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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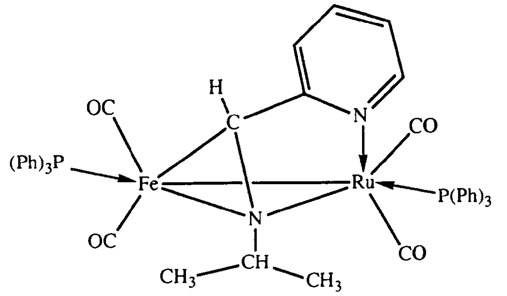
Abstract. $[FeRu(C_9H_{12}N_2)(C_{18}H_{15}P)_2(CO)_4]$, M_r = 941.8, triclinic, $P\bar{1}$, a = 11.625 (3), b = 14.269 (3), c = 15.963 (2) Å, α = 90.95 (1), β = 100.23 (1), γ = 101.95 (2)°, V = 2546 (1) Å^3, Z = 2, D_x = 1.2 g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, μ = 56.7 cm^{-1}, F(000) = 964, room 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 $-13 \le h \le 0$, $-16 \le k \le 16$ $-17 \le l \le 17$. 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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Table 1. *Fractional coordinates of the non*-H *atoms and equivalent isotropic thermal parameters*

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

	x	y	z	U_{eq}(Å^2)
Ru	0.72175 (8)	0.26949 (6)	0.28547 (5)	0.0365 (4)
Fe	0.7711 (1)	0.2150 (1)	0.1390 (1)	0.0344 (9)
P(1)	0.6330 (2)	0.3585 (2)	0.3767 (2)	0.039 (2)
P(2)	0.8019 (2)	0.2025 (2)	0.0080 (2)	0.037 (1)
C(1)	0.817 (1)	0.2137 (9)	0.3662 (8)	0.058 (8)
C(2)	0.601 (1)	0.1597 (8)	0.2773 (7)	0.048 (7)
C(3)	0.676 (1)	0.0997 (8)	0.1253 (8)	0.048 (7)
C(4)	0.904 (1)	0.1892 (8)	0.1937 (8)	0.047 (7)
C(5)	0.541 (1)	0.4198 (9)	0.1179 (7)	0.056 (8)
C(6)	0.462 (1)	0.2415 (9)	0.1054 (7)	0.056 (8)
C(7)	0.7859 (9)	0.3607 (7)	0.1370 (6)	0.036 (6)
C(8)	0.5671 (9)	0.3221 (8)	0.0991 (6)	0.042 (6)
C(12)	0.946 (1)	0.4331 (9)	0.3491 (7)	0.051 (7)
C(13)	1.037 (1)	0.5109 (9)	0.3398 (8)	0.059 (8)
C(14)	1.045 (1)	0.5396 (8)	0.2580 (9)	0.060 (8)
C(15)	0.964 (1)	0.4907 (8)	0.1887 (7)	0.048 (7)
C(16)	0.8734 (9)	0.4141 (7)	0.2033 (7)	0.040 (6)
C(21)	0.476 (1)	0.3613 (9)	0.3404 (7)	0.044 (7)
C(22)	0.435 (1)	0.4467 (8)	0.3295 (7)	0.050 (7)
C(23)	0.313 (1)	0.4442 (9)	0.3032 (8)	0.059 (8)
C(24)	0.233 (1)	0.357 (1)	0.2869 (8)	0.067 (9)
C(25)	0.271 (1)	0.2710 (9)	0.2975 (8)	0.061 (8)
C(26)	0.393 (1)	0.2750 (9)	0.3234 (8)	0.057 (8)
C(31)	0.7037 (9)	0.4855 (7)	0.3959 (7)	0.039 (6)
C(32)	0.735 (1)	0.5299 (9)	0.4763 (7)	0.054 (8)
C(33)	0.793 (1)	0.629 (1)	0.4883 (9)	0.08 (1)
C(34)	0.816 (1)	0.682 (1)	0.418 (1)	0.08 (1)
C(35)	0.782 (1)	0.6364 (9)	0.3364 (8)	0.062 (9)
C(36)	0.727 (1)	0.5389 (8)	0.3251 (7)	0.050 (7)
C(41)	0.632 (1)	0.3182 (8)	0.4857 (7)	0.044 (7)
C(42)	0.731 (1)	0.291 (1)	0.5302 (8)	0.058 (8)
C(43)	0.734 (1)	0.260 (1)	0.6146 (8)	0.07 (1)
C(44)	0.637 (2)	0.259 (1)	0.6522 (8)	0.08 (1)
C(45)	0.538 (1)	0.288 (1)	0.6093 (9)	0.08 (1)
C(46)	0.535 (1)	0.318 (1)	0.5258 (8)	0.065 (9)
C(51)	0.797 (1)	0.0825 (7)	−0.0377 (7)	0.043 (6)
C(52)	0.759 (1)	0.0556 (9)	−0.1226 (8)	0.060 (8)
C(53)	0.767 (1)	−0.035 (1)	−0.1541 (8)	0.07 (1)
C(54)	0.813 (1)	−0.0955 (8)	−0.101 (1)	0.065 (9)
C(55)	0.852 (1)	−0.0707 (9)	−0.0169 (9)	0.066 (9)
C(56)	0.845 (1)	0.0192 (8)	0.0163 (8)	0.053 (8)
C(61)	0.693 (1)	0.2479 (7)	−0.0690 (7)	0.041 (6)
C(62)	0.717 (1)	0.3414 (8)	−0.0952 (7)	0.051 (7)
C(63)	0.624 (1)	0.3799 (9)	−0.1415 (8)	0.07 (1)
C(64)	0.509 (1)	0.324 (1)	−0.1662 (8)	0.08 (1)
C(65)	0.486 (1)	0.231 (1)	−0.1369 (9)	0.069 (9)
C(66)	0.576 (1)	0.1928 (8)	−0.0919 (7)	0.052 (7)
C(71)	0.947 (1)	0.2670 (7)	−0.0132 (7)	0.041 (6)
C(72)	1.031 (1)	0.3180 (9)	0.0507 (8)	0.054 (8)
C(73)	1.147 (1)	0.362 (1)	0.0366 (9)	0.07 (1)
C(74)	1.175 (1)	0.350 (1)	−0.043 (1)	0.07 (1)
C(75)	1.092 (1)	0.300 (1)	−0.1077 (9)	0.07 (1)
C(76)	0.977 (1)	0.2574 (9)	−0.0944 (8)	0.063 (9)
N(1)	0.6764 (7)	0.3099 (6)	0.1574 (5)	0.035 (5)
N(2)	0.8654 (7)	0.3870 (6)	0.2842 (5)	0.039 (5)
O(1)	0.884 (1)	0.1792 (8)	0.4137 (6)	0.097 (8)
O(2)	0.5310 (8)	0.0901 (7)	0.2777 (6)	0.073 (6)
O(3)	0.6183 (9)	0.0236 (6)	0.1158 (7)	0.080 (7)
O(4)	0.9894 (8)	0.1699 (7)	0.2341 (6)	0.072 (6)

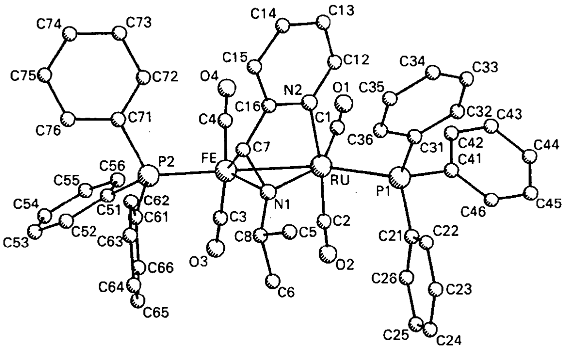

Fig. 1. The molecular structure of the title compound showing the atom numbering.

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

Ru—Fe	2.645 (2)	C(3)—O(3)	1.15 (1)
Ru—P(1)	2.412 (3)	C(4)—O(4)	1.17 (1)
Ru—C(1)	1.85 (1)	C(5)—C(8)	1.52 (2)
Ru—C(2)	1.86 (1)	C(6)—C(8)	1.52 (1)
Ru—N(1)	2.138 (8)	C(7)—C(16)	1.42 (1)
Ru—N(2)	2.111 (8)	C(7)—N(1)	1.42 (1)
Fe—P(2)	2.195 (3)	C(8)—N(1)	1.48 (1)
Fe—C(3)	1.77 (1)	C(12)—C(13)	1.40 (2)
Fe—C(4)	1.75 (1)	C(12)—N(2)	1.33 (1)
Fe—C(7)	2.05 (1)	C(13)—C(14)	1.39 (2)
Fe—N(1)	1.962 (9)	C(14)—C(15)	1.39 (2)
C(1)—O(1)	1.18 (2)	C(15)—C(16)	1.41 (1)
C(2)—O(2)	1.15 (1)	C(16)—N(2)	1.37 (1)

Fe—Ru—P(1)	155.61 (9)	C(4)—Fe—N(1)	134.2 (5)
Fe—Ru—C(1)	103.9 (5)	C(7)—Fe—N(1)	41.4 (4)
Fe—Ru—C(2)	89.4 (4)	Ru—C(1)—O(1)	175 (1)
Fe—Ru—N(1)	47.0 (3)	Ru—C(2)—O(2)	175 (1)
Fe—Ru—N(2)	84.6 (3)	Fe—C(3)—O(3)	177 (1)
P(1)—Ru—C(1)	100.3 (5)	Fe—C(4)—O(4)	176 (1)
P(1)—Ru—C(2)	94.1 (4)	Fe—C(7)—C(16)	114.4 (8)
P(1)—Ru—N(1)	108.6 (3)	Fe—C(7)—N(1)	65.9 (5)
P(1)—Ru—N(2)	92.0 (3)	C(16)—C(7)—N(1)	118.5 (9)
C(1)—Ru—C(2)	89.4 (5)	C(5)—C(8)—C(6)	111 (1)
C(1)—Ru—N(1)	149.8 (5)	C(5)—C(8)—N(1)	109.9 (8)
C(1)—Ru—N(2)	90.6 (4)	C(6)—C(8)—N(1)	111.0 (9)
C(2)—Ru—N(1)	96.8 (4)	C(13)—C(12)—N(2)	123 (1)
C(2)—Ru—N(2)	173.8 (5)	C(12)—C(13)—C(14)	118 (1)
N(1)—Ru—N(2)	80.1 (3)	C(13)—C(14)—C(15)	120 (1)
Ru—Fe—P(2)	166.3 (1)	C(14)—C(15)—C(16)	119 (1)
Ru—Fe—C(3)	98.3 (4)	C(7)—C(16)—C(15)	123 (1)
Ru—Fe—C(4)	89.7 (4)	C(7)—C(16)—N(2)	116.0 (9)
Ru—Fe—C(7)	73.9 (3)	C(15)—C(16)—N(2)	120.9 (9)
Ru—Fe—N(1)	52.8 (2)	Ru—N(1)—Fe	80.2 (3)
P(2)—Fe—C(3)	90.2 (4)	Ru—N(1)—C(7)	105.2 (6)
P(2)—Fe—C(4)	99.2 (4)	Ru—N(1)—C(8)	136.9 (7)
P(2)—Fe—C(7)	93.2 (3)	Fe—N(1)—C(7)	72.7 (6)
P(2)—Fe—N(1)	114.3 (3)	Fe—N(1)—C(8)	124.9 (7)
C(3)—Fe—C(4)	101.1 (5)	C(7)—N(1)—C(8)	115.2 (8)
C(3)—Fe—C(7)	147.7 (5)	Ru—N(2)—C(12)	128.9 (8)
C(3)—Fe—N(1)	108.8 (5)	Ru—N(2)—C(16)	112.0 (6)
C(4)—Fe—C(7)	110.0 (5)	C(12)—N(2)—C(16)	119.0 (9)

1983). A final difference Fourier map revealed residual electron density between −1.1 and 1.8 e Å^{-3}. Scattering factors were taken from Cromer & Mann (1968); *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55). Anomalous dispersion for Fe and Ru was corrected for. All calculations were performed with *XTAL*3.0 (Hall & Stewart, 1990), unless stated otherwise. Final positional parameters for the non-H atoms are listed in Table 1,* selected bond lengths and bond angles in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule, showing the numbering scheme, is given in Fig. 1.

Related literature. The title compound is one of the first iron–ruthenium complexes with a six-electron donating *R*-pyridine-2-carbaldehyde-imine ligand and it is the first example of such a complex with ligands other than carbonyl (Kraakman, Elsevier *et al.*, 1991). The elucidation of the structure of this complex proved to be of great importance for the

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and bond angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54618 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0278]

determination of the mechanism of the reactions of FeRu(CO)$_6$(α-diimine) complexes with molecular hydrogen (Zoet *et al.*, 1989); these reactions in the presence of an additional ligand led to the formation of the title compound. The synthesis of this compound will be presented in a separate paper together with its chemical and spectroscopic properties (Kraakman, Goubitz, Numan & Vrieze, 1991).

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Structure of α-Cyclopiazonic Acid

BY PETRUS H. VAN ROOYEN

Department of Chemistry, University of Pretoria, Pretoria 0002, *South Africa*

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Abstract. $C_{20}H_{20}N_2O_3$, $M_r = 336.1$, tetragonal, $P4_32_12$, $a = 10.406\,(1)$, $c = 61.549\,(14)$ Å, $V = 6664\,(2)$ Å^3, $Z = 16$, $D_x = 1.34$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 6.20$ cm^{-1}, $F(000) = 2832$, $T = 298$ K, final $wR = 0.064$ ($R = 0.077$) for 3357 reflections and 470 variable parameters. α-Cyclopiazonic acid is the main toxic principle of the strain CSIR 1082 (ATCC 36064 or NRRL 3523) of *Penicillium griseofulvum* Dierckx. The molecule crystallizes as an *exo*-enol rather than the *endo*-enol tautomer as suggested [Holzapfel (1968). *Tetrahedron*, **24**, 2101–2119], and the *exo*-cyclic enolic moieties have different orientations in the two molecules in the asymmetric unit.

Experimental. The title compound was obtained as described previously, and isolated as pale brown octahedral crystals from MeOH/CHCl$_3$ (m.p. 522–524 K) (Holzapfel, 1968). Crystal $0.17 \times 0.19 \times 0.24$ mm, Philips PW 1100 diffractometer, graphite monochromator, unit cell from 25 reflections ($17 < \theta < 23°$), 5924 reflections for $5 < \theta < 60°$ in the range $0 < h < 11$, $0 < k < 11$, $0 < l < 69$ using ω–2θ scans, peak scan width 0.40°, scan speed 0.96° min^{-1}, backgrounds not measured but assumed isotropic and calculated as a function of θ from the counts of systematically absent reflections. Three standard reflections measured every 130 reflections. Lp correction applied, no decay or absorption corrections. 3357 unique reflections with $F > 2\sigma(F)$ used, structure solved using *SHELXS*86 (Sheldrick, 1986), all hydrogen atoms placed in calculated positions (C—H 1.08 Å, H—C—H 109.5°, C=C—H 120.0°), least-squares refinement on F using *SHELX*76 (Sheldrick, 1976), $\sigma^{-2}(F)$ weights, all non-hydrogen atoms anisotropic, hydrogen atoms isotropic and constrained to ride upon their associated heavy atoms with a common thermal parameter that refined to $U_{\rm iso} = 0.105\,(4)$ Å^2. Final $wR = 0.064$, $R = 0.077$, maximum positional shift/e.s.d. less than 0.7, residual electron density = 0.40 e Å^{-3}. Scattering factors from *SHELX*76. Table 1 gives the atom parameters.* Fig. 1 shows the molecular structure and the numbering scheme of molecule *A*, drawn by *ORTEP* (Johnson, 1965). For the second molecule *B* the positions of O2 and C18 in Fig. 1 should be interchanged. Table 2 gives selected bond distances and bond angles.

Related literature. The existence in solution of the *exo*-cyclic enol tautomer is also indicated in NMR studies of related compounds (Nolte, Steyn & Wessels, 1980; Steyn & Wessels, 1978).

The author wishes to thank Dr C. P. Gorst-Allman for a sample of the title compound.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54501 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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