despite their differing electronic properties these groups did not induce any significant difference in the Ni—S(dithionato) bond distances.

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Structure of *trans*-Dichlorotetrakis(pyrazole-N^2)rhodium(III) Chloride

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Abstract. [RhCl$_2$(C$_3$H$_4$N$_2$)$_4$]Cl, M_r = 481.58, triclinic, $P\bar{1}$, a = 7.969 (2), b = 8.402 (2), c = 14.747 (3) Å, α = 103.24 (3), β = 90.05 (3), γ = 111.29 (3)°, V = 892 (1) Å^3, Z = 2, D_x = 1.794 g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 14.08 cm^{-1}, $F(000)$ = 480, T = 298 K, final R = 0.049 for 2817 unique observed reflections. The two *trans*-[Rh(pz)$_4$Cl$_2$]$^+$ cations are located at non-equivalent centers of symmetry in the unit cell, so that the rings which are *trans* to each other must be coplanar. The dihedral angles made by the pyrazole rings with the equatorial plane containing the Rh and the four coordinated N atoms are 124.1 (1) and 112.5 (2)° for the first cation and 107.5 (1) and 111.2 (2)° for the second cation. The cations have almost ideal octahedral geometry, with average bond lengths of 2.334 (7) and 2.038 (4) Å for Rh—Cl and Rh—N, respectively. The Cl ions (Cl3) show hydrogen bonding of the type N—H···Cl to several NH groups on adjacent cations. The H···Cl distances are 2.186 (3), 2.370 (3), 2.720 (3) and 2.395 (3) Å, while the N···Cl distances are 3.092 (3), 3.194 (3), 3.389 (3) and 3.233 (3) Å for N2, N4, N6 and N8 (or their symmetry related atoms), respectively.

Experimental. The title compound (1) was prepared during the synthesis of a series of Rh^{III} complexes

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with pyrazole and thiazole ligands. The solid product was obtained from the reaction of aqueous RhCl$_3$ with pyrazole. Suitable crystals formed by slow evaporation from acetonitrile.

(1)
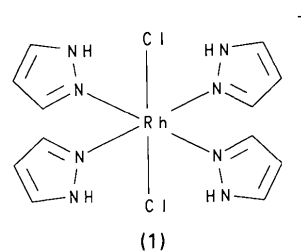
A yellow plate (D_m not determined), 0.15 × 0.20 × 0.25 mm, was used for data collection on an Enraf–Nonius CAD-4 diffractometer with monochromated Mo $K\alpha$ radiation, using ω–2θ scans of width (0.9 + 0.34tanθ)°, to $2\theta_{max}$ = 50°. Lattice parameters were determined from least-squares fit of 25 reflections in the range 10 < θ < 15°. No systematic absences were observed. Data were collected for $\pm h$, $\pm k$, $\pm l$, $-9 \le h \le 9$, $-9 \le k \le 9$, $-17 \le l \le 17$. Three standard reflections ($\bar{2}$00, 1$\bar{2}$1 and 00$\bar{4}$) showed total intensity loss of 0.7% during 41.5 h exposure. R_{int} = 0.023. Of a total of 6244 reflections measured, 2817 were unique withi F^2 > 3σ^2(F_o), where $\sigma(F_o)$ = [$\sigma^2(I_{raw})$ + (0.04F_o^2)^2]$^{1/2}$/2F_o. Absorption corrections were from ψ scans, with relative transmission factors 0.511–0.998. The structure was solved with the Enraf–

Table 1. *Positional parameters and equivalent isotropic thermal parameters* ($Å^2$) *for trans-*[Rh(pz)₄Cl₂]Cl

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Rh1	0.000	0.000	0.000	1.982 (8)
Rh2	0.500	0.000	0.500	1.784 (7)
Cl1	−0.1603 (1)	0.1833 (1)	0.04547 (6)	2.88 (2)
Cl2	0.8107 (1)	0.1655 (1)	0.51267 (6)	2.64 (2)
Cl3	0.0263 (1)	0.4089 (1)	0.29716 (8)	3.80 (2)
N1	−0.0410 (4)	−0.0815 (4)	0.1212 (2)	2.51 (7)
N2	−0.0509 (4)	0.0166 (4)	0.2051 (2)	3.04 (7)
N3	0.2362 (4)	0.2038 (4)	0.0565 (2)	2.50 (7)
N4	0.2506 (5)	0.3479 (4)	0.1219 (2)	3.53 (8)
N5	0.4843 (4)	0.0123 (3)	0.3642 (2)	2.45 (6)
N6	0.6114 (4)	0.1317 (4)	0.3289 (2)	2.68 (7)
N7	0.5487 (4)	−0.2255 (3)	0.4581 (2)	2.19 (6)
N8	0.4269 (4)	−0.3773 (4)	0.4046 (2)	2.66 (7)
C1	−0.0868 (6)	−0.0801 (5)	0.2679 (3)	3.7 (1)
C2	−0.0990 (6)	−0.2452 (5)	0.2249 (3)	3.51 (9)
C3	−0.0677 (5)	−0.2400 (5)	0.1333 (3)	3.02 (9)
C4	0.4237 (6)	0.4608 (5)	0.1391 (3)	3.9 (1)
C5	0.5239 (6)	0.3874 (5)	0.0841 (3)	3.9 (1)
C6	0.4023 (5)	0.2279 (5)	0.0324 (3)	3.21 (9)
C7	0.5658 (5)	0.1130 (5)	0.2388 (3)	3.34 (9)
C8	0.4032 (6)	−0.0214 (6)	0.2144 (3)	4.1 (1)
C9	0.3541 (6)	−0.0836 (5)	0.2937 (3)	3.5 (1)
C10	0.5027 (6)	−0.4974 (4)	0.3807 (3)	3.39 (9)
C11	0.6791 (6)	−0.4225 (5)	0.4182 (3)	3.60 (9)
C12	0.7033 (5)	−0.2514 (5)	0.4674 (3)	3.06 (8)

Table 2. *Selected bond lengths* ($Å$) *and angles* (°) *for trans-*[Rh(pz)₄Cl₂]Cl

Cation centered on Rh1		Cation centered on Rh2	
Rh1—Cl1	2.326 (1)	Rh2—Cl2	2.341 (1)
Rh1—N1	2.042 (3)	Rh2—N5	2.036 (3)
Rh1—N3	2.043 (2)	Rh2—N7	2.030 (3)
N—N(av.)	1.336 (1)	N—N(av.)	1.348 (1)
C—N(av.)	1.331 (2)	C—N(av.)	1.339 (1)
C—C(av.)	1.365 (1)	C—C(av.)	1.377 (2)
Cl1—Rh1—N1	91.2 (1)	Cl2—Rh2—N5	89.19 (8)
Cl1—Rh1—N3	90.3 (1)	Cl2—Rh2—N7	90.03 (8)
N1—Rh1—N3	92.7 (1)	N5—Rh2—N7	89.5 (1)
Rh1—N—NH(av.)	125.8 (2)	Rh2—N—NH(av.)	123.4 (1)
Rh1—N—C(av.)	128.1 (2)	Rh2—N—C(av.)	129.9 (8)
N—NH—C(av.)	110.3 (1)	N—NH—C(av.)	110.3 (2)
NH—C—C(av.)	108.2 (2)	NH—C—C(av.)	108.0 (4)
C—C—C(av.)	105.0 (2)	C—C—C(av.)	105.9 (4)
C—C—N(av.)	110.5 (1)	C—C—N(Av.)	109.1 (1)
C—N—NH(av.)	106.0 (1)	C—N—NH(av.)	106.6 (5)

Nonius *SDP* programs (Frenz, 1986), heavy-atom and difference Fourier methods, followed by iso-tropic then anisotropic refinement of F of all non-H atoms, with H atoms at calculated positions using a riding model [C—H = 0.95 Å, U(H) = $1.3U$(C)]. For 220 parameters refined, $R = 0.049$, $wR = 0.059$ {$w = 1/[\sigma^2(F_o)]$}, $S = 2.225$. Difference syntheses showed no densities above 0.42 e $Å^{-3}$ (minimum −0.46 e $Å^{-3}$). Maximum $\Delta/\sigma < 0.01$. Neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Tables 2.2B and 2.3.1.), and were corrected for anomalous dis-persion (Cromer & Liberman, 1970).

The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1, and selected bond lengths and angles in Table 2.* An *ORTEP*II plot (Johnson, 1976) of the two cations with the atom numbering is shown in Fig. 1, and the packing of the molecules (Motherwell & Clegg, 1978) in Fig. 2.

Related literature. The *trans* geometry of the complex cations is consistent with assignments for this and related complexes based on spectral data (Gillard & Wilkinson, 1967) and with the structure found for the analogous pyridine (py) complex *trans-*[Rh(py)₄Br₂]Br.6H₂O (Muir, Gómez, Muir & Sánchez, 1987). However, the symmetry of the cations does not permit a propeller-like arrangement for the heterocyclic rings, such as that observed in pyridine complexes, which have dihedral angles of about 45° (Muir *et al.*, 1987; Acharya, Tavale & Guru Row, 1984; Rozière, Lehmann & Potier, 1979). The hydrogen-bonding distances are indicated by light lines in Fig. 1, and are within the ranges indicative of hydrogen bonding of the type NH···Cl (Hamilton & Ibers, 1968). The Rh—Cl and Rh—N
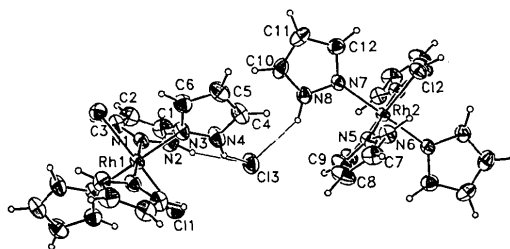
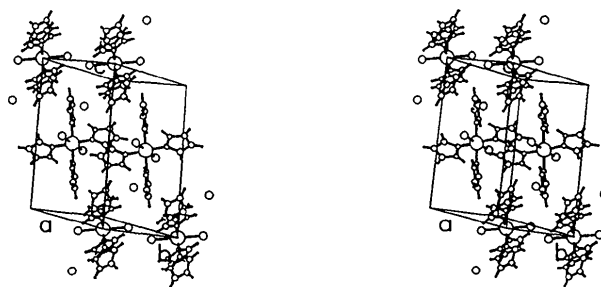Fig. 1. Labeling of atoms in the two *trans-*[Rh(pz)₄Cl₂]⁺ cations (50% probability ellipsoids).

Fig. 2. Packing diagram of *trans-*[Rh(pz)₄Cl₂]Cl molecules in the unit cell.

distances found for the cations are comparable to those found for *trans*-Cl and *trans*-N atoms in other complexes of Rh^{III} (Siripaisarnpipat & Schlemper, 1984; Muir *et al.*, 1987; Acharya *et al.*, 1984). The average C—N, N—N and C—C distances and bond angles for the two cations are different but are within the ranges found in other nitrogen heterocyclic compounds (Carmona, Lahoz, Oro, Pilar Lamata & Buzarra, 1991).

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Structure of [SbCl{Mn(CO)2(η5-C5H5)}{Co(CO)3(PPh3)}2].C6H5Cl

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Abstract. Octacarbonyl-1κ^2C,2κ^3C,3κ^3C-chloro-4κCl-[1(η^5)-cyclopentadienyl]bis(triphenylphosphine)-2κP,3κP-antimonydicobaltmanganese(2Co—Sb,-Mn—Sb) chlorobenzene solvate, [Co2MnSbCl-(C5H5)(C18H15P)2(CO)8].C6H5Cl, $M_r = 1256.32$, monoclinic, $P2_1/c$, $a = 16.632$ (4), $b = 16.906$ (3), $c = 19.332$ (3) Å, $\beta = 93.15$ (2)°, $V = 5428$ (2) Å^3, $Z = 4$, $D_x = 1.54$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 15.21$ cm^{-1}, $F(000) = 2512$, $T = 298$ K, $R = 0.062$ for 2821 unique observed reflections. The title complex contains an Sb atom in a slightly distorted tetrahedral environment, coordinated to two Co atoms [Sb—Co 2.569 (3) and 2.574 (3) Å], one Mn atom [Sb—Mn 2.460 (4) Å] and one Cl atom [Sb—Cl 2.439 (6) Å].

Experimental. The title complex was obtained in low yield from a reaction between Mn(CO)2(thf)(η-C5H5) and the crude product of a reaction between SbCl3 and two equivalents of K[Co(CO)3(PPh3)] in thf

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solution. Infrared data: ν(CO) (thf solution) 2046w, 2025m, 1986s, 1965m, 1921m and 1871m cm^{-1}. Analysis calculated for C49H35ClCo2MnO8P2Sb: C 51.5; H 3.1%. Analysis found: C 51.9; H 3.1%. Dark purple crystals were obtained from the crude reaction mixture by solvent diffusion from chlorobenzene/hexane. A crystal of dimensions *ca* 0.4 × 0.5 × 0.4 mm (systematic absences: $k = 2n + 1$ in 0*k*0; *l* = 2*n* + 1 in *h*0*l*) was used for data collection on an Enraf–Nonius CAD-4F diffractometer with graphite monochromator and in the $\theta/2\theta$-scan mode. Cell parameters were refined by least-squares methods from setting angles of 22 independent reflections ($\theta \simeq 10°$). Intensities were measured to $\theta = 22.5°$ over *hkl* range 0 to 17, 0 to 18, −20 to 20. Standard reflections $\overline{4}28$, 91$\overline{1}$ and 7$\overline{11}$ were measured every 2 h and showed 30% decay over 118.2 h of data collection. 7687 data were measured, of which 7097 were independent. 2821 data with $I > 2.0\sigma(I)$ were considered observed and used in structure determination and refinement. R_{int} before absorption correction was 0.035. Data were corrected for decomposition,