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Structure of $[\eta\text{-}C_5(CH_3)_5]RuCl_2(NC_5H_5)$

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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, β = 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 $[\eta\text{-}C_5(CH_3)_5]RuCl_2(NC_5H_5)$ were obtained by treating a solution of $[\{\eta\text{-}C_5(CH_3)_5\}RuCl(\mu\text{-}Cl)]_2$ (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_{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 $[\{\eta\text{-}C_5(CH_3)_5\}RuCl(\mu\text{-}Cl)]_2$ is similar to its Rh analogue (Koelle & Kossakowski, 1989; Churchill, Julis & Rotella, 1977). No other $[\eta\text{-}C_5(CH_3)_5]$ derivatives of Ru^{III} have been structurally characterized. The structures of a number of Ru^{II} derivatives, notably $[\{\eta\text{-}C_5(CH_3)_5\}Ru(\mu_3\text{-}Cl)]_4$ (Fagan, Mahoney, Calabrese & Williams, 1990) and $[\eta\text{-}C_5(CH_3)_5]RuCl_2(\eta^2{:}\eta^4\text{-}\mu_2\text{-}C_4H_4)Ru[\eta\text{-}C_5(CH_3)_5]$ (Campion, Heyn & Tilley, 1990), have been determined.

* 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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Table 1. *Atomic coordinates of the non-H atoms with e.s.d.'s in parentheses*

B_{iso} is the mean of the principal axes of the thermal ellipsoid.

	x	y	z	B_{iso} (Å^2)
Ru	0.87654 (6)	¼*	0.33182 (5)	2.173 (22)
Cl	1.07911 (17)	0.88628 (9)	0.30806 (14)	3.50 (5)
N	1.0151 (7)	¼*	0.5930 (6)	2.50 (20)
C(101)	1.0432 (7)	0.6645 (4)	0.6782 (6)	3.27 (19)
C(102)	1.1008 (7)	0.6630 (4)	0.8484 (6)	3.96 (23)
C(103)	1.1300 (11)	¼*	0.9374 (8)	3.9 (3)
C(1)	0.5864 (8)	¼*	0.3756 (7)	2.65 (25)
C(11)	0.5424 (10)	¼*	0.5390 (8)	3.9 (3)
C(2)	0.6078 (6)	0.6637 (3)	0.2848 (5)	2.48 (16)
C(21)	0.5929 (7)	0.5582 (4)	0.3322 (7)	3.93 (22)
C(3)	0.6377 (6)	0.6969 (3)	0.1318 (5)	2.65 (17)
C(31)	0.6485 (7)	0.6322 (4)	−0.0094 (6)	3.86 (22)

* y coordinate fixed by symmetry.

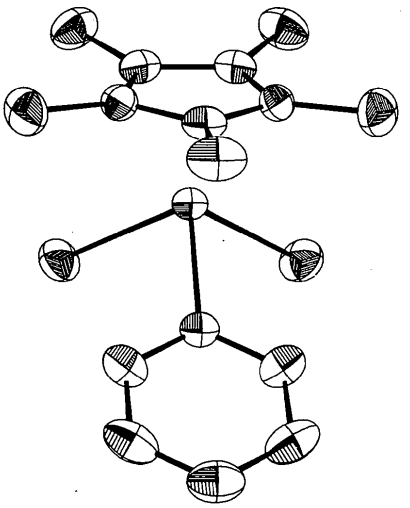

Fig. 1. Structure of $[\eta\text{-}C_5(CH_3)_5]RuCl_2(NC_5H_5)$.

Table 2. *Important interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Ru—Cl	2.386 (1)	Cl—Ru—Cl(*a*)	100.21 (5)
Ru—N	2.150 (5)	Cl—Ru—N	88.28 (9)
Ru—C(1)	2.227 (6)	Cl—Ru—Cp*	123.4 (2)
Ru—C(2)	2.195 (4)	N—Ru—Cp*	123.6 (2)
Ru—C(3)	2.163 (4)		
Ru—Cp*	1.819 (4)		

Cp* is the centroid of the C_5 ring of the $C_5(CH_3)_5$ group [obtained by averaging the coordinates of C(1), C(2), C(2)*a*, C(3) and C(3)*a*].

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Structure of *trans*-Di-μ-chloro-dichlorobis(triphenylphosphite)dipalladium

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Abstract. $[Pd_2Cl_4(C_{18}H_{15}O_3P)_2]$, $M_r = 975.19$, monoclinic, $P2_1/c$, $a = 10.034$ (2), $b = 16.278$ (2), $c = 13.129$ (2) Å, $\beta = 112.04$ (1)°, $V = 1987.5$ (5) Å^3, $Z = 2$, $D_x = 1.63$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, μ(Mo $K\alpha$) = 12.77 cm^{-1}, $F(000) = 968$, $T = 294$ K, $R = 0.0504$ for 1811 unique observed reflections, $I > 2\sigma(I)$. The structure consists of centrosymmetric dimers, with the Pd^{II} ions joined by unsymmetrical double Cl^- bridges. A terminal Cl^- and a $P(OPh)_3$ ligand complete the near square-planar coordination of each Pd. Bond lengths: Pd—P 2.187 (3), Pd—Cl(terminal) 2.269 (3), Pd—Cl(bridging, *trans* to P) 2.413 (2), Pd—Cl(bridging, *trans* to Cl) 2.309 (2) Å.

Experimental. Compound isolated from the reaction of orthomanganated triphenylphosphite, $(PhO)_2(\overline{C_6H_4O})PMn(CO)_4$ with $PdCl_2$; a more direct synthesis is available (Chatt & Venanzi, 1957). Plate-like

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