and H atoms in fixed calculated positions. At final convergence, $R = 0.0498$, $wR = 0.0691$, $S = 1.252$ for 202 parameters; $(\Delta/\sigma)_{max}$ in the final cycle was less than 0.10; maximum and minimum residues in the final ΔF synthesis were 1.53 and $-0.80\,e\,\text{Å}^{-3}$, respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0.000373F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for Rh (Cromer & Mann, 1968). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, while selected bond lengths, bond angles and torsion angles appear in Table 2.* The atom-numbering scheme for the cation is shown in Fig. 1, which was generated using the interactive version of *ORTEPII* (Johnson, 1971) included in the *GX* crystallographic program system (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55418 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0111]

Related literature. This is a rare example of a trigonal bipyramidal complex containing the [9]aneS$_3$ macrocycle. Other examples are [Rh([9]aneS$_3$)(1,5-cyclo-octadiene)]$^+$ (Blake, Halcrow & Schröder, 1991) and [Pt([9]aneS$_3$)(PPh$_3$)$_2$]$^{2+}$ (Blake, Roberts & Schröder, 1992).

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(Diisopropyldithiocarbamato-S,S')-(1,3-diphenyl-1,3-propanedithionato-S,S')nickel(II)

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Abstract. [Ni(C$_7$H$_{14}$NS$_2$)(C$_{15}$H$_{11}$S$_2$)], $M_r = 490.4$, monoclinic, $P2_1$, $a = 11.415$ (5), $b = 11.185$ (5), $c = 9.075$ (4) Å, $\beta = 96.93$ (3)°, $V = 1150$ Å^3, $Z = 2$, $D_x = 1.416$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.204$ mm^{-1}, $F(000) = 512$, $T = 298$ K, $R = 0.0537$ for 1915 unique observed reflections. The metal atom is in a distorted square-planar coordination; the Ni atom is coplanar with the four S atoms, but successive S—Ni—S angles are 77.49 (8) (within the dithiocarbamate ligand), 87.12 (9), 102.70 (9) (within the 1,3-dithionate ligand) and 92.57 (9)°. Despite its potential mirror symmetry, the molecule possesses no crystallographically imposed symmetry and shows inequivalent Ni—S distances to both ligands.

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Experimental. The title compound [NiL^1L^2] was prepared by refluxing [NiL^1_2] and [NiL^2_2] in toluene for 15 h; crystals were obtained from CH$_2$Cl$_2$/petroleum spirit (333–353 K boiling range). A red acicular crystal, $1.50 \times 0.24 \times 0.14$ mm, was used for data collection on a Stoe Stadi-2 two-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Cell

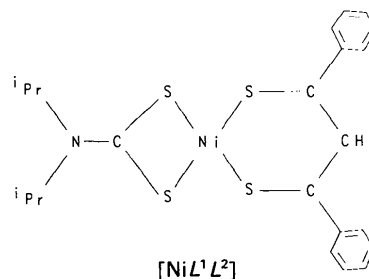[NiL^1L^2]

$[Ni(C_7H_{14}NS_2)(C_{15}H_{11}S_2)]$

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Ni	0.34869 (8)	0.2500*	0.27258 (10)	0.0415 (5)
S(1)	0.19336 (17)	0.27495 (22)	0.10003 (22)	0.0490 (12)
S(2)	0.42064 (17)	0.36686 (25)	0.10898 (23)	0.0502 (12)
S(3)	0.51438 (16)	0.26581 (24)	0.40396 (21)	0.0489 (12)
S(4)	0.25178 (18)	0.1449 (3)	0.40581 (23)	0.0502 (12)
C(1)	0.2812 (6)	0.3701 (8)	0.0111 (8)	0.042 (4)
N(1)	0.2479 (6)	0.4316 (7)	−0.1077 (7)	0.044 (4)
C(2)	0.3282 (7)	0.5172 (10)	−0.1724 (10)	0.056 (6)
C(21)	0.4316 (8)	0.4520 (10)	−0.2308 (11)	0.067 (6)
C(22)	0.3654 (9)	0.6168 (10)	−0.0691 (11)	0.069 (6)
C(3)	0.1247 (7)	0.4235 (9)	−0.1825 (9)	0.051 (5)
C(31)	0.1251 (9)	0.3687 (14)	−0.3363 (11)	0.077 (7)
C(32)	0.0618 (8)	0.5391 (12)	−0.1844 (13)	0.078 (7)
C(1S)	0.5318 (7)	0.1776 (8)	0.5542 (8)	0.044 (5)
C(2S)	0.4476 (7)	0.1097 (8)	0.6117 (8)	0.044 (5)
C(3S)	0.3294 (7)	0.0940 (8)	0.5643 (8)	0.045 (4)
C(11P)	0.2584 (5)	0.0250 (5)	0.6622 (6)	0.044 (5)
C(12P)	0.2808 (5)	0.0399 (5)	0.8156 (6)	0.059 (6)
C(13P)	0.2170 (5)	−0.0259 (5)	0.9094 (6)	0.069 (7)
C(14P)	0.1308 (5)	−0.1065 (5)	0.8498 (6)	0.064 (6)
C(15P)	0.1084 (5)	−0.1213 (5)	0.6963 (6)	0.067 (6)
C(16P)	0.1722 (5)	−0.0556 (5)	0.6025 (6)	0.058 (6)
C(21P)	0.6547 (5)	0.1775 (4)	0.6301 (6)	0.042 (5)
C(22P)	0.7079 (5)	0.2832 (4)	0.6855 (6)	0.054 (5)
C(23P)	0.8233 (5)	0.2814 (4)	0.7564 (6)	0.072 (7)
C(24P)	0.8855 (5)	0.1738 (4)	0.7721 (6)	0.074 (7)
C(25P)	0.8322 (5)	0.0681 (4)	0.7167 (6)	0.074 (7)
C(26P)	0.7168 (5)	0.0699 (4)	0.6457 (6)	0.057 (6)

* Coordinate fixed to define origin.

Table 2. *Bond lengths* (Å) *and angles* ($°$)

Ni—S(1)	2.237 (2)	N(1)—C(3)	1.489 (11)
Ni—S(2)	2.209 (2)	C(2)—C(21)	1.535 (14)
Ni—S(3)	2.118 (2)	C(2)—C(22)	1.484 (15)
Ni—S(4)	2.095 (2)	C(3)—C(31)	1.525 (15)
S(1)—C(1)	1.728 (8)	C(3)—C(32)	1.478 (15)
S(2)—C(1)	1.727 (8)	C(1S)—C(2S)	1.376 (12)
S(3)—C(1S)	1.675 (8)	C(1S)—C(21P)	1.486 (10)
S(4)—C(3S)	1.695 (8)	C(2S)—C(3S)	1.377 (12)
C(1)—N(1)	1.296 (11)	C(3S)—C(11P)	1.488 (10)
N(1)—C(2)	1.493 (12)		

S(1)—Ni—S(2)	77.49 (8)	N(1)—C(2)—C(22)	112.0 (8)
S(1)—Ni—S(3)	164.20 (9)	C(21)—C(2)—C(22)	113.7 (8)
S(1)—Ni—S(4)	92.57 (9)	N(1)—C(3)—C(31)	109.5 (8)
S(2)—Ni—S(3)	87.12 (9)	N(1)—C(3)—C(32)	112.5 (8)
S(2)—Ni—S(4)	169.93 (10)	C(31)—C(3)—C(32)	113.3 (9)
S(3)—Ni—S(4)	102.70 (9)	S(3)—C(1S)—C(2S)	127.9 (7)
Ni—S(1)—C(1)	87.0 (3)	S(3)—C(1S)—C(21P)	113.0 (5)
Ni—S(2)—C(1)	87.9 (3)	C(2S)—C(1S)—C(21P)	119.1 (7)
Ni—S(3)—C(1S)	114.8 (3)	C(1S)—C(2S)—C(3S)	130.9 (8)
Ni—S(4)—C(3S)	114.8 (3)	S(4)—C(3S)—C(2S)	127.9 (7)
S(1)—C(1)—S(2)	107.3 (4)	S(4)—C(3S)—C(11P)	114.5 (5)
S(1)—C(1)—N(1)	126.0 (6)	C(2S)—C(3S)—C(11P)	117.6 (7)
S(2)—C(1)—N(1)	126.7 (6)	C(3S)—C(11P)—C(12P)	119.1 (6)
C(1)—N(1)—C(2)	122.4 (7)	C(3S)—C(11P)—C(16P)	120.9 (6)
C(1)—N(1)—C(3)	120.8 (7)	C(1S)—C(21P)—C(22P)	120.8 (5)
C(2)—N(1)—C(3)	116.8 (7)	C(1S)—C(21P)—C(26P)	119.2 (5)
N(1)—C(2)—C(21)	111.4 (8)		

parameters were derived from the setting angles of ten reflections with $18 \le 2\theta \le 32°$. For data collection at $T = 298$ K, ω scans with scan width [2.0 + 1.0(sinμ/tanθ)]° were made to $2\theta_{max} = 50°$, and for h $-13 \rightarrow 13$, k $0 \rightarrow 12$, l $0 \rightarrow 10$. No standard reflections were monitored, and no absorption correction was made. 2183 unique reflections were measured, giving 1915 with $F \ge 6\sigma(F)$ for structure solution. The

Ni-atom position, located in a Patterson synthesis, was input to *DIRDIF* (Beurskens, Bosman, Doesburg, van den Hark, Prick, Noordik, Beurskens, Gould & Parthasarathi, 1983) which revealed the remaining non-H atoms. Refinement used full-matrix least squares on F (*SHELX*76; Sheldrick, 1976). Anisotropic thermal parameters were refined for all non-H atoms; phenyl rings were refined with idealized D_{3h} symmetry; H atoms were in fixed calculated positions. At final convergence, R = 0.0537, $wR = 0.0636$, $S = 1.380$ for 228 parameters. $(\Delta/\sigma)_{max}$ in the final cycle was 0.01; maximum and minimum residues in the final ΔF synthesis were 0.60 and -1.51 e Å^{-3}, respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0.000924F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for Ni (Cromer & Mann, 1968). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, while selected bond lengths and valence angles appear in Table 2.* The atom-numbering scheme for the molecule is shown in Fig. 1, which was generated using the interactive version of *ORTEP*II (Johnson, 1971) included in the *GX* crystallographic program system (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Related literature. We have previously reported the structure of the related species in which the phenyl substituents were replaced by one CH$_3$— and one CF$_3$— group (Anderson, Blake & Heath, 1989) but

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55453 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1008]
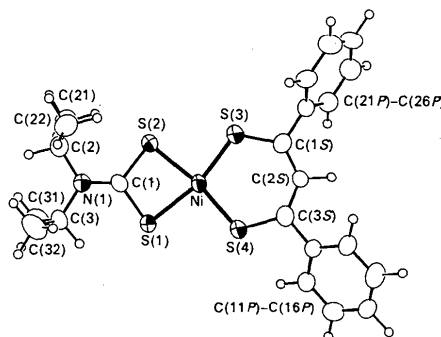

Fig. 1. A general view of the molecule showing the atom-numbering scheme. Thermal ellipsoids are drawn at the 30% probability level, except those of H which have artificial radii of 0.10 Å for clarity.

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)
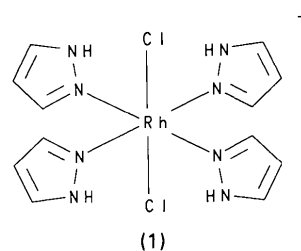
A yellow plate (D_m not determined), 0.15 × 0.20 × 0.25 mm, was used for data collection on an Enraf-Nonius CAD-4 diffractometer with monochromated Mo $K\alpha$ radiation, using ω–2θ scans of width (0.9 + 0.34tanθ)°, to $2\theta_{max}$ = 50°. Lattice parameters were determined from least-squares fit of 25 reflections in the range 10 < θ < 15°. No systematic absences were observed. Data were collected for ±h, ±k, ±l, −9 ≤ h ≤ 9, −9 ≤ k ≤ 9, −17 ≤ l ≤ 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-