

Fig. 1. Molecular structure and labelling scheme for (1).

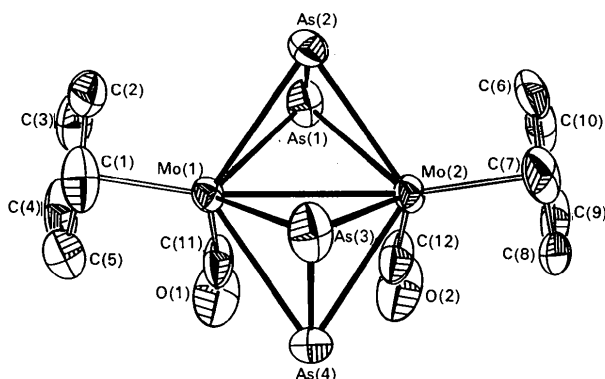


Fig. 2. Molecular structure and labelling scheme for (2).

on 181 parameters; all non-H atoms anisotropic, H atoms idealized and updated (C—H = 0.96 Å,  $U = 1.2U$  of attached C).  $R_F = 4.77\%$ ,  $wR_F = 5.03\%$ ,  $S = 1.276$ ,  $w^{-1} = \sigma^2(F_o) + gF_o^2$ ,  $g = 0.001$ ;  $(\Delta/\sigma)_{\max} = 0.043$ ;  $\Delta\rho_{\max} = 1.009$ ,  $\Delta\rho_{\min} = -1.208 \text{ e \AA}^{-3}$ ; atomic scattering factors from *International Tables for X-ray*

*Crystallography* (1974); *SHELXTL* computer program (Sheldrick, 1985).

Atomic coordinates and equivalent isotropic thermal parameters for (1) and (2) are given in Tables 1 and 2 respectively. Bond lengths and angles for (1) are given in Table 3. Bond lengths and angles for (2) are given in Table 4. Figs. 1 and 2 show the labelled molecular structures for (1) and (2) respectively.\*

**Related literature.** (1) is an analogue of a structure previously reported by Foley, Rheingold & Sullivan (1982). (2) is an analogue of a structure previously reported by DiMaio & Rheingold (1987). Three similar Mo—As clusters have been reported by Scherer, Sitzmann & Wolmershauser (1986).

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\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters, and figures showing unit-cell packing have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52328 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of [(1,2,3- $\eta$ )-Cyclotrideceny]bis(triphenylphosphine)rhodium Benzene Solvate

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**Abstract.** [Rh(C<sub>13</sub>H<sub>23</sub>)(P(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>)<sub>2</sub>].0.5C<sub>6</sub>H<sub>6</sub>,  $M_r = 845.9$ , triclinic,  $P\bar{1}$ ,  $a = 12.260$  (6),  $b = 13.05$  (1),  $c = 15.91$  (1) Å,  $\alpha = 71.30$  (6),  $\beta = 76.24$  (6),  $\gamma = 67.71$  (5)°,  $V = 2211$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.27 \text{ g cm}^{-3}$ ,

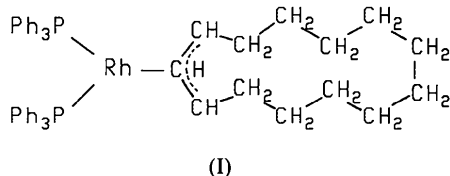
$\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu = 4.8 \text{ cm}^{-1}$ ,  $F(000) = 886$ ,  $T = 293 \text{ K}$ ,  $R = 0.043$  for 6030 reflections with  $F_o^2 > 3\sigma(F_o^2)$ . The Rh atom is  $\eta^3$ -bonded to the allyl portion of the 13-carbon-ring ligand. The ring atoms

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flanking the allyl unit are in the *trans* configuration. In the allyl sequence the bond distances are 1.380 (5) and 1.411 (5) Å; the bond angles of these three atoms are 123.9 to 125.7 (4)° and their distances to the Rh atom are 2.120 (4), 2.180 (5) and 2.231 (3) Å.

**Experimental.** Title compound (I) obtained by the reaction of bis(triphenylphosphine)rhodium hydride with 1,2-cyclotridecadiene. Data crystal obtained by slow evaporation of a benzene solution. Orange crystal approximately 0.3 × 0.3 × 0.3 mm mounted under argon in a capillary tube. Intensities measured with an Enraf-Nonius CAD-4 diffractometer using  $\omega$ - $2\theta$  scans of 4–16° min<sup>-1</sup> in  $\theta$ . Unit cell determined from least-squares analysis of angle data for 25 reflections with  $26 < 2\theta < 28^\circ$ . Absorption correction based on  $\psi$  scans varied from 0.95 to 1.00. Data collected to  $(\sin\theta)/\lambda$  of 0.59 Å<sup>-1</sup>,  $-14 \leq h \leq 14$ ,  $-15 \leq k \leq 15$ ,  $0 \leq l \leq 18$ . Variations in the intensities of three standard reflections (373, 255, 559) over 71.9 h of data collection resulted in a correction which ranged from 0.89 to 1.00. 8061 reflections measured, 7758 unique ( $R_{\text{int}} = 0.02$ ), 1728 reflections with  $I < 3\sigma(I)$  where  $\sigma^2(I) = \sigma_{\text{cs}}^2(I) + (0.08I)^2$ ;  $\sigma_{\text{cs}}(I)$  is standard deviation of  $I$  based on counting statistics. Solved by direct methods using MULTAN11/82 (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 8.0 Å<sup>2</sup>. The three C atoms of the benzene solvate (located on an inversion center) were refined isotropically while all other non-H atoms were refined anisotropically for a total of 481 parameters.  $R = 0.043$ ,  $wR = 0.056$ ,  $S = 1.6$ , where non-Poisson  $w^{-1} = [\sigma^2(I) + (0.05I)^2]/4F^2$ . Final  $(\Delta/\sigma)_{\text{max}} < 0.2$ ,  $\Delta\rho_{\text{max}} = 0.63$  (7) and  $\Delta\rho_{\text{min}} = -0.61$  (7) e Å<sup>-3</sup> 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. Fig. 1 shows the molecule with the numbering scheme.



\* Tables of distances and angles in the phenyl groups and in the standard portion of the allyl 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 52315 (59 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates and isotropic or equivalent isotropic thermal parameters

The thermal parameters are of the form  $B = 8\pi^2 U$  and  $B_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$ .

	x	y	z	$B/B_{\text{eq}}$ (Å <sup>2</sup> )
Rh	0.21416 (2)	0.33505 (2)	0.32990 (2)	3.433 (6)
P(1)	0.18748 (7)	0.19508 (7)	0.29147 (6)	3.19 (2)
P(2)	0.31258 (7)	0.41249 (7)	0.20339 (6)	3.43 (2)
C(1)	0.0328 (3)	0.2135 (3)	0.2852 (2)	3.70 (8)
C(2)	0.0032 (3)	0.1388 (3)	0.2574 (3)	4.6 (1)
C(3)	-0.1139 (3)	0.1572 (4)	0.2516 (3)	5.3 (1)
C(4)	-0.2019 (3)	0.2506 (4)	0.2732 (3)	5.4 (1)
C(5)	-0.1739 (4)	0.3263 (4)	0.2988 (3)	5.4 (1)
C(6)	0.0572 (3)	0.3089 (3)	0.3054 (2)	4.3 (1)
C(7)	0.2368 (3)	0.0596 (3)	0.3766 (3)	4.5 (9)
C(8)	0.3481 (4)	0.0297 (4)	0.4005 (3)	5.9 (1)
C(9)	0.3913 (4)	-0.0682 (5)	0.4655 (4)	7.4 (2)
C(10)	0.3250 (5)	-0.1384 (5)	0.5079 (4)	8.0 (2)
C(11)	0.2160 (5)	-0.1107 (5)	0.4862 (4)	8.3 (2)
C(12)	0.1701 (4)	-0.0108 (4)	0.4206 (3)	6.0 (1)
C(13)	0.2562 (3)	0.1489 (3)	0.1875 (2)	3.75 (8)
C(14)	0.3337 (4)	0.0406 (4)	0.1842 (3)	5.7 (1)
C(15)	0.3754 (5)	0.0107 (4)	0.1041 (4)	7.1 (2)
C(16)	0.3395 (4)	0.0875 (4)	0.0260 (3)	6.3 (1)
C(17)	0.2644 (4)	0.1957 (4)	0.0274 (3)	5.4 (1)
C(18)	0.2229 (3)	0.2259 (3)	0.1074 (2)	4.43 (9)
C(19)	0.2288 (3)	0.4988 (3)	0.1091 (2)	3.67 (8)
C(20)	0.2781 (3)	0.5565 (4)	0.0297 (3)	4.9 (1)
C(21)	0.2110 (4)	0.6195 (4)	-0.0399 (3)	5.9 (1)
C(22)	0.0953 (4)	0.6272 (4)	-0.0303 (3)	5.6 (1)
C(23)	0.0431 (3)	0.5754 (3)	0.0488 (3)	5.1 (1)
C(24)	0.1095 (3)	0.5106 (3)	0.1190 (3)	4.19 (9)
C(25)	0.4430 (3)	0.3044 (3)	0.1613 (2)	3.75 (8)
C(26)	0.4769 (4)	0.2975 (3)	0.0739 (3)	4.9 (1)
C(27)	0.5768 (4)	0.2092 (4)	0.0518 (3)	6.3 (1)
C(28)	0.6421 (4)	0.1285 (4)	0.1172 (4)	6.3 (1)
C(29)	0.6090 (4)	0.1342 (4)	0.2038 (3)	5.6 (1)
C(30)	0.5099 (3)	0.2216 (3)	0.2260 (3)	4.6 (1)
C(31)	0.3791 (3)	0.5173 (3)	0.2045 (2)	3.99 (9)
C(32)	0.3036 (4)	0.6196 (3)	0.2217 (3)	5.4 (1)
C(33)	0.3460 (5)	0.7013 (4)	0.2252 (4)	6.8 (1)
C(34)	0.4665 (4)	0.6808 (4)	0.2114 (4)	7.5 (1)
C(35)	0.5420 (4)	0.5816 (4)	0.1935 (4)	7.1 (1)
C(36)	0.4991 (3)	0.4992 (3)	0.1896 (3)	5.2 (1)
C(37)	0.1424 (4)	0.2792 (3)	0.4733 (3)	4.9 (1)
C(38)	0.0340 (4)	0.2443 (4)	0.5125 (3)	6.5 (1)
C(39)	0.0226 (6)	0.1977 (7)	0.6099 (4)	15.6 (2)
C(40)	0.0458 (9)	0.2348 (9)	0.6728 (4)	15.4 (4)
C(41)	0.1446 (8)	0.1775 (7)	0.7206 (6)	13.2 (3)
C(42)	0.173 (1)	0.2429 (7)	0.7657 (6)	17.5 (4)
C(43)	0.2330 (9)	0.3072 (9)	0.7295 (5)	21.3 (3)
C(44)	0.3190 (7)	0.3025 (7)	0.6492 (4)	14.5 (3)
C(45)	0.3154 (7)	0.4120 (6)	0.5903 (4)	14.6 (2)
C(46)	0.3874 (5)	0.4195 (5)	0.5033 (4)	10.5 (2)
C(47)	0.3630 (4)	0.3668 (4)	0.4406 (3)	6.4 (1)
C(48)	0.2416 (4)	0.4267 (3)	0.4117 (3)	5.2 (1)
C(49)	0.1413 (4)	0.3910 (4)	0.4487 (3)	5.4 (1)
C(53)	0.0539 (6)	0.8839 (6)	0.0373 (5)	9.5 (2)*
C(54)	0.0848 (6)	0.9687 (6)	0.0567 (4)	9.4 (2)*
C(55)	-0.0302 (6)	0.9196 (5)	-0.0181 (4)	9.1 (2)*

\* Atoms refined isotropically.

Table 2. Selected bond distances (Å) and angles (°) with e.s.d.'s in parentheses

Rh	P(1)	2.2543 (12)	P(1)	C(13)	1.844 (3)		
Rh	P(2)	2.2347 (8)	P(2)	C(19)	1.829 (3)		
Rh	C(37)	2.231 (4)	P(2)	C(25)	1.839 (3)		
Rh	C(48)	2.179 (6)	P(2)	C(31)	1.845 (5)		
Rh	C(49)	2.119 (5)	C(37)	C(49)	1.380 (7)		
P(1)	C(1)	1.842 (4)	C(48)	C(49)	1.411 (7)		
P(1)	C(7)	1.835 (3)					
P(1)	Rh	P(2)	101.56 (3)	C(47)	C(48)	C(49)	125.7 (4)
P(1)	Rh	C(37)	93.63 (12)	C(37)	C(49)	C(48)	125.3 (4)
P(1)	Rh	C(48)	160.08 (12)	Rh	P(1)	C(1)	116.01 (12)
P(1)	Rh	C(49)	127.92 (14)	Rh	P(1)	C(7)	109.25 (14)
P(2)	Rh	C(37)	163.34 (12)	Rh	P(1)	C(13)	125.61 (12)
P(2)	Rh	C(48)	95.49 (12)	Rh	P(2)	C(19)	117.51 (12)
P(2)	Rh	C(49)	129.96 (14)	Rh	P(2)	C(25)	112.23 (12)
C(38)	C(37)	C(49)	123.9 (4)	Rh	P(2)	C(31)	119.13 (12)

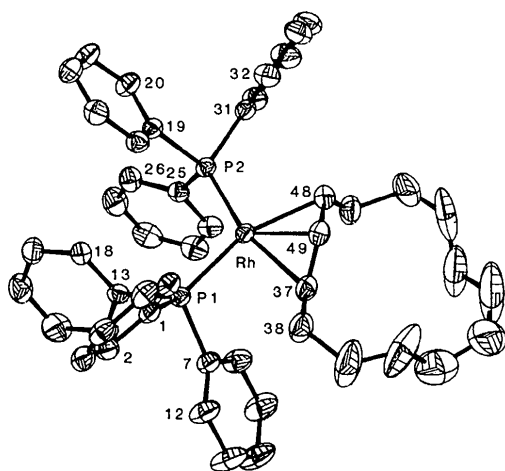


Fig. 1. ORTEP (Johnson, 1976) drawing and numbering scheme. The ellipsoids are drawn at the 30% probability surface.

**Related literature.** The syntheses, reactions and structural aspects of allyl complexes of rhodium have been the subject of numerous reviews; recent surveys include those by Collman, Hegedus, Norton & Finke (1987) and by Hughes (1982). The structure reported here can be compared to that of a cyclooctenyl-rhodium complex (Pickardt & Stuhler, 1980) and

that of a methylallyl-rhodium complex (Hewitt & deBoer, 1968).

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## ( $\eta^2$ -1,3-Di-*tert*-butyl-2,5-dioxo-3-cyclopentene-1-carbonitrile)bis(triphenylphosphine)-platinum(0)–Diethyl Ether

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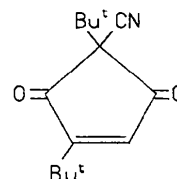
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**Abstract.** [Pt(C<sub>14</sub>H<sub>19</sub>NO<sub>2</sub>){P(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>}<sub>2</sub>].C<sub>4</sub>H<sub>10</sub>O,  $M_r = 1027.1$ , triclinic,  $P\bar{1}$ ,  $a = 11.240$  (4),  $b = 13.748$  (5),  $c = 16.224$  (6) Å,  $\alpha = 76.77$  (3),  $\beta = 79.56$  (3),  $\gamma = 83.09$  (3)°,  $V = 2392$  (12) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.426$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 31.9$  cm<sup>-1</sup>,  $F(000) = 1044$ ,  $T = 296$  K,  $R_F = 4.11\%$  for 5014 observed reflections and 479 parameters. The structure is consistent with a Pt<sup>0</sup> olefin diphosphine complex. The Pt–olefin plane is twisted 11.4° out of the PtP<sub>2</sub> plane by the presence of the bulky *tert*-Bu group on the cyclopentenedione ring. The dihedral angle between the ring plane and the Pt–olefin plane is 95.8°. The two Pt–C distances differ

significantly: the distance to the ring position bearing the *tert*-Bu group is 2.170 (7) Å, while the distance to the unsubstituted C atom is much shorter, 2.121 (8) Å.

**Experimental.** The title compound was obtained by the addition of the ligand (see below) to [Pt(C<sub>2</sub>H<sub>4</sub>)(PPh<sub>3</sub>)<sub>2</sub>] (Macklin, 1988). Yellow crystals



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