Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

Ru(1)—C(1)	2.224 (7)	Ru(1)—C(2)	2.205 (6)
Ru(1)—C(3)	2.213 (7)	Ru(1)—C(4)	2.221 (7)
Ru(1)—C(5)	2.219 (7)	Ru(1)—N(1)	2.070 (4)
Ru(1)—P(1)	2.318 (1)	Ru(1)—P(2)	2.323 (2)
N(1)—C(6)	1.146 (7)	C(6)—S(1)	1.634 (6)
N(1)—Ru(1)—P(1)	94.6 (1)	N(1)—Ru(1)—P(2)	87.1 (1)
P(1)—Ru(1)—P(2)	99.0 (1)	Ru(1)—N(1)—C(6)	168.4 (5)
N(1)—C(6)—S(1)	178.7 (6)		

final refinement (maximum $\Delta/\sigma = 0.005$, mean $\Delta/\sigma = 0.000$). The largest unassigned electron density peaks (0.87, -0.90 e Å^{-3}) were adjacent to the Ru atom.

Fractional atomic coordinates are given in Table 1 and selected bond distances and bond angles in Table 2.* Fig. 1 shows thermal ellipsoids drawn at the 30% probability level for the title compound.

Related literature. Only one previous structural characterization of a Ru thiocyanate-*N* complex has been reported (Herber, Nan, Potenza, Schugar & Bino, 1989). In the complex bis[(2,2′-bipyridyl-*N*,*N*′)thiocyanato-*N*]ruthenium the Ru—N bond length is 2.055 (5) Å, the angle at the ligand N atom is 168.2 (5)°, and the angle N—C—S is 177.5 (6)°.

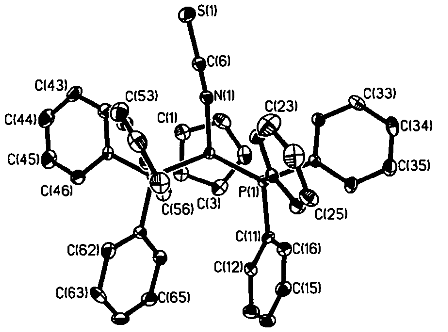

Fig. 1. Plot showing 30% thermal ellipsoids for the title compound.

The angles are consistent with the Ru centre in this complex and in the title compound being a good π donor (Drew, bin-Othman & Nelson, 1976).

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* Lists of structure factors, anisotropic displacement parameters, bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54663 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HE0034]

Acta Cryst. (1992). C**48**, 545–547

Structure of *cyclo*-Tris{dichloro-μ-[methylenebis(dimethylphosphine)-κP:κP]-[methylenebis(dimethylphosphine)-κ^2P]ruthenium(II)}–Ethanol–Toluene (1/1/1)

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Abstract. $[Ru_3(\mu\text{-}C_5H_{14}P_2)_3(C_5H_{14}P_2)_3Cl_6].C_7H_8.C_2H_6O$, $M_r = 1469.9$, triclinic, $P\bar{1}$, $a = 13.208$ (2), $b = 13.316$ (3), $c = 20.828$ (7) Å, $\alpha = 77.04$ (2), $\beta = 72.53$ (2), $\gamma = 72.35$ (2)°, $V = 3294$ (1) Å^3, $Z = 2$, $D_x = 1.48$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 1.13$ mm^{-1}, $F(000) = 1508$, $T = 298$ K, final $R = 0.057$, $wR = 0.048$ for 8141 reflections with $I > 4\sigma(I_o)$ and 614 variable parameters. The 12-membered ring

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0108-2701/92/030545-03$03.00

Table 1. *Fractional coordinates* (× 10^4) *and equivalent isotropic thermal factors* (Å^2 × 10^3)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	3026 (7)	−702 (6)	2919 (4)	45 (2)
P(1)	3084 (2)	685 (2)	2633 (1)	44 (1)
C(2)	1786 (7)	1359 (8)	2373 (5)	66 (3)
C(3)	2838 (9)	1210 (8)	3423 (5)	68 (3)
Ru(1)	4657 (1)	881 (1)	1769 (1)	34 (1)
Cl(1)	4462 (2)	−509 (2)	1258 (1)	47 (1)
P(2)	5983 (2)	1216 (2)	776 (1)	47 (1)
C(4)	6775 (9)	2188 (8)	657 (6)	70 (3)
C(5)	6961 (8)	165 (8)	316 (5)	66 (3)
C(6)	5007 (8)	1858 (7)	256 (4)	57 (3)
P(3)	3794 (2)	2093 (2)	969 (1)	47 (1)
C(7)	3475 (9)	3516 (7)	1004 (6)	71 (3)
C(8)	2696 (8)	1990 (8)	655 (5)	57 (3)
Cl(2)	4796 (2)	2304 (2)	2261 (1)	57 (1)
P(4)	5945 (2)	−234 (2)	2357 (1)	39 (1)
C(9)	7197 (7)	244 (7)	2106 (5)	53 (3)
C(10)	5611 (8)	−290 (7)	3276 (4)	51 (3)
C(11)	6391 (7)	−1644 (6)	2210 (4)	35 (2)
P(5)	7343 (2)	−2780 (2)	2606 (1)	39 (1)
C(12)	8448 (7)	−2266 (7)	2610 (5)	51 (3)
C(13)	6703 (8)	−2976 (7)	3515 (4)	46 (2)
Ru(2)	7884 (1)	−4288 (1)	2053 (1)	36 (1)
Cl(3)	8364 (2)	−3145 (2)	976 (1)	55 (1)
P(6)	9755 (2)	−4734 (2)	2046 (2)	54 (1)
C(14)	10233 (9)	−5535 (8)	2781 (5)	71 (3)
C(15)	10700 (8)	−3905 (8)	1721 (6)	77 (4)
C(16)	10179 (8)	−5618 (8)	1409 (6)	77 (3)
P(7)	8799 (2)	−5748 (2)	1469 (2)	56 (1)
C(17)	8782 (8)	−5781 (8)	605 (5)	69 (3)
C(18)	8775 (10)	−7124 (7)	1878 (6)	94 (4)
Cl(4)	7439 (2)	−5454 (2)	3111 (1)	60 (1)
P(8)	6151 (2)	−4235 (2)	1948 (1)	39 (1)
C(19)	6038 (8)	−5543 (7)	1896 (6)	72 (3)
C(20)	5830 (8)	−3467 (8)	1166 (4)	56 (3)
C(21)	4963 (6)	−3792 (6)	2656 (4)	37 (2)
P(9)	3493 (2)	−3692 (2)	2774 (1)	43 (1)
C(22)	3361 (9)	−4997 (8)	2717 (6)	70 (3)
C(23)	3138 (8)	−2982 (8)	1987 (5)	61 (3)
Ru(3)	2385 (1)	−3094 (1)	3800 (1)	38 (1)
Cl(5)	3826 (2)	−2677 (2)	4110 (1)	51 (1)
P(10)	2784 (3)	−4764 (2)	4453 (2)	65 (1)
C(24)	2275 (11)	−5875 (9)	4429 (7)	103 (5)
C(25)	4137 (10)	−5366 (10)	4595 (8)	138 (6)
C(26)	2007 (14)	−4345 (9)	5249 (6)	156 (6)
P(11)	1373 (2)	−2983 (2)	4933 (1)	55 (1)
C(27)	1536 (10)	−2217 (10)	5492 (5)	85 (4)
C(28)	−111 (9)	−2873 (13)	5211 (6)	115 (5)
Cl(6)	864 (2)	−3463 (2)	3536 (1)	64 (1)
P(12)	1867 (2)	−1282 (2)	3355 (1)	46 (1)
C(29)	1067 (8)	−413 (7)	4012 (5)	64 (3)
C(30)	1032 (8)	−930 (9)	2742 (5)	66 (3)
C(31)	6449 (8)	68 (11)	4817 (4)	129 (6)
C(32)	7346 (8)	−802 (11)	4672 (4)	109 (5)
C(33)	8161 (8)	−728 (11)	4068 (4)	118 (6)
C(34)	8078 (8)	215 (11)	3609 (4)	130 (7)
C(35)	7180 (8)	1085 (11)	3753 (4)	189 (9)
C(36)	6366 (8)	1011 (11)	4357 (4)	136 (7)
C(37)	5389 (14)	1927 (14)	4470 (10)	518 (26)†
O	209 (19)	49 (31)	935 (16)	388 (17)
C(38)	201 (29)	671 (38)	361 (16)	526 (42)
C(39)	18 (13)	879 (16)	−333 (15)	292 (11)

† Isotropic temperature factor.

Table 2. *Selected bond distances* (Å) *and valence angles* (°)

Ru—P (average)	2.345 (3)
Ru—Cl (average)	2.432 (3)
C—P (average)	1.83 (2)
P—Ru—P (binding, average)	97.0 (1)
P—Ru—P (chelating, average)	71.1 (1)
P—C—P (binding, average)	129.5 (6)
P—C—P (chelating, average)	97.0 (6)
Cl—Ru—Cl (average)	177.9 (1)

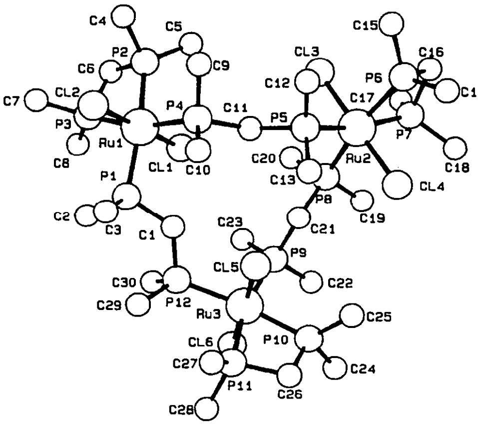

Fig. 1. Perspective view with atomic numbering scheme of non-H atoms of the molecule. The solvent molecules {toluene [C(31) to C(37)] and ethanol [O, C(38) and C(39)]} are not shown.

is formed by three Ru—P—C—P units, and each Ru atom also has a chelating P—C—P ligand. The compound crystallizes with toluene and ethanol molecules included in the asymmetric unit.

Experimental. The title compound was formed by treatment of a suspension of $[RuCl_2(C_8H_{12})]_x$, C_8H_{12} = 1,5-cyclooctadiene, in toluene at 383 K with $Me_2PCH_2PMe_2$ (2.1 mole equivalents). After filtration and concentration of the toluene solution, the product was recovered (60% yield) by addition of ethanol.

Recrystallization of this compound from a toluene/ethanol solution yielded diffraction-quality single crystals: crystal size 0.24 × 0.16 × 0.24 mm; Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation (λ = 0.7107 Å); lattice parameters refined using 25 reflections in the range $7.8 < \theta < 14.4°$; 14 320 reflections for $3 \leq \theta \leq 27°$ in the range $0 \leq h \leq 16$, $-16 \leq k \leq 16$, $-26 \leq l \leq 26$ using $\omega/2\theta$ scans where ω changed as $(0.58 + 0.34\tan\theta)°$ with a variable but maximum speed that corresponded to 5.49° min^{-1}. Three standard reflections (513, 417, 012) measured every 60 min varied by 18.6%, linear decay correction applied. Lorentz and polarization corrections and empirical absorption correction based on the ψ scans of nine reflections were also applied (North, Phillips & Mathews, 1968): transmission factors 0.999–0.846, average 0.925.

Structure solution: Patterson and difference Fourier analysis using *SHELX*76 (Sheldrick, 1976), H atoms in calculated positions (C—H = 1.08 Å, H—C—H = 109.5°). All non-H atoms except the methyl C atom of the toluene of crystallization were refined anisotropically, H atoms were assigned a common isotropic thermal parameter that refined to U_{iso} = 0.127 (4) Å^2, using 8141 reflections with $I > 4\sigma(I_o)$, and $\sigma^{-2}(F)$ weights (Sheldrick, 1976), F magnitudes, $wR = 0.048$ and $R = 0.057$ for 614 parameters. $(\Delta/\sigma)_{\max} = 0.6$, maximum residual electron density 1.13 e Å^{-3} (1 Å from Ru). Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV), and anomalous-dispersion corrections for Ru from Cromer & Liberman (1970). Table 1 gives the atomic parameters, Table 2 gives selected bond distances and valence angles, and Fig. 1 shows the molecular structure and atomic numbering scheme, drawn by *ORTEP* (Johnson, 1965).*

Each octahedral Ru atom is coordinated with both binding and chelating P—C—P ligands, and *trans* bonded Cl atoms. Three atoms [Ru(1), P(5) and Ru(3)] deviate more than 1 Å from the least-squares plane formed by the 12 ring atoms, and all the other atoms are less than 0.5 Å from this plane.

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* Lists of structure factors, bond lengths and angles, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54621 (43 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 (3-Benzylacetylacetonato-κ²*O*,*O*′)carbonyl(triphenylphosphine)-rhodium(I)

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Abstract. $[\mathrm{Rh(C_{12}H_{13}O_2)(CO)\{P(C_6H_5)_3\}}]$, M_r = 582.4, triclinic, $P\bar{1}$, a = 13.004 (2), b = 10.968 (2), c = 11.230 (1) Å, α = 114.42 (1), β = 87.57 (1), γ = 112.54 (1)°, V = 1334.1 (4) Å^3, Z = 2, D_m = 1.46, D_x = 1.450 g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 7.17 cm^{-1}, F(000) = 596, T = 295 K, final R = 0.0266 for 3969 observed reflections. The complex has a square-planar geometry about the Rh atom. The bond distances in the coordination polyhedron are: Rh—P = 2.243 (1), Rh—O(1) = 2.016 (2), Rh—O(2) = 2.048 (2) and Rh—CO = 1.801 (4) Å.

Experimental. The title complex was prepared by mixing equimolar amounts of $\mathrm{PPh_3}$ and $[\mathrm{Rh(bzaa)(CO)_2}]$ (bzaa = 3-benzylacetylacetonato anion) in acetone. Slow evaporation of the acetone solution at 295 K yielded yellow needle-like crystals suitable for crystal structure determination. The density was determined by the flotation method in aqueous sodium perchlorate. Data were collected on an Enraf–Nonius CAD-4F diffractometer with graphite-monochromated Mo $K\alpha$ radiation, using a crystal of size 0.20 × 0.15 × 0.25 mm. The $\omega/2\theta$-scan technique was used with variable scan width $\Delta\omega = (0.60 + 0.35\tan\theta)°$, a scan speed maximum of 5.49° min^{-1} in ω and a maximum scan time of 60 s per reflection. The unit-cell parameters were determined from a least-squares fit of 25 reflections with $15 < \theta < 20°$; the total measuring range was $3 < \theta < 25°$. The intensity data were corrected for Lorentz and polarization effects, and for absorption with the empirical method by North, Phillips & Mathews (1968) with minimum and maximum correction factors of 0.961 and 0.999, respectively. Three control reflections were measured every 3600 s of X-ray exposure time, and varied from the initial value by 2%. The intensity data were corrected for this decay. All possible reflections with $0.07 < (\sin\theta)/\lambda < 0.60$ Å^{-1} in the index range $-18 < h < 18$, $0 < k < 15$, $-15 < l < 15$, gave 4490 unique reflections of which 3969 were considered as observed with $I > 3\sigma(I)$, and $R_{\mathrm{int}} = 0.0117$. The structure was solved by the heavy-atom method using *SHELXS*86 (Sheldrick, 1990) and was subjected to least-squares refinement on F with 332 positional and anisotropic thermal parameters for all the non-H atoms, using *SHELX*76 (Sheldrick, 1976). The H-atom positions were calculated riding on the adjacent C atoms