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Structure of *mer*-Trichlorotris(pyridine)rhodium(III), $[RhCl_3(C_5H_5N)_3]$*

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Abstract. $M_r = 443{\cdot}45$, monoclinic, $P2_1/n$, $a = 9{\cdot}008\,(2)$, $b = 12{\cdot}457\,(2)$, $c = 15{\cdot}445\,(2)$ Å, $\beta = 90{\cdot}87\,(2)°$, $V = 1732{\cdot}9$ Å^3, $Z = 4$, D_m(flotation) = 1·63, $D_x = 1{\cdot}65$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 13{\cdot}74$ mm^{-1}, $F(000) = 888{\cdot}0$, $T = 293$ K, $R = 0{\cdot}052$ for 1171 reflections. The rhodium atom has nearly regular octahedral coordination with three meridional chlorines with bond lengths ⟨Rh—Cl⟩ = 2·334 (5) and ⟨Rh—N⟩ = 2·057 (13) Å. The three pyridine moieties are planar and are nearly perpendicular to each other. The molecules are held together by van der Waals forces.

Introduction. Rhodium complexes with pyridine have not been well studied in spite of their growing importance as homogeneous catalysts, *e.g.* for carbonylation of nitro compounds (Hammond & Franco, 1974). It is often desirable to use catalysts with well defined structures so that a suitable mechanism for the reaction can be evolved. In this context, the structure of $Rh(py)_3Cl_3$ has been studied.

Experimental. Synthesis from $RhCl_3.3H_2O$ and pyridine (Chaudhari & Rode, 1983); crystal (orange colour, needle shape) approximate dimensions 0·18 × 0·15 × 0·25 mm used for data collection; lattice parameters from 25 reflections ($12 < 2\theta < 35°$); intensity data collected on Enraf–Nonius CAD-4F-11M single-crystal X-ray diffractometer; graphite-monochromated Mo $K\alpha$ radiation; 3 standard reflections (060, 402, $0\bar{4}8$), ≤ 5% intensity variation; $\omega/2\theta$ scan mode; scan speed 1° min^{-1}; $\theta \leq 24°$; 3054 reflections collected, 1171 judged significant ($|F_o| \geq 3\sigma|F_o|$); h 0–10, k 0–13, l 0–±17; no correction for absorption; structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); Fourier map based on rhodium peak and four other peaks which formed the right kind of geometry revealed the entire structure. Full-matrix refinement of scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for H atoms, located from difference map) converged to $R = 0{\cdot}052$ and $R_w = 0{\cdot}054$; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = (18{\cdot}0 + 1{\cdot}0|F_o| + 0{\cdot}014|F_o|^2)^{-1}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); max. $\Delta/\sigma = 0{\cdot}1$, final $\Delta\rho$ excursions $< |0{\cdot}3|$ e Å^{-3}; *LALS* (Gantzel, Sparks & Trueblood, 1961) used for refinement.

Discussion. The atomic parameters with their standard deviations for non-H atoms are given in Table 1. Bond lengths and angles are given in Table 2.† Fig. 1 gives a perspective view (*ORTEP*; Johnson, 1965) of the molecule along with the numbering of atoms. The rhodium has a nearly regular octahedral coordination with three meridional chlorines. The ⟨Rh—Cl⟩ distance of 2·334 (5) Å and ⟨Rh—N(py)⟩ distance of 2·057 (13) Å agree well with those reported (Rozière, Lehmann & Potier, 1979; Murray-Rust, 1977).This coordination around rhodium particularly resembles that in trichloro(dimethyl sulfoxide)bis(pyridine)-rhodium (Colamarino & Orioli, 1976) with ⟨Rh—Cl⟩ distance of 2·334 (5) Å and ⟨Rh—N(py)⟩ distance of 2·052 (13) Å, the averages being over the two independent molecules. The planar pyridine moieties are nearly perpendicular to each other and their geometry is in good agreement with the structure of *trans*-dichlorotetrakis(pyridine)rhodium(III)

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† Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes' information have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39349 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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hydrogendinitrate (Rozière, Lehmann & Potier, 1979) with ⟨C—N⟩ distance of 1·39 (2) Å and ⟨C—C⟩ distance of 1·39 (2) Å. The crystal structure is stabilized by van der Waals interactions.

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Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic thermal parameters for non-hydrogen atoms with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
Rh	9934 (2)	19107 (1)	−2526 (1)	3·1 (1)
Cl(1)	9278 (5)	10690 (4)	−3248 (3)	4·1 (1)
Cl(2)	10576 (5)	7518 (3)	−1818 (3)	4·2 (1)
Cl(3)	11213 (5)	10087 (4)	−1454 (3)	3·9 (1)
N(1)	8004 (14)	9172 (10)	−1843 (10)	3·6 (2)
N(2)	11883 (14)	9028 (11)	−3206 (8)	3·3 (2)
N(3)	8838 (15)	8218 (10)	−3462 (8)	3·5 (2)
C(1)	9079 (18)	8452 (12)	−4324 (10)	3·3 (2)
C(2)	8352 (21)	7877 (16)	−4956 (12)	4·7 (3)
C(3)	7423 (20)	7037 (14)	−4753 (11)	3·1 (3)
C(4)	7193 (17)	6834 (13)	−3855 (9)	4·6 (2)
C(5)	7918 (16)	7423 (13)	−3247 (10)	3·5 (2)
C(6)	8003 (20)	8952 (14)	−1004 (11)	3·7 (3)
C(7)	6712 (19)	8992 (14)	− 505 (10)	4·3 (2)
C(8)	5400 (23)	9214 (19)	− 922 (12)	5·7 (3)
C(9)	5420 (19)	9521 (14)	−1777 (12)	4·2 (3)
C(10)	6757 (18)	9484 (16)	−2219 (10)	4·0 (2)
C(11)	12633 (20)	9897 (15)	−3411 (11)	4·0 (2)
C(12)	13930 (20)	9905 (14)	−3881 (11)	5·7 (3)
C(13)	14443 (21)	8899 (15)	−4182 (12)	4·4 (3)
C(14)	13692 (20)	7999 (15)	−3975 (11)	4·7 (3)
C(15)	12386 (20)	8062 (12)	−3478 (11)	5·9 (3)

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

Rh—Cl(1)	2·337 (5)	Rh—N(1)	2·05 (1)
Rh—Cl(2)	2·330 (4)	Rh—N(2)	2·06 (1)
Rh—Cl(3)	2·346 (5)	Rh—N(3)	2·06 (1)
N(1)—C(6)	1·32 (2)	N(1)—C(10)	1·32 (2)
C(6)—C(7)	1·41 (2)	C(7)—C(8)	1·37 (3)
C(8)—C(9)	1·37 (3)	C(9)—C(10)	1·39 (2)
N(2)—C(11)	1·32 (2)	N(2)—C(15)	1·35 (2)
C(11)—C(12)	1·38 (2)	C(12)—C(13)	1·42 (2)
C(13)—C(14)	1·35 (3)	C(14)—C(15)	1·42 (2)
N(3)—C(1)	1·38 (2)	N(3)—C(5)	1·34 (2)
C(1)—C(2)	1·37 (2)	C(2)—C(3)	1·38 (2)
C(3)—C(4)	1·43 (2)	C(4)—C(5)	1·35 (2)

Cl(1)—Rh—Cl(2)	179·4 (2)	N(1)—Rh—N(2)	179·4 (5)
Cl(1)—Rh—Cl(3)	90·9 (2)	N(1)—Rh—N(3)	89·0 (5)
Cl(1)—Rh—N(1)	90·1 (4)	N(2)—Rh—N(3)	91·1 (5)
Cl(1)—Rh—N(2)	90·4 (4)	C(6)—N(1)—C(10)	119 (1)
Cl(1)—Rh—N(3)	90·2 (4)	C(6)—N(1)—Rh	121 (1)
Cl(2)—Rh—Cl(3)	89·7 (2)	C(10)—N(1)—Rh	121 (1)
Cl(2)—Rh—N(1)	89·9 (4)	C(11)—N(2)—C(15)	119 (1)
Cl(2)—Rh—N(2)	89·5 (4)	C(11)—N(2)—Rh	122 (1)
Cl(2)—Rh—N(3)	89·2 (4)	C(15)—N(2)—Rh	119 (1)
Cl(3)—Rh—N(1)	91·6 (4)	C(1)—N(3)—C(5)	120 (1)
Cl(3)—Rh—N(2)	88·4 (4)	C(1)—N(3)—Rh	118·8 (9)
Cl(3)—Rh—N(3)	178·8 (4)	C(5)—N(3)—Rh	121·0 (9)
N(3)—C(1)—C(2)	120 (1)	C(1)—C(2)—C(3)	121 (1)
C(2)—C(3)—C(4)	117 (1)	C(3)—C(4)—C(5)	120 (1)
N(3)—C(5)—C(4)	121 (1)	N(1)—C(6)—C(7)	123 (1)
C(6)—C(7)—C(8)	118 (1)	C(7)—C(8)—C(9)	119 (1)
C(8)—C(9)—C(10)	119 (1)	N(1) C(10)—C(9)	122 (1)
N(2)—C(11)—C(12)	125 (1)	C(11)—C(12)—C(13)	117 (1)
C(12)—C(13)—C(14)	119 (1)	C(13)—C(14)—C(15)	120 (1)
N(2)—C(15)—C(14)	120 (1)		

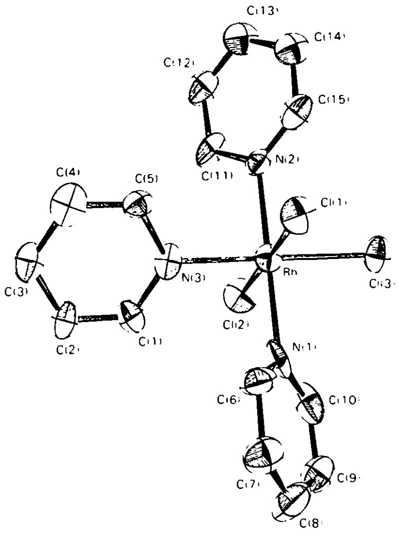

Fig. 1. An *ORTEP* drawing of Rh(py)$_3$Cl$_3$ with thermal ellipsoids scaled to 50% probability.

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