Acta Cryst. (1989). C**45**, 1430–1431

Structure of (Diisopropyldithiocarbamato)(1,1,1-trifluoro-2,4-pentanedithionato)-nickel(II)

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(*Received* 14 *October* 1988; *accepted* 26 *April* 1989)

Abstract. [$\mathrm{Ni(C_5H_4F_3S_2)(C_7H_{14}NS_2)}$], M_r = 420·20, orthorhombic, *Pbca*, *a* = 23·007 (16), *b* = 14·918 (11), *c* = 10·761 (10) Å, *V* = 3693 Å^3, *Z* = 8, D_x = 1·511 Mg m^{-3}, λ(Mo *Kα*) = 0·71073 Å, μ = 1·508 mm^{-1}, *F*(000) = 1728, *T* = 293 K, *R* = 0·0813 for 1332 unique observed reflections. The Ni atom is coordinated by a plane of four S atoms, but these are markedly distorted from square-planarity as a result of the differing bites of the ligands which form the four- and six-membered chelate rings: for the dithiocarbamate S donors the S—Ni—S angle is 77·52 (14)°, while for the pentanedithione it is 101·71 (15)°. The different electronic properties of the $\mathrm{CF_3}$ and $\mathrm{CH_3}$ groups on the pentanedithione do not result in significantly different bond lengths between its two S donors and the metal centre.

Experimental. Compound [Ni(L^1)(L^2)] prepared by refluxing NiL^1_2 and NiL^2_2 together in toluene, where L^1 and L^2 are 1,1,1-trifluoro-2,4-pentanedithione and diisopropyldithiocarbamate. For crystallographic work, this material was recrystallized from dichloromethane/petroleum spirit (60–80° boiling fraction). Lath-shaped dark-red crystal, 0·76 × 0·16 × 0·10 mm, Stadi-2 two-circle diffractometer, graphite-monochromated Mo *Kα* X-radiation, cell parameters from 14 *hk*0 and 3 00*l* centred reflections. For data collection, ω scans with width [1·0 + 0·5(sinμ/tanθ)]°, $2\theta_{\max}$ = 50°, *h* 0→27, *k* 0→17, *l* 0→12, no significant crystal movement or decay, no absorption correction, 3412 reflections, giving 1332 with $F \geq 4\sigma(F)$ for structure solution [the Ni position from a Patterson synthesis was input to *DIRDIF* (Beurskens *et al.*, 1983) which located the S atoms: thereafter iterative cycles of least-squares refinement and difference Fourier synthesis located the remaining non-H atoms] and refinement [using full-matrix least squares on *F* (Sheldrick, 1976)]. The $\mathrm{CF_3}$ group was found to be disordered and this was modelled by refining two orientations for the group: residual disorder is the most likely reason for the slightly high *R* values quoted below. Anisotropic thermal parameters for all ordered non-H atoms, H atoms refined in fixed, calculated positions. At convergence, *R*, *wR*

Table 1. *Atomic coordinates with e.s.d.'s in parentheses*

$U_{\rm eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	*y*	*z*	$U_{\rm iso}$ or $U_{\rm eq}$(Å^2)
Ni(1)	0·06796 (6)	0·12628 (10)	0·06842 (15)	0·0785 (10)
S(1)	0·08709 (14)	0·18311 (24)	0·2550 (3)	0·094 (3)
S(2)	0·14750 (14)	0·05226 (25)	0·1238 (4)	0·100 (3)
S(3)	0·06425 (16)	0·05700 (23)	−0·1038 (3)	0·092 (3)
S(4)	−0·00632 (14)	0·20900 (21)	0·0574 (4)	0·091 (3)
C(1)	0·1459 (5)	0·1130 (8)	0·2589 (14)	0·086 (10)
N(1)	0·1818 (5)	0·1077 (8)	0·3564 (14)	0·091 (10)
C(2)	0·1715 (7)	0·1640 (13)	0·4707 (17)	0·118 (15)
C(21)	0·1512 (8)	0·1105 (12)	0·5709 (18)	0·183 (19)
C(22)	0·2167 (7)	0·2268 (11)	0·4918 (17)	0·197 (20)
C(3)	0·2324 (7)	0·0485 (10)	0·3548 (18)	0·125 (14)
C(31)	0·2196 (6)	−0·0486 (9)	0·3580 (13)	0·129 (13)
C(32)	0·2803 (6)	0·0761 (10)	0·2756 (15)	0·142 (14)
C(1S)	−0·0460 (5)	0·1942 (10)	−0·0738 (16)	0·091 (11)
C(2S)	−0·0368 (6)	0·1388 (10)	−0·1722 (14)	0·087 (11)
C(3S)	0·0085 (6)	0·0800 (9)	−0·1956 (14)	0·091 (11)
C(4)	−0·1031 (9)	0·2560 (14)	−0·0736 (19)	0·135 (16)
C(5)	0·0053 (5)	0·0314 (8)	−0·3176 (14)	0·108 (12)
F(1)	−0·1271 (6)	0·2630 (11)	0·0344 (17)	0·114 (7)
F(2)	−0·1412 (6)	0·2247 (10)	−0·1584 (14)	0·101 (7)
F(3)	−0·0853 (7)	0·3371 (12)	−0·1135 (22)	0·142 (8)
F(1′)	−0·0957 (9)	0·3326 (16)	−0·018 (3)	0·115 (11)
F(2′)	−0·1140 (14)	0·2785 (23)	−0·186 (3)	0·182 (14)
F(3′)	−0·1399 (11)	0·2063 (22)	−0·015 (3)	0·150 (12)

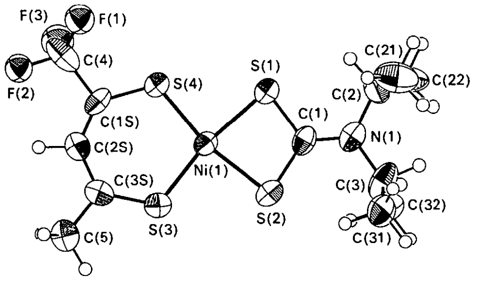

Fig. 1. View of the molecule showing atom-numbering scheme. Thermal ellipsoids are drawn at the 30% probability level, excepting those of H which have artificial radii of 0·10 Å for clarity. Only one orientation of the disordered $\mathrm{CF_3}$ group is shown.

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0108-2701/89/091430-02$03.00

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

Ni(1)—S(1)	2·224 (4)	C(3)—C(31)	1·479 (22)
Ni(1)—S(2)	2·219 (4)	C(3)—C(32)	1·453 (23)
Ni(1)—S(3)	2·124 (4)	C(1S)—C(2S)	1·359 (21)
Ni(1)—S(4)	2·111 (4)	C(1S)—C(4)	1·61 (3)
S(1)—C(1)	1·711 (14)	C(2S)—C(3S)	1·385 (21)
S(2)—C(1)	1·713 (14)	C(3S)—C(5)	1·502 (20)
S(3)—C(3S)	1·654 (15)	C(4)—F(1)	1·29 (3)
S(4)—C(1S)	1·696 (15)	C(4)—F(2)	1·35 (3)
C(1)—N(1)	1·337 (18)	C(4)—F(3)	1·35 (3)
N(1)—C(2)	1·508 (22)	C(4)—F(1′)	1·30 (3)
N(1)—C(3)	1·463 (21)	C(4)—F(2′)	1·28 (4)
C(2)—C(21)	1·42 (3)	C(4)—F(3′)	1·29 (4)
C(2)—C(22)	1·417 (25)		

S(1)—Ni(1)—S(2)	77·52 (14)	C(31)—C(3)—C(32)	116·3 (14)
S(1)—Ni(1)—S(3)	168·98 (15)	S(4)—C(1S)—C(2S)	130·1 (12)
S(1)—Ni(1)—S(4)	89·31 (14)	S(4)—C(1S)—C(4)	111·4 (11)
S(2)—Ni(1)—S(3)	91·46 (14)	C(2S)—C(1S)—C(4)	118·6 (14)
S(2)—Ni(1)—S(4)	166·78 (15)	C(1S)—C(2S)—C(3S)	130·1 (14)
S(3)—Ni(1)—S(4)	101·71 (15)	S(3)—C(3S)—C(2S)	127·3 (12)
Ni(1)—S(1)—C(1)	86·9 (5)	S(3)—C(3S)—C(5)	117·4 (10)
Ni(1)—S(2)—C(1)	87·0 (5)	C(2S)—C(3S)—C(5)	115·3 (13)
Ni(1)—S(3)—C(3S)	116·8 (5)	C(1S)—C(4)—F(1)	113·5 (16)
Ni(1)—S(4)—C(1S)	114·0 (5)	C(1S)—C(4)—F(2)	109·4 (15)
S(1)—C(1)—S(2)	108·6 (7)	C(1S)—C(4)—F(3)	105·5 (16)
S(1)—C(1)—N(1)	122·9 (10)	C(1S)—C(4)—F(1′)	113·5 (18)
S(2)—C(1)—N(1)	128·4 (11)	C(1S)—C(4)—F(2′)	108·0 (21)
C(1)—N(1)—C(2)	120·7 (13)	C(1S)—C(4)—F(3′)	101·9 (19)
C(1)—N(1)—C(3)	121·2 (13)	F(1)—C(4)—F(2)	111·0 (18)
C(2)—N(1)—C(3)	118·0 (13)	F(1)—C(4)—F(3)	110·2 (18)
N(1)—C(2)—C(21)	111·0 (15)	F(2)—C(4)—F(3)	107·0 (18)
N(1)—C(2)—C(22)	112·6 (15)	F(1′)—C(4)—F(2′)	103·6 (24)
C(21)—C(2)—C(22)	119·4 (16)	F(1′)—C(4)—F(3′)	111·4 (23)
N(1)—C(3)—C(31)	115·6 (13)	F(2′)—C(4)—F(3′)	118·8 (26)
N(1)—C(3)—C(32)	116·1 (14)		

$= 0{\cdot}0831$, 0·0662, $S = 1{\cdot}150$ for 188 parameters, $(\Delta/\sigma)_{\max}$ in final cycle 0·05, max. and min. residues in final difference Fourier synthesis 0·53, $-0{\cdot}43$ e Å^{-3} respectively. The weighting scheme, $w^{-1} = \sigma^2(F) + 0{\cdot}000225F^2$, gave satisfactory agreement analysis. 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 angles appear in Table 2.* The atom-numbering scheme for the molecule is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

We thank the SERC for support.

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* Lists of structure factors, torsion angles, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51909 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1989). C**45**, 1431–1433

Structure of (*tert*-Butylisonitrile)(chloro)(1-methyl-2-phenylimidazolato-$C^{2\prime}$,N^3)-palladium(II) Dichloromethane Solvate at 213 K

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(*Received* 17 *January* 1989; *accepted* 11 *May* 1989)

Abstract. $[Pd(C_5H_9N)(C_{10}H_9N_2)Cl].CH_2Cl_2$, $M_r =$ 934·23, monoclinic, $P2_1/c$, $a = 13{\cdot}689$ (3), $b =$ 24·432 (2), $c = 12{\cdot}111$ (2) Å, $\beta = 107{\cdot}57$ (3)°, $V =$ 3861·6 Å^3, $Z = 8$, $D_x = 1{\cdot}607$ g cm^{-3}, Mo $K\alpha$, $\lambda =$ 0·71073 Å, $\mu = 13{\cdot}6$ cm^{-1}, $F(000) = 1872$, $T =$ 213 K. The final R value is 0·034 for 3177 significant

0108-2701/89/091431-03$03.00