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6-(2,6-Dimethylphenylimino)-1,1,1-tris[(2,6-dimethylphenyl)isocyano]-10-methoxycarbonyl-8-oxo-2,5-bis(2-propyl)-9-oxa-2,5-diaza-1-ferratricyclo[5.2.1.$0^{4,10}$]dec-2-ene

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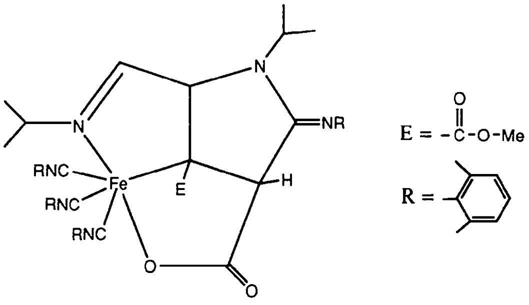
Abstract. $[\mathrm{Fe}(C_{49}H_{56}N_6O_4)]$, $M_r = 848.9$, monoclinic, $P2_1/a$, $a = 14.47\ (3)$, $b = 19.749\ (2)$, $c = 17.50\ (2)$ Å, $\beta = 110.6\ (1)°$, $V = 4681\ (12)$ Å^3, $Z = 4$, $D_x = 1.2$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.5418$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 29.5$ cm^{-1}, $F(000) = 1800$, $T = 254.6$ K, final $R = 0.056$ for 1811 observed reflections. The crystals were of such a quality that 1146 reflections remained unobserved. They contain molecules in which the Fe atom is six coordinate. There are no unusual bond lengths and angles.

Experimental. A brick-shaped crystal (I) (dimensions 0.15 × 0.25 × 0.40 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.



(I)

Table 1. *Fractional coordinates for the non-*H *atoms and equivalent isotropic thermal parameters*

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

	x	y	z	U_{eq}(Å^2)
Fe	−0.0446 (1)	0.1005 (1)	0.2078 (1)	0.031 (1)
C(1)	−0.0144 (8)	0.0036 (5)	0.2546 (6)	0.023 (8)
C(2)	−0.0707 (9)	−0.0474 (5)	0.1925 (7)	0.029 (9)
C(3)	−0.1082 (8)	−0.0160 (6)	0.1097 (7)	0.029 (8)
C(4)	0.0906 (9)	−0.0869 (6)	0.2465 (7)	0.036 (9)
C(5)	0.0926 (8)	−0.0108 (5)	0.2654 (7)	0.025 (8)
C(6)	0.1199 (9)	0.0339 (6)	0.2052 (7)	0.039 (9)
C(7)	−0.0294 (9)	−0.0023 (6)	0.3325 (7)	0.041 (9)
C(8)	−0.143 (1)	−0.0255 (8)	0.4022 (8)	0.07 (1)
C(9)	−0.030 (1)	−0.1745 (6)	0.1891 (7)	0.05 (1)
C(10)	−0.115 (1)	−0.1874 (7)	0.1095 (9)	0.07 (1)
C(11)	−0.058 (1)	−0.1986 (7)	0.2603 (9)	0.08 (1)
C(12)	−0.1479 (9)	0.0760 (6)	0.0183 (7)	0.039 (9)
C(13)	−0.2331 (9)	0.1225 (6)	0.0106 (7)	0.044 (9)
C(14)	−0.064 (1)	0.1076 (7)	−0.0041 (8)	0.06 (1)
C(15)	0.2582 (9)	−0.1108 (6)	0.3278 (7)	0.034 (8)
C(16)	0.3361 (9)	−0.1062 (7)	0.2996 (7)	0.045 (9)
C(17)	0.428 (1)	−0.0901 (7)	0.3509 (8)	0.06 (1)
C(18)	0.445 (1)	−0.0779 (7)	0.4321 (9)	0.07 (1)
C(19)	0.369 (1)	−0.0823 (8)	0.4629 (8)	0.09 (1)
C(20)	0.272 (1)	−0.1016 (7)	0.4090 (7)	0.05 (1)
C(21)	0.318 (1)	−0.1196 (7)	0.2114 (8)	0.05 (1)
C(22)	0.193 (1)	−0.1121 (8)	0.4468 (8)	0.08 (1)
C(23)	0.0252 (9)	0.1359 (6)	0.3077 (7)	0.033 (9)
C(24)	0.1376 (9)	0.1739 (6)	0.4528 (7)	0.032 (9)
C(25)	0.235 (1)	0.1533 (6)	0.4746 (8)	0.05 (1)
C(26)	0.297 (1)	0.1732 (7)	0.5528 (8)	0.06 (1)
C(27)	0.261 (1)	0.2124 (7)	0.6005 (9)	0.08 (1)
C(28)	0.163 (1)	0.2312 (7)	0.5765 (8)	0.07 (1)
C(29)	0.0987 (9)	0.2105 (6)	0.4996 (7)	0.038 (9)
C(30)	0.272 (1)	0.1123 (8)	0.421 (1)	0.09 (1)
C(31)	−0.009 (1)	0.2293 (8)	0.4743 (9)	0.07 (1)
C(32)	−0.0583 (8)	0.1871 (6)	0.1636 (7)	0.031 (8)
C(33)	−0.0851 (9)	0.3105 (6)	0.1078 (7)	0.035 (9)
C(34)	−0.030 (1)	0.3352 (6)	0.0655 (9)	0.05 (1)
C(35)	−0.052 (1)	0.3997 (7)	0.0318 (8)	0.06 (1)
C(36)	−0.128 (1)	0.4347 (7)	0.0442 (8)	0.07 (1)
C(37)	−0.179 (1)	0.4095 (7)	0.0901 (8)	0.06 (1)
C(38)	−0.1595 (9)	0.3444 (6)	0.1216 (7)	0.04 (1)
C(39)	0.051 (1)	0.2958 (8)	0.054 (1)	0.09 (1)
C(40)	−0.216 (1)	0.3168 (7)	0.1711 (9)	0.06 (1)
C(41)	−0.1605 (8)	0.1043 (6)	0.2238 (7)	0.028 (8)
C(42)	−0.3214 (9)	0.0995 (6)	0.2548 (7)	0.041 (9)
C(43)	−0.380 (1)	0.0435 (6)	0.2291 (8)	0.05 (1)
C(44)	−0.465 (1)	0.0369 (7)	0.2481 (9)	0.06 (1)
C(45)	−0.488 (1)	0.0875 (8)	0.2935 (9)	0.08 (1)
C(46)	−0.427 (1)	0.1427 (8)	0.3197 (8)	0.07 (1)
C(47)	−0.345 (1)	0.1496 (7)	0.2984 (8)	0.05 (1)
C(48)	−0.356 (1)	−0.0108 (8)	0.180 (1)	0.09 (1)
C(49)	−0.282 (1)	0.2119 (7)	0.3254 (9)	0.07 (1)
N(1)	−0.0026 (6)	−0.1031 (5)	0.1968 (5)	0.030 (6)
N(2)	−0.1067 (7)	0.0475 (4)	0.1028 (5)	0.018 (6)
N(3)	0.1630 (7)	−0.1299 (5)	0.2738 (6)	0.037 (7)
N(4)	0.0713 (7)	0.1558 (5)	0.3730 (6)	0.035 (7)
N(5)	−0.0662 (7)	0.2439 (5)	0.1409 (5)	0.033 (7)
N(6)	−0.2352 (7)	0.1040 (5)	0.2350 (5)	0.043 (7)
O(1)	0.0785 (5)	0.0904 (4)	0.1868 (4)	0.033 (5)
O(2)	0.1829 (7)	0.0122 (4)	0.1763 (6)	0.067 (7)
O(3)	−0.1210 (6)	−0.0235 (4)	0.3263 (4)	0.044 (6)
O(4)	0.0319 (6)	0.0121 (4)	0.3990 (5)	0.049 (6)

A total of 2957 unique reflections was measured within the range $-13 \leq h \leq 0$, $0 \leq k \leq 16$ $-13 \leq l \leq 14$. Of these, 1811 were above the significance level of $2.5\sigma(I)$. The maximum value of $(\sin\theta)/\lambda$ was 0.42 Å^{-1}. Two standard reflections (020, $\bar{2}02$) were measured hourly; no significant decrease was detected during the 35 h collecting time. Unit-cell parameters were refined by a least-squares fitting procedure using 23 reflections with $50 < 2\theta < 58°$. Corrections for Lorentz and polarization effects were applied. The Fe atom was found by direct methods. The other atoms were located using the program *AUTOFOUR* (Kinneging & de Graaf, 1984). The

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

Fe—C(1)	2.07 (1)	C(2)—C(3)	1.49 (2)
Fe—C(23)	1.82 (1)	C(2)—N(1)	1.46 (1)
Fe—C(32)	1.86 (1)	C(3)—N(2)	1.26 (1)
Fe—C(41)	1.80 (1)	C(4)—C(5)	1.54 (2)
Fe—N(2)	2.025 (8)	C(4)—N(1)	1.36 (1)
Fe—O(1)	1.95 (1)	C(5)—C(6)	1.53 (2)
C(1)—C(2)	1.49 (1)	C(6)—O(1)	1.25 (1)
C(1)—C(2)	1.52 (2)		
C(1)—Fe—C(23)	90.4 (5)	Fe—C(1)—C(2)	110.6 (7)
C(1)—Fe—C(32)	173.5 (6)	Fe—C(1)—C(5)	106.6 (8)
C(1)—Fe—C(41)	93.2 (5)	C(2)—C(1)—C(5)	103 (1)
C(1)—Fe—N(2)	81.1 (4)	C(1)—C(2)—C(3)	110.1 (9)
C(1)—Fe—O(1)	84.4 (4)	C(1)—C(2)—N(1)	106.7 (8)
C(23)—Fe—C(32)	89.2 (5)	C(3)—C(2)—N(1)	112 (1)
C(23)—Fe—C(41)	93.7 (6)	C(2)—C(3)—N(2)	120 (1)
C(23)—Fe—N(2)	170.3 (4)	C(5)—C(4)—N(1)	107.9 (9)
C(23)—Fe—O(1)	89.3 (5)	C(1)—C(5)—C(4)	102.5 (9)
C(32)—Fe—C(41)	93.3 (6)	C(1)—C(5)—C(6)	107.9 (9)
C(32)—Fe—N(2)	98.7 (4)	C(4)—C(5)—C(6)	114 (1)
C(32)—Fe—O(1)	89.1 (5)	C(5)—C(6)—O(1)	118 (1)
C(41)—Fe—N(2)	91.4 (5)	C(2)—N(1)—C(4)	110.7 (9)
C(41)—Fe—O(1)	176.2 (4)	Fe—N(2)—C(3)	116.4 (7)
N(2)—Fe—O(1)	85.3 (4)	Fe—O(1)—C(6)	115.3 (9)

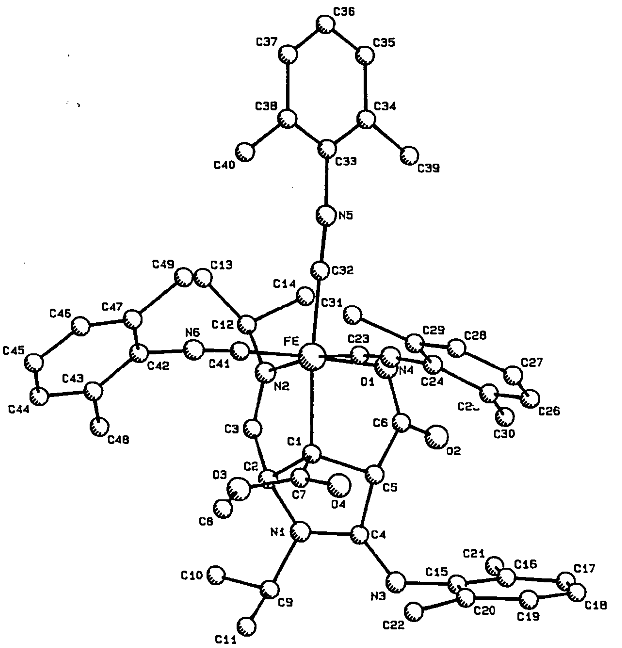

Fig. 1. The molecular structure of the title compound showing the atom numbering.

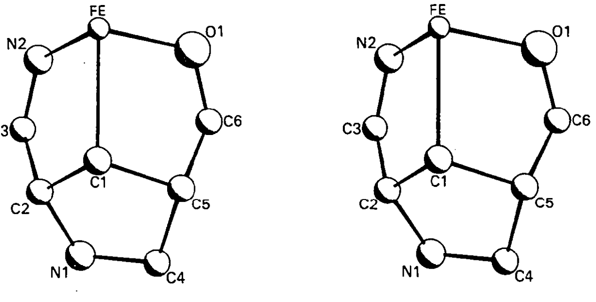

Fig. 2. Stereoscopic view of the tricyclic moiety.

positions of the H atoms were calculated and kept fixed during refinement with $U = 0.09$ Å^2. Block-diagonal least-squares refinement on F of 541 parameters, anisotropic for the non-H atoms, converged to $R = 0.056$, $wR = 0.062$, $(\Delta/\sigma)_{\max} = 0.49$ with $w = (22.12 + F_{\text{obs}} + 0.007F^2_{\text{obs}})^{-1}$. An empirical absorption correction was applied, with corrections in the range 0.84–1.35 (*DIFABS*; Walker & Stuart, 1983). A final difference Fourier map revealed a residual electron density between −0.3 and 0.4 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 was corrected for. All calculations were performed with *XTAL*2.6 (Hall & Stewart, 1989) unless stated otherwise. Final positional parameters for the non-H atoms are listed in Table 1,* selected bond lengths and bond angles are in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule, showing the numbering scheme, is given in Fig. 1 and a stereoscopic view of the tricyclic moiety is in Fig. 2. The conformation of the three five-membered rings according to the classification of Altona, Geise & Romers (1968) is as follows. Ring 1 [C(1), C(2), C(3), N(2) and Fe], $\Delta = 0.23$, half-chair. Ring 2 [C(1), C(2), C(4), C(5) and N(1)], $\Delta = 6.8$, pseudo half-chair. Ring 3 [C(1), C(5), C(6), O(1) and Fe], $\Delta = 19.9$, distorted envelope.

Related literature. The title compound is one of the first examples of a tricyclic structure with the metal incorporated into one of the rings. The synthesis and chemical properties will be discussed in a separate paper (de Lange, Frühauf, van Wijnkoop & Vrieze, 1991). This compound is another proof of the synthesis of polycyclic structures starting from Fe (α-diimine) and acetylenes (Frühauf, Siels & Stam, 1989; de Lange, Frühauf, van Wijnkoop, Vrieze, Wang, Heijdenrijk & Stam, 1990).

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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 54581 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0257]

References

Altona, C., Geise, H. J. & Romers, C. (1968). *Tetrahedron*, **42**, 13.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

Frühauf, H.-W., Siels, F. & Stam, C. H. (1989). *Organometallics*, **8**, 2338–2343.

Hall, S. R. & Stewart, J. M. (1989). Editor. *XTAL*2.6 *User's Manual.* Univs. of Western Australia, Australia, and Maryland, USA.

Kinneging, A. J. & de Graaf, R. A. G. (1984). *J. Appl. Cryst.* **17**, 364–366.

LANGE, P. P. M. DE, FRÜHAUF, H.-W., VAN WIJNKOOP, M. & VRIEZE, K. (1991). To be published.

LANGE, P. P. M. DE, FRÜHAUF, H.-W., VAN WIJNKOOP, M., VRIEZE, K., WANG, Y., HEIJDENRIJK, D. & STAM, C. H. (1990). *Organometallics*, **9**, 1691–1694.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* A**39**, 158–166.

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Structure of [η-$C_5(CH_3)_5$]$RuCl_2(NC_5H_5)$

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Abstract. Dichloro(η-pentamethylcyclopentadienyl)(pyridine)ruthenium, [$RuCl(C_{10}H_{15})(C_5H_5N)$], M_r = 386.31, monoclinic, $P2_1/m$, $a = 7.2112$ (5), $b = 13.430$ (1), $c = 8.4573$ (7) Å, $\beta = 106.540$ (6)°, $V = 785.2$ (1) Å^3, $Z = 2$, $D_x = 1.63$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 13.1 cm^{-1}, $F(000) = 390$, $T = 295$ K, $R = 0.027$, $wR = 0.051$ for 1283 unique observed reflections [$I_o \geq 2.5\sigma(I)$]. The Ru has a piano-stool coordination of η-$C_5(CH_3)_5$, two Cl atoms and the N of pyridine [Ru—Cl 2.386 (1), Ru—N 2.150 (5) Å and Ru—C_5-ring centroid 1.819 (4) Å].

Experimental. Orange plates of [η-$C_5(CH_3)_5$]Ru-$Cl_2(NC_5H_5)$ were obtained by treating a solution of [{η-$C_5(CH_3)_5$}RuCl(μ-Cl)]$_2$ (Tilley, Grubbs & Bercaw, 1984; Oshima, Suzuki & Moro-oka, 1984) in tetrahydrofuran with pyridine (Bottomley, McKenzie-Boone & Sutton, 1991). A crystal of dimensions 0.35 × 0.15 × 0.25 mm was coated with Apiezon grease, sealed in a capillary and mounted on an Enraf–Nonius CAD-4 diffractometer. Lattice constants were obtained by accurate centring of 25 reflections in the range $30 < 2\theta < 40°$. Intensities were measured using the $\omega/2\theta$-scan mode to a $2\theta_{\max}$ of 50° ($h_{\max}$ 8, $k_{\max}$ 15, $l_{\max}$ 10). Three standard reflections were monitored every hour; there was no significant change in their intensity. The intensities of 2590 reflections were measured and averaged to yield 1450 unique reflections ($R_{\rm int} = 0.014$) of which 1283 were judged as significant by the criterion that $I > 2.5\sigma(I)$. No absorption correction was made. The structure was solved and refined using *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). The structure could only be solved in $P2_1$, but refinement showed clearly that $P2_1/m$ was the correct space group. The function minimized was $\sum w(\Delta F)^2$, where $w = 1[\sigma(F)^2 + 0.001F^2]$ and σ was obtained from counting statistics. All non-H atoms were refined with anisotropic thermal parameters. All of the H atoms were observed in a difference Fourier synthesis. Their positions were idealized to sp^2 (C_5H_5N) or sp^3 [$C_5(CH_3)_5$] geometry, and they were allowed to ride on the C atom to which they were attached (C—H = 0.96 Å) with fixed isotropic thermal parameters. Full-matrix least-squares refinement of 94 parameters for 1283 reflections gave a final $R = 0.027$, $wR = 0.051$ and a goodness of fit of 1.38. The largest Δ/σ was 0.002. A final difference synthesis had a maximum peak of 0.32 e Å^{-3}, located between C(102) and C(103) of the C_5H_5N ring, and a minimum hole of −0.65 e Å^{-3} 1.0 Å from Ru. Scattering factors for neutral atoms, corrected for the real and imaginary parts of the anomalous dispersion, were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Positional parameters are listed in Table 1,* selected bond lengths and angles in Table 2 and an *ORTEP* (Johnson, 1976) diagram of the structure is shown in Fig. 1.

Related literature. The structure of [{η-$C_5(CH_3)_5$}RuCl(μ-Cl)]$_2$ is similar to its Rh analogue (Koelle & Kossakowski, 1989; Churchill, Julis & Rotella, 1977). No other [η-$C_5(CH_3)_5$] derivatives of Ru$^{\rm III}$ have been structurally characterized. The structures of a number of Ru$^{\rm II}$ derivatives, notably [{η-$C_5(CH_3)_5$}Ru(μ_3-Cl)]$_4$ (Fagan, Mahoney, Calabrese & Williams, 1990) and [η-$C_5(CH_3)_5$]$RuCl_2$(η^2:η^4-μ_2-C_4H_4)Ru[η-$C_5(CH_3)_5$] (Campion, Heyn & Tilley, 1990), have been determined.

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* Lists of H-atom positions, anisotropic thermal parameters, structure-factor amplitudes, further bond distances and angles and a labelled diagram of the molecule have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54510 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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