nitudes, $wR = 0.048$ 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-κ^2O,O')carbonyl(triphenylphosphine)-rhodium(I)

BY A. ROODT, J. G. LEIPOLDT, J. C. SWARTS AND G. J. J. STEYN

Department of Chemistry, University of the Orange Free State, PO Box 339, *Bloemfontein* 9300, *South Africa*

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Abstract. $[Rh(C_{12}H_{13}O_2)(CO)\{P(C_6H_5)_3\}]$, $M_r = 582.4$, triclinic, $P\bar{1}$, $a = 13.004$ (2), $b = 10.968$ (2), $c = 11.230$ (1) Å, $\alpha = 114.42$ (1), $\beta = 87.57$ (1), $\gamma = 112.54$ (1)°, $V = 1334.1$ (4) $Å^3$, $Z = 2$, $D_m = 1.46$, $D_x = 1.450$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 7.17$ 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 PPh_3 and $[Rh(bzaa)(CO)_2]$ (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 $R_{\text{int}} = 0.0117$. 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

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Table 1. *Fractional coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3) *for* [Rh(bzaa)(CO){P(C$_6$H$_5$)$_3$}] *with e.s.d.'s in parentheses*

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

	x	*y*	*z*	U_{eq}
Rh	1680.5 (2)	2990.0 (3)	2140.9 (3)	37.44 (6)
P	2101.0 (7)	5372.4 (10)	2783.2 (8)	36.4 (2)
O(1)	34 (2)	2598 (3)	2146 (3)	46 (1)
O(2)	1215 (2)	818 (3)	1661 (3)	51 (1)
C(21)	2777 (3)	6203 (4)	1679 (3)	44 (1)
C(31)	3032 (3)	6576 (4)	4353 (3)	41 (1)
C(11)	836 (3)	5739 (3)	3010 (3)	37 (1)
O(30)	4064 (3)	3455 (5)	2099 (4)	96 (1)
C(30)	3145 (3)	3303 (5)	2128 (4)	57 (1)
C(1)	223 (4)	−1633 (4)	1219 (5)	69 (1)
C(2)	227 (3)	−148 (4)	1544 (3)	47 (1)
C(3)	−775 (3)	69 (4)	1690 (3)	45 (1)
C(4)	−809 (3)	1409 (4)	1950 (3)	42 (1)
C(5)	−1909 (3)	1585 (5)	2026 (5)	63 (1)
C(40)	−1873 (3)	−1211 (4)	1530 (4)	57 (1)
C(41)	−2234 (3)	−1323 (4)	2800 (4)	49 (1)
C(42)	−3323 (4)	−2277 (5)	2772 (5)	70 (1)
C(43)	−3669 (4)	−2442 (6)	3903 (6)	88 (2)
C(44)	−2955 (5)	−1690 (7)	5061 (6)	87 (2)
C(45)	−1884 (4)	−722 (6)	5115 (5)	76 (1)
C(46)	−1532 (3)	−552 (5)	3982 (4)	60 (1)
C(12)	523 (3)	6327 (4)	4248 (4)	50 (1)
C(13)	−498 (3)	6468 (5)	4362 (4)	60 (1)
C(14)	−1216 (3)	6001 (4)	3243 (4)	55 (1)
C(15)	−918 (3)	5388 (4)	2001 (4)	49 (1)
C(16)	102 (3)	5254 (4)	1882 (3)	44 (1)
C(22)	2682 (3)	7463 (4)	1760 (4)	54 (1)
C(23)	3231 (4)	8110 (5)	943 (5)	71 (1)
C(24)	3860 (4)	7508 (6)	56 (5)	78 (1)
C(25)	3938 (4)	6262 (6)	−42 (5)	77 (1)
C(26)	3390 (3)	5596 (5)	768 (4)	57 (1)
C(32)	3267 (3)	5930 (5)	5084 (3)	52 (1)
C(33)	3995 (3)	6797 (5)	6280 (4)	64 (1)
C(34)	4474 (3)	8273 (5)	6731 (4)	65 (1)
C(35)	4237 (3)	8942 (5)	6025 (4)	62 (1)
C(36)	3513 (3)	8089 (4)	4832 (4)	53 (1)

Table 2. *Selected interatomic bond distances* (Å) *and angles* (°) *for* [Rh(bzaa)(CO){P(C$_6$H$_5$)$_3$}] *with e.s.d.'s in parentheses*

Rh—P	2.243 (1)	Rh—O(1)	2.016 (2)
Rh—O(2)	2.048 (2)	Rh—C(30)	1.801 (4)
O(1)—C(4)	1.279 (4)	O(2)—C(2)	1.283 (4)
O(30)—C(30)	1.142 (4)	C(1)—C(2)	1.513 (5)
C(2)—C(3)	1.404 (5)	C(3)—C(4)	1.394 (5)
C(3)—C(40)	1.522 (5)	C(4)—C(5)	1.508 (5)
C(40)—C(41)	1.520 (5)		

P—Rh—O(1)	90.0 (1)	P—Rh—O(2)	175.6 (1)
O(1)—Rh—O(2)	86.9 (1)	P—Rh—C(30)	91.1 (1)
O(1)—Rh—C(30)	178.9 (2)	O(2)—Rh—C(30)	91.9 (2)
Rh—O(1)—C(4)	129.4 (2)	Rh—O(2)—C(2)	128.1 (2)
Rh—C(30)—O(30)	177.8 (4)	O(2)—C(2)—C(1)	112.7 (4)
O(2)—C(2)—C(3)	126.5 (3)	C(1)—C(2)—C(3)	120.8 (4)
C(2)—C(3)—C(4)	122.4 (3)	C(2)—C(3)—C(40)	119.0 (3)
C(4)—C(3)—C(40)	118.5 (3)	O(1)—C(4)—C(3)	126.5 (3)
O(1)—C(4)—C(5)	112.4 (3)	C(3)—C(4)—C(5)	121.1 (3)
C(3)—C(40)—C(41)	115.7 (3)		

assuming C—H = 1.08 Å, and refined with an overall temperature factor. Neutral-atom scattering factors were taken from Cromer & Mann (1968) and anomalous-dispersion corrections for rhodium from *International Tables for X-ray Crystallography* (1962, Vol. III, p. 216). Final $R = 0.0266$ and $wR = 0.0318$ (unit weights). S was not calculated. The residual electron density was $\Delta\rho_{max} = 0.33$ and $\Delta\rho_{min} = -0.44$ e Å^{-3}, with positional $(\Delta/\sigma)_{max}$ for displacement parameters = 0.27. Final atomic coordinates are given in Table 1,* selected geometrical parameters in Table 2, a perspective view (Johnson, 1976) with atomic labels in Fig. 1 and a stereoview of the packing in the unit cell in Fig. 2.

Related literature. The benzyl substituent in the 3-position of the β-diketone makes it possible to support a β-diketone metal complex on a polymer such as polystyrene and thus 'heterogenize' the particular homogeneous catalyst. The present structure determination was undertaken to determine the influence of the benzyl substituent on the Rh—P bond distance, being a possible indicator for comparison of: the relative *trans* influence (Graham, Lamprecht, Potgieter, Roodt & Leipoldt, 1991) of bidentate donor atoms in this type of complex and; the reactivity towards oxidative addition reactions (Basson, Leipoldt, Purcell & Schoeman, 1990), which is very important in catalytic applications such as hydrogenation. No observed influence could be detected structurally, thereby indicating that the reactivity of the β-diketone complex would most probably not be altered significantly by a benzyl

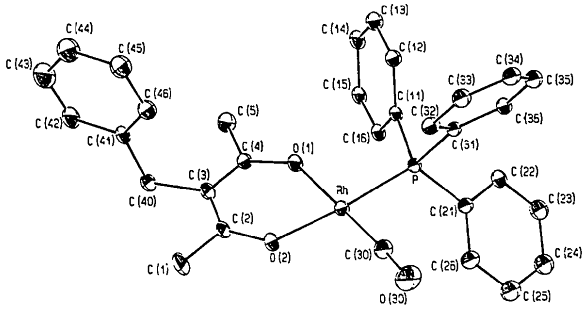

Fig. 1. Perspective view and atom labelling of the molecule (H atoms omitted for clarity).

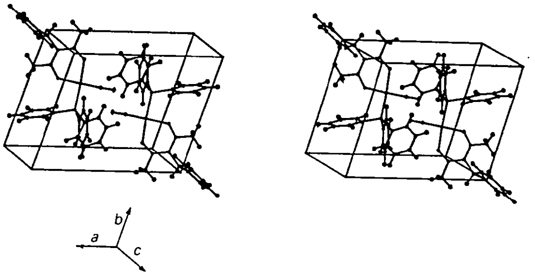

Fig. 2. Stereoview showing the packing in the unit cell.

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* Lists of anisotropic thermal parameters, least-squares planes and deviations, structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54614 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0211]

substituent in the 3-position of the β-diketone. Linking the benzyl group to a polymer support would therefore, in turn, not influence the Rh center significantly so that the initially designed characteristics of the homogeneous catalyst would be left in tact while being 'heterogenized'.

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Structure of 1,1,2,2-Tetracarbonyl{*N*-[(2-pyridyl)methylidene]isopropylamine-μ-N^1,2-σ-N^2,1-η^2-*CH*=N^1}-1,2-bis(triphenylphosphine)ironruthenium(*Fe*—*Ru*)

BY MILCO NUMAN, KEES GOUBITZ AND HENK SCHENK

Laboratory for Crystallography, University of Amsterdam, Nieuwe Achtergracht 166, *Amsterdam, The Netherlands*

AND MARCO J. A. KRAAKMAN

Laboratory for Inorganic Chemistry, University of Amsterdam, Amsterdam, The Netherlands

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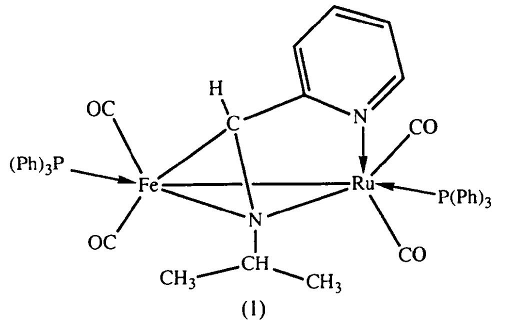
Abstract. $[FeRu(C_9H_{12}N_2)(C_{18}H_{15}P)_2(CO)_4]$, M_r = 941.8, triclinic, $P\bar{1}$, a = 11.625 (3), b = 14.269 (3), c = 15.963 (2) Å, α = 90.95 (1), β = 100.23 (1), γ = 101.95 (2)°, V = 2546 (1) Å^3, Z = 2, D_x = 1.2 g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, μ = 56.7 cm^{-1}, F(000) = 964, room temperature. Final R = 0.064 for 5886 observed reflections. In the title compound the Fe and Ru atoms are six-coordinate. There are no unusual bond lengths or angles.

Experimental. The title compound (1) is shown in the scheme below. A brick-shaped crystal (dimensions 0.65 × 0.40 × 0.30 mm approximately) was used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation and θ–2θ scan. A total of 7484 unique reflections was measured within the range $-13 \leq h \leq 0$, $-16 \leq k \leq 16$ $-17 \leq l \leq 17$. Of these, 5886 were above the significance level of $2.5\sigma(I)$. The maximum value of $(\sin\theta)/\lambda$ was 0.56 Å^{-1}. Two standard reflections ($\bar{2}00$, $\bar{1}2\bar{2}$) were measured hourly; they showed a 14% decrease during the 83 h collecting time, which was corrected for. Unit-cell parameters were refined by a least-squares fitting procedure using 23 reflections with $80 < 2\theta < 84°$. Corrections for Lorentz and polarization effects were applied. The Fe, Ru and both P atoms were found by direct methods. The other non-H atoms were located using difference Fourier maps.



(1)

The H atoms were initially placed in calculated positions. Block-diagonal least-squares refinement on F, anisotropic for the non-H atoms, isotropic for the H atoms, restraining the latter in such a way that the distance to their carrier remained constant at approximately 1.09 Å, converged to R = 0.064, wR = 0.097, $(\Delta/\sigma)_{\max}$ = 0.39, $w = (5.98 + F_{\rm obs} + 0.0102F^2_{\rm obs})^{-1}$, 701 parameters refined. An empirical absorption correction was applied, with corrections in the range 0.711–1.498 (*DIFABS*; Walker & Stuart,

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