

References

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A Second Triclinic Polymorph of Bis[1,2-bis(*n*-butylthio)ethylene-1,2-dithiolato-*S,S'*]-nickel

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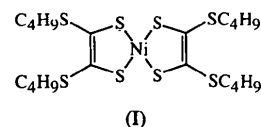
Abstract

The title compound, $[\text{Ni}(\text{C}_{10}\text{H}_{18}\text{S}_4)_2]$, contains discrete centrosymmetric molecules and is a polymorph of a previously reported structure [Charlton, Hill, Underhill, Malik, Hursthouse, Karaulov & Møller (1994). *J. Mater. Chem.* **4**, 1861–1866]. Unlike this earlier form, the title molecule, including the alkyl chains, is essentially planar and contains no short $\text{S}\cdots\text{S}$ intermolecular contacts.

Comment

The title compound, (I), was prepared during the attempted preparation of a nickel complex from a bis(tetrathiafulvalene)thiolate (Le Narvor, Robertson, Wallace, Kilburn, Underhill, Bartlett & Webster, 1996).

The reaction product was not the target species and an inspection of the Cambridge Structural Database (Allen *et al.*, 1991) revealed a compound with the same molecular formula (CCDB refcode: YISDUZ) (Charlton, Hill, Underhill, Malik, Hursthouse, Karaulov & Møller, 1994), which was described as a green solid. The two unit cells were not related and we have here an example of polymorphism.



The crystal structure of (I) is found to contain discrete centrosymmetric molecules (Fig. 1). The atom-labelling scheme has been chosen to match that of Charlton *et al.* (1994) and selected bond distances and angles are listed in Table 2. Not surprisingly, the bond lengths and angles of the two studies agree well and the major differences are in the way that the molecules pack together and in the conformation of one of the chains. The green form (Charlton *et al.*, 1994) has one C_4 chain coplanar with the $\text{Ni}(\text{S}_2\text{C}_2\text{S}_2)$ mean plane and one twisted out of the plane (maximum deviation 4.02 Å), whereas in the brown form (present work), both C_4 residues are essentially coplanar, with a maximum deviation of 0.223 (6) Å from the plane defined by the Ni1, S1, S2, S3, S4, C1 and C6 atoms. There are no short $\text{S}\cdots\text{S}$ intermolecular distances, the shortest being $\text{S2}\cdots\text{S3}(x, y, 1+z)$ of 3.885 (1) Å. The effect of crystal packing on molecular geometry has been discussed recently (Martín & Orpen, 1996) and in the present example, the major differences are in the torsion angles.

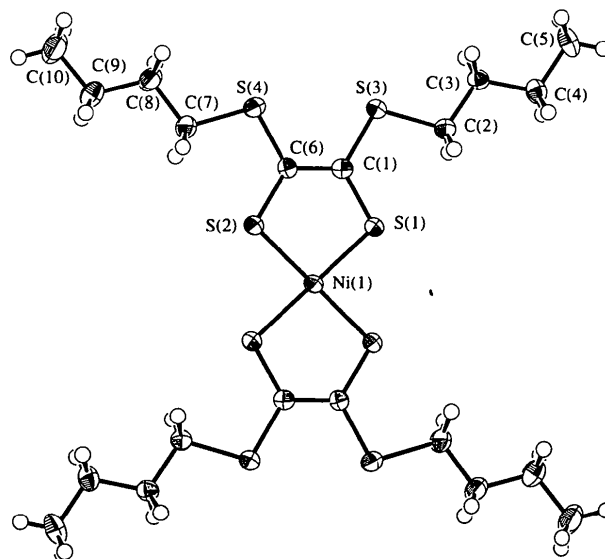


Fig. 1. The molecular structure of $[\text{Ni}(\text{C}_{10}\text{H}_{18}\text{S}_4)_2]$ showing the atom-labelling scheme and displacement ellipsoids drawn at the 60% probability level.

Experimental

Recrystallization of the crude reaction mixture from the attempted preparation of a nickel complex from a bis(tetrathiafulvalene)thiolate (Le Narvor, Robertson, Wallace, Kilburn, Underhill, Bartlett & Webster, 1996) from dichloromethane-diethyl ether solution afforded the title compound as dark brown needles and the structure was established during the subsequent X-ray examination.

Crystal data

[Ni(C₁₀H₁₈S₄)₂]

M_r = 591.68

Triclinic

*P*1

a = 8.7043 (10) Å

b = 14.988 (2) Å

c = 5.5288 (6) Å

α = 93.431 (11)°

β = 99.220 (9)°

γ = 100.388 (10)°

V = 697.4 (2) Å³

Z = 1

D_x = 1.409 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71069 Å

Cell parameters from 25 reflections

θ = 23.6–24.7°

μ = 1.307 mm⁻¹

T = 150 (2) K

Lath

0.8 × 0.3 × 0.1 mm

Dark brown

Data collection

Rigaku AFC-7S diffractometer

ω/2θ scans

Absorption correction:

ψ scans (North, Phillips & Mathews, 1968)

T_{min} = 0.64, *T_{max}* = 0.85

2641 measured reflections

2462 independent reflections

1916 observed reflections

[*I* > 2σ(*I*)]

R_{int} = 0.0234

θ_{max} = 24.99°

h = 0 → 10

k = -17 → 17

l = -6 → 6

3 standard reflections

monitored every 150

reflections

intensity decay: none

Refinement

Refinement on *F*²

R(*F*) = 0.0343

w*R*(*F*²) = 0.1047

S = 1.067

2461 reflections

188 parameters

Only coordinates of H atoms refined

w = 1/[σ²(*F_o*²) + (0.0619*P*)² + 0.2155*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.083

Δρ_{max} = 0.760 e Å⁻³

Δρ_{min} = -0.597 e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Ni1	0	0	0	0.0192 (2)
S1	-0.14454 (9)	0.04280 (5)	-0.30260 (14)	0.0230 (2)
S2	0.08008 (9)	0.13582 (5)	0.16301 (14)	0.0225 (2)
S3	-0.20508 (9)	0.22554 (5)	-0.45067 (14)	0.0241 (2)
S4	0.01961 (10)	0.31910 (5)	0.0243 (2)	0.0267 (2)
C1	-0.1129 (4)	0.1583 (2)	-0.2450 (6)	0.0206 (6)
C2	-0.3337 (4)	0.1424 (2)	-0.6851 (6)	0.0241 (7)

C3	-0.4238 (4)	0.1943 (2)	-0.8691 (6)	0.0235 (7)
C4	-0.5291 (4)	0.1317 (3)	-1.0789 (7)	0.0288 (8)
C5	-0.6204 (5)	0.1836 (3)	-1.2608 (8)	0.0397 (9)
C6	-0.0113 (4)	0.2005 (2)	-0.0360 (6)	0.0197 (6)
C7	0.1637 (4)	0.3403 (2)	0.3076 (7)	0.0281 (7)
C8	0.1958 (5)	0.4402 (2)	0.3996 (7)	0.0328 (8)
C9	0.3127 (5)	0.4602 (3)	0.6390 (7)	0.0350 (8)
C10	0.3426 (6)	0.5586 (3)	0.7439 (9)	0.0473 (11)

Table 2. Selected geometric parameters (Å, °)

Ni1—S2	2.1236 (8)	S3—C2	1.823 (3)
Ni1—S1	2.1303 (8)	S4—C6	1.752 (3)
S1—C1	1.706 (3)	S4—C7	1.815 (3)
S2—C6	1.706 (3)	C1—C6	1.378 (5)
S3—C1	1.757 (3)		
S2—Ni1—S1	92.02 (3)	C6—C1—S3	119.0 (2)
C1—S1—Ni1	104.21 (11)	S1—C1—S3	121.2 (2)
C6—S2—Ni1	104.67 (11)	C1—C6—S2	119.2 (2)
C1—S3—C2	103.77 (15)	C1—C6—S4	119.8 (2)
C6—S4—C7	102.7 (2)	S2—C6—S4	121.0 (2)
C6—C1—S1	119.9 (2)		

All H atoms were located in electron density maps and their coordinates refined. They were given a common refined atomic displacement parameter.

Data collection: *MSCI/AF C Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCI/AF C Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1028). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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