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Tetrakis(1*H*-pyrazole-κN^2)bis(thiocyanato-κN)nickel(II)

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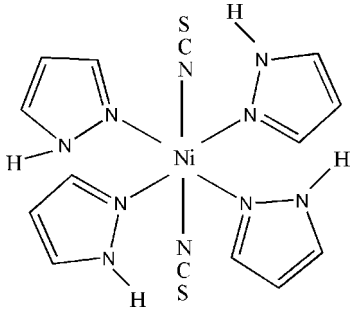
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.035; wR factor = 0.093; data-to-parameter ratio = 17.6.

In the title mononuclear complex, $[Ni(NCS)_2(C_3H_4N_2)_4]$, the six-coordinate Ni^{II} atom assumes an octahedral geometry and is located on an inversion centre. The crystal packing is stabilized by N—H···S, N—H···N and C—H···S hydrogen bonds.

Related literature

For related crystal structures, see: Zarbaa *et al.* (2004); Shi *et al.* (2006).



Experimental

Crystal data

$[Ni(NCS)_2(C_3H_4N_2)_4]$
M_r = 447.20
Monoclinic, $C2/c$
a = 14.046 (3) Å
b = 10.863 (2) Å
c = 14.862 (3) Å
β = 117.485 (2)°
V = 2011.6 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.19 mm^{-1}
T = 298 (2) K
0.48 × 0.26 × 0.21 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.598, T_{max} = 0.788
5718 measured reflections
2188 independent reflections
1867 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.093
S = 1.06
2188 reflections
124 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N3	2.0716 (16)	Ni1—N1	2.1120 (17)
Ni1—N4	2.1020 (17)		
N3—Ni1—N4	89.54 (8)	N4—Ni1—N1	90.47 (7)
N3—Ni1—N1	89.59 (7)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···N3^{i}	0.84	2.40	2.966 (3)	125
N2—H1···S1^{ii}	0.84	2.71	3.346 (2)	134
C6—H6···S1^{iii}	0.93	2.85	3.686 (3)	150

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z$; (ii) $x-\frac{1}{2}, y+\frac{1}{2}, z$; (iii) $x, y+1, z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2144).

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supplementary materials

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Tetrakis(1*H*-pyrazole-κN^2)bis(thiocyanato-κN)nickel(II)

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Comment

Metal complexes containing thiocyanate with pyrazole and its derivatives as ligands play a pivotal role in the area of modern coordination chemistry (Zarbaa *et al.*, 2004; Shi *et al.*, 2006). The interest in this area prompted us to synthesize the title complex, and here we report its crystal structure (Fig. 1).

The Ni^{II} atom lies on an inversion centre and assumes a slightly distorted octahedral geometry (Table 1). Table 2 and Fig. 2 give information on N—H···S, N—H···N and C—H···S hydrogen bonds, which form a supramolecular three-dimensional structure.

Experimental

Pyrazole (0.1053 g, 1.55 mmol), NaNCS (0.0711 g, 0.877 mmol) and $Ni(ClO_4)_2{\cdot}6H_2O$ (0.1568 g, 0.429 mmol) were dissolved in 3 × 5 ml H_2O, and the three solutions were mixed together and stirred for a few minutes. Blue single crystals were obtained after the mixed solution had been allowed to stand at room temperature for two weeks.

Refinement

H atoms of N—H were located in a difference Fourier map and refined as riding in their as-found positions, with $U_{iso}(H) = 1.2U_{eq}(N)$. Other H atoms were placed in calculated positions, and refined as riding, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

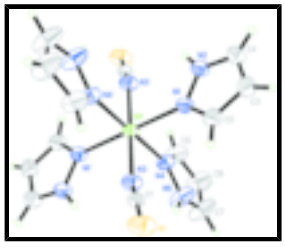

Fig. 1. The molecular structure of (I) showing the atom numbering scheme with displacement ellipsoids drawn at the 30% probability level. [Symmetry code: (i) −*x* + 1/2, −*y* + 3/2, −*z*].

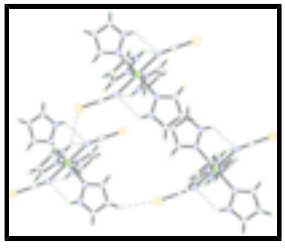

Fig. 2. The hydrogen bonding (dashed lines).

Tetrakis(1*H*-pyrazole-κ*N*^{2})bis(thiocyanato-κ*N*)nickel(II)

Crystal data

$[Ni(NCS)_2(C_3H_4N_2)_4]$	F_{000} = 920
M_r = 447.20	D_x = 1.477 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 2696 reflections
a = 14.046 (3) Å	θ = 2.5–26.7º
b = 10.863 (2) Å	μ = 1.19 mm^{-1}
c = 14.862 (3) Å	*T* = 298 (2) K
β = 117.485 (2)º	Bar, blue
V = 2011.6 (7) Å^3	0.48 × 0.26 × 0.21 mm
Z = 4	

Data collection

Bruker SMART APEX CCD diffractometer	2188 independent reflections
Radiation source: fine-focus sealed tube	1867 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 298(2) K	θ_{max} = 27.0º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −17→17
T_{min} = 0.598, T_{max} = 0.788	*k* = −13→9
5718 measured reflections	*l* = −18→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.035	H-atom parameters constrained
$wR(F^2)$ = 0.093	$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.6564P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$
2188 reflections	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
124 parameters	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.2500	0.7500	0.0000	0.04033 (14)
S1	0.45000 (6)	0.38423 (6)	0.09641 (6)	0.0763 (2)
N1	0.26313 (13)	0.71955 (15)	0.14564 (13)	0.0470 (4)
N3	0.36411 (14)	0.61469 (16)	0.02753 (14)	0.0539 (4)
N2	0.20074 (15)	0.78111 (17)	0.17699 (14)	0.0529 (4)
H1	0.1537	0.8281	0.1356	0.064*
C1	0.40149 (15)	0.51988 (19)	0.05644 (14)	0.0442 (4)
N4	0.37288 (14)	0.88231 (16)	0.05969 (14)	0.0519 (4)
C2	0.32207 (17)	0.6455 (2)	0.22228 (16)	0.0559 (5)
H2	0.3734	0.5911	0.2226	0.067*
N5	0.47618 (16)	0.8519 (2)	0.10443 (19)	0.0836 (7)
H8	0.4916	0.7781	0.1016	0.100*
C5	0.3738 (2)	1.0024 (2)	0.0677 (2)	0.0794 (8)
H5	0.3127	1.0514	0.0437	0.095*
C3	0.2974 (2)	0.6598 (2)	0.30146 (18)	0.0671 (6)
H3	0.3281	0.6189	0.3634	0.081*
C4	0.2183 (2)	0.7468 (2)	0.2695 (2)	0.0658 (7)
H4	0.1835	0.7764	0.3053	0.079*
C7	0.5409 (2)	0.9486 (3)	0.1390 (3)	0.1087 (12)
H7	0.6155	0.9476	0.1722	0.130*
C6	0.4781 (3)	1.0467 (3)	0.1170 (3)	0.1089 (12)
H6	0.4996	1.1284	0.1315	0.131*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0391 (2)	0.0375 (2)	0.0467 (2)	0.00696 (12)	0.02175 (16)	0.00747 (13)
S1	0.0814 (5)	0.0640 (4)	0.0994 (5)	0.0357 (3)	0.0553 (4)	0.0367 (3)
N1	0.0448 (9)	0.0493 (9)	0.0499 (9)	0.0059 (7)	0.0245 (8)	0.0063 (7)
N3	0.0521 (10)	0.0495 (10)	0.0652 (11)	0.0147 (8)	0.0313 (9)	0.0107 (8)
N2	0.0497 (10)	0.0582 (10)	0.0568 (10)	0.0081 (8)	0.0295 (9)	0.0064 (8)
C1	0.0416 (10)	0.0532 (12)	0.0426 (10)	0.0075 (8)	0.0235 (8)	0.0053 (8)

N4	0.0457 (9)	0.0501 (10)	0.0572 (10)	0.0012 (7)	0.0213 (8)	0.0070 (8)
C2	0.0537 (12)	0.0603 (13)	0.0536 (12)	0.0111 (9)	0.0246 (10)	0.0127 (9)
N5	0.0479 (11)	0.0571 (12)	0.120 (2)	0.0010 (9)	0.0165 (12)	0.0125 (12)
C5	0.0638 (15)	0.0527 (14)	0.117 (2)	−0.0021 (11)	0.0375 (16)	−0.0044 (14)
C3	0.0697 (15)	0.0800 (16)	0.0511 (12)	0.0017 (12)	0.0273 (11)	0.0135 (11)
C4	0.0672 (15)	0.0827 (18)	0.0581 (14)	−0.0035 (11)	0.0379 (13)	−0.0009 (11)
C7	0.0530 (15)	0.0742 (19)	0.160 (3)	−0.0144 (14)	0.0157 (18)	0.001 (2)
C6	0.081 (2)	0.0628 (18)	0.161 (3)	−0.0235 (16)	0.037 (2)	−0.015 (2)

Geometric parameters (Å, °)

Ni1—N3^{i}	2.0716 (16)	N4—N5	1.329 (3)
Ni1—N3	2.0716 (16)	C2—C3	1.381 (3)
Ni1—N4^{i}	2.1020 (17)	C2—H2	0.930
Ni1—N4	2.1020 (17)	N5—C7	1.327 (3)
Ni1—N1^{i}	2.1120 (17)	N5—H8	0.8361
Ni1—N1	2.1120 (17)	C5—C6	1.387 (4)
S1—C1	1.617 (2)	C5—H5	0.930
N1—C2	1.326 (2)	C3—C4	1.366 (4)
N1—N2	1.345 (2)	C3—H3	0.930
N3—C1	1.146 (2)	C4—H4	0.930
N2—C4	1.333 (3)	C7—C6	1.324 (4)
N2—H1	0.8364	C7—H7	0.930
N4—C5	1.309 (3)	C6—H6	0.930
N3^{i}—Ni1—N3	180.0	C5—N4—Ni1	133.71 (17)
N3^{i}—Ni1—N4^{i}	89.54 (8)	N5—N4—Ni1	122.36 (14)
N3—Ni1—N4^{i}	90.46 (8)	N1—C2—C3	111.2 (2)
N3^{i}—Ni1—N4	90.46 (8)	N1—C2—H2	124.4
N3—Ni1—N4	89.54 (8)	C3—C2—H2	124.4
N4^{i}—Ni1—N4	180.00 (7)	C7—N5—N4	113.0 (2)
N3^{i}—Ni1—N1^{i}	89.59 (7)	C7—N5—H8	129.1
N3—Ni1—N1^{i}	90.41 (7)	N4—N5—H8	117.5
N4^{i}—Ni1—N1^{i}	90.47 (7)	N4—C5—C6	110.9 (3)
N4—Ni1—N1^{i}	89.53 (7)	N4—C5—H5	124.6
N3^{i}—Ni1—N1	90.41 (7)	C6—C5—H5	124.6
N3—Ni1—N1	89.59 (7)	C4—C3—C2	105.4 (2)
N4^{i}—Ni1—N1	89.53 (7)	C4—C3—H3	127.3
N4—Ni1—N1	90.47 (7)	C2—C3—H3	127.3
N1^{i}—Ni1—N1	180.00 (9)	N2—C4—C3	106.6 (2)
C2—N1—N2	104.61 (17)	N2—C4—H4	126.7
C2—N1—Ni1	134.41 (15)	C3—C4—H4	126.7
N2—N1—Ni1	120.96 (13)	C6—C7—N5	106.4 (3)
C1—N3—Ni1	152.96 (16)	C6—C7—H7	126.8
C4—N2—N1	112.21 (18)	N5—C7—H7	126.8
C4—N2—H1	129.5	C7—C6—C5	105.8 (3)
N1—N2—H1	117.9	C7—C6—H6	127.1

N3—C1—S1	177.92 (19)	C5—C6—H6	127.1
C5—N4—N5	103.89 (19)		
$N3^{i}$—Ni1—N1—C2	−178.9 (2)	N1—Ni1—N4—C5	−95.1 (3)
N3—Ni1—N1—C2	1.1 (2)	$N3^{i}$—Ni1—N4—N5	172.6 (2)
$N4^{i}$—Ni1—N1—C2	91.5 (2)	N3—Ni1—N4—N5	−7.4 (2)
N4—Ni1—N1—C2	−88.5 (2)	$N1^{i}$—Ni1—N4—N5	−97.82 (19)
$N3^{i}$—Ni1—N1—N2	2.81 (16)	N1—Ni1—N4—N5	82.18 (19)
N3—Ni1—N1—N2	−177.19 (16)	N2—N1—C2—C3	0.1 (3)
$N4^{i}$—Ni1—N1—N2	−86.73 (16)	Ni1—N1—C2—C3	−178.42 (16)
N4—Ni1—N1—N2	93.27 (16)	C5—N4—N5—C7	−0.5 (4)
$N4^{i}$—Ni1—N3—C1	−45.5 (4)	Ni1—N4—N5—C7	−178.5 (2)
N4—Ni1—N3—C1	134.5 (4)	N5—N4—C5—C6	0.5 (4)
$N1^{i}$—Ni1—N3—C1	−136.0 (4)	Ni1—N4—C5—C6	178.1 (2)
N1—Ni1—N3—C1	44.0 (4)	N1—C2—C3—C4	0.5 (3)
C2—N1—N2—C4	−0.7 (3)	N1—N2—C4—C3	1.0 (3)
Ni1—N1—N2—C4	178.07 (15)	C2—C3—C4—N2	−0.9 (3)
$N3^{i}$—Ni1—N4—C5	−4.7 (3)	N4—N5—C7—C6	0.4 (4)
N3—Ni1—N4—C5	175.3 (3)	N5—C7—C6—C5	−0.1 (5)
$N1^{i}$—Ni1—N4—C5	84.9 (3)	N4—C5—C6—C7	−0.2 (5)

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···$N3^{i}$	0.84	2.40	2.966 (3)	125
N2—H1···$S1^{ii}$	0.84	2.71	3.346 (2)	134
C6—H6···$S1^{iii}$	0.93	2.85	3.686 (3)	150

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*; (ii) *x*−1/2, *y*+1/2, *z*; (iii) *x*, *y*+1, *z*.

Fig. 1

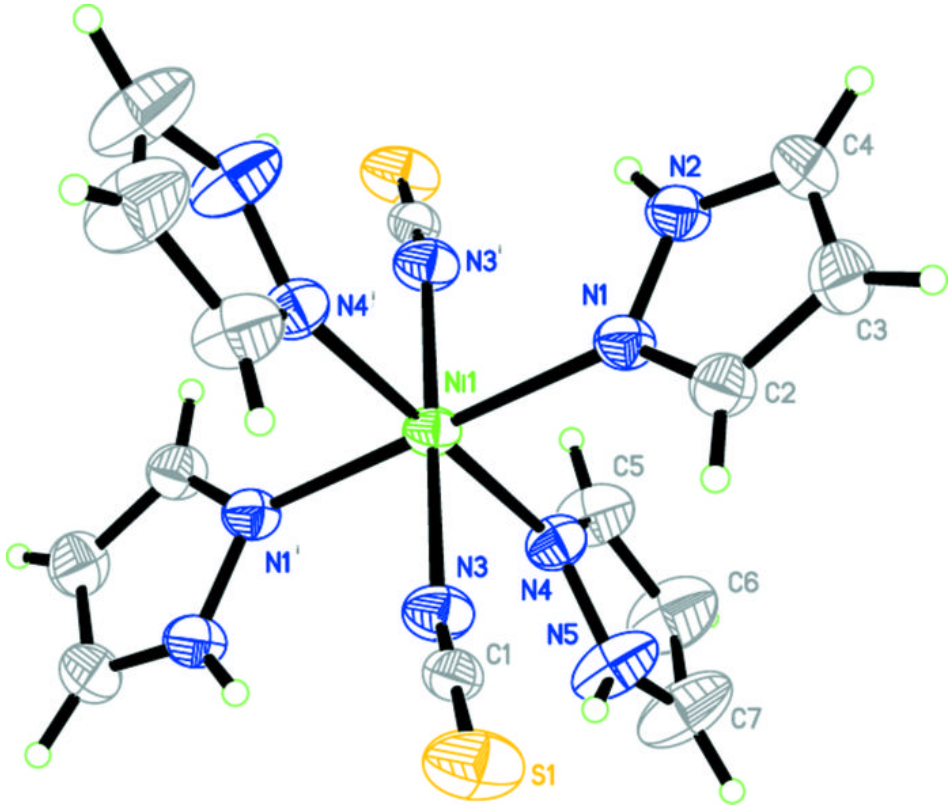

Fig. 2