C(4)	127·6 (9)
Rh—P(1)—C(22)	119·4 (3)		

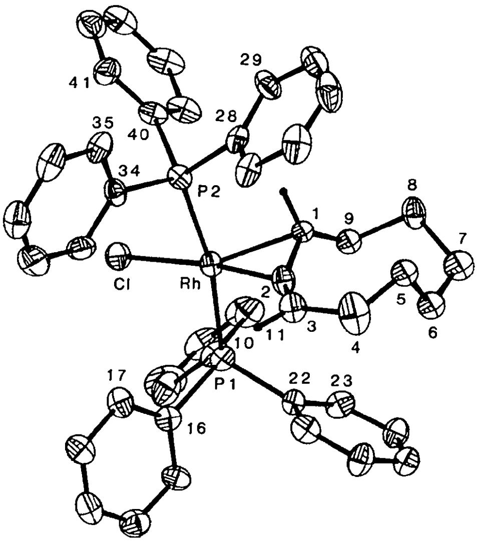


Fig. 1. *ORTEP* (Johnson, 1976) drawing and numbering scheme. The ellipsoids are drawn at the 30% probability surface; all H atoms except H1 and H3 have been omitted and H atoms have been given arbitrary radii for figure clarity. This figure shows the disorder component given by the atoms with unprimed labels in Table 1.

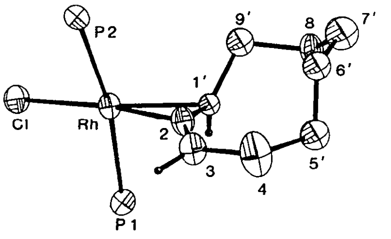


Fig. 2. *ORTEP* (Johnson, 1976) drawing for the disorder component which uses the five C atoms with primed labels. The orientation of the drawing is identical with that of Fig. 1; the phenyl rings have been omitted for figure clarity.

ordered atoms were refined with 50% occupancy factors and isotropic thermal parameters while the other non-H atoms were refined anisotropically for a total of 437 parameters, $R = 0·059$, $wR = 0·073$, GOF = 1·3, where non-Poisson $w^{-1} = [\sigma^2(I) + (0·08I)^2]/4F^2$. Final $(\Delta/\sigma)_{\max} < 0·38$, $\Delta\rho_{\max} = 1·19\ (11)$ (all six peaks above 0·64 were within 0·99 Å of the Rh position) and $\Delta\rho_{\min} = -0·48\ (11)$ e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*.* Table 1 gives the atomic coordinates and Table 2 selected bond distances and angles. Figs. 1 and 2 show the molecule with the numbering scheme.

Related literature. The structure of this diene complex can be compared most directly with that of $[RhI(PPh_3)_2(C_3H_4)]$ (Kashiwagi, Yasuoka, Kasai & Kukudo, 1969). Other Rh–diene complexes have been reported by Hewitt & DeBoer (1971) and by Racanelli, Pantini, Immirzi, Allegra & Porri (1969). Transition-metal–allene complexes are reviewed by Shaw & Stringer (1973).

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* Lists of distances and angles in the phenyl groups and in the saturated portion of the cyclononadiene ring, anisotropic temperature factors, structure factors, and H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51893 (50 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Hexakis(acetonitrile)ruthenium(II) *p*-Toluenesulfonate Dihydrate at 100 K

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Abstract. $[Ru(C_2H_3N)_6][C_7H_7O_3S]_2.2H_2O$, $M_r = 725·8$, triclinic, $P\bar{1}$, $a = 8·274\ (3)$, $b = 8·957\ (3)$, $c = 11·538\ (7)$ Å, $\alpha = 107·92\ (4)$, $\beta = 89·47\ (3)$, $\gamma = 92·44\ (3)$°, $V = 812·9$ Å^3, $Z = 1$, $D_m(298\ K) = 1·430\ (1)$, $D_x(100\ K) = 1·482$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 6·47$ cm^{-1}, $F(000) = 374$, $R = 0·019$ for 3316 reflections with $I > 3\sigma(I)$ measured at 100 K. The Ru environment is close to octahedral with average Ru—N = 2·029 (2), C—N = 1·139 (1) and C—C = 1·456 (2) Å. The N—Ru—N angles are between 89·19 (3) and 90·87 (3)°.

Experimental. The Ru(acetonitrile)$_6^{2+}$ complex was prepared as previously described (Rapaport, Helm, Merbach, Bernhard & Ludi, 1988). Density at 298 K by flotation. Crystals grown by cooling an acetone–water solution from 313 to 277 K. A section (0·29 × 0·26 × 0·22 mm) cut from a prismatic crystal was sealed in a Lindemann capillary and measured on an Enraf–Nonius CAD-4 diffractometer equipped with a liquid-nitrogen attachment. Unit-cell data from 22 reflections with $13 < \theta < 19$°. Data collection [ω scan, $(1·6 + 0·35\tan\theta)$° (20–27°), $(1·7 + 0·35\tan\theta)$°

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