Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

catena-Poly[[bis(*O*-isopropyl dithiocarbonato-κ^2S,S')nickel(II)]-μ-1,2-di-4-pyridylethane-$\kappa^2N:N'$]

Denise D. Hill and Edward R. T. Tiekink*

Department of Chemistry, University of Texas at San Antonio, One UTSA Circle, San Antonio, Texas 78249-0698, USA
Correspondence e-mail: edward.tiekink@utsa.edu

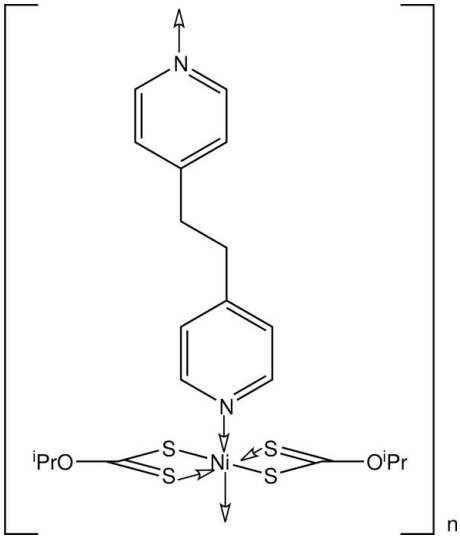
Received 18 October 2007; accepted 18 October 2007

Key indicators: single-crystal X-ray study; T = 98 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.088; data-to-parameter ratio = 17.8.

The Ni atom in the zigzag polymeric title complex, $[Ni(C_4H_7OS_2)_2(C_{12}H_{12}N_2)]_n$, is situated on a twofold axis and the bridging ligand situated about a centre of inversion. The structure features a distorted octahedral *cis*-NiN_2S_4 coordination geometry defined by two chelating *O*-isopropyl dithiocarbonate ligands and two N atoms derived from the bidentate bridging ligands.

Related literature

For the synthesis, see Lai *et al.* (2004); Cox & Tiekink (1999). For related structures, see Tiekink & Haiduc (2005); Dakternieks *et al.* (2006).



Experimental

Crystal data

$[Ni(C_4H_7OS_2)_2(C_{12}H_{12}N_2)]$
M_r = 513.38
Monoclinic, $C2/c$
a = 10.1460 (6) Å
b = 15.8638 (10) Å
c = 15.0545 (9) Å
β = 104.256 (3)°
V = 2348.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.20 mm^{-1}
T = 98 (2) K
0.30 × 0.11 × 0.05 mm

Data collection

Rigaku AFC12κ/SATURN724 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.832, T_{max} = 1
(expected range = 0.784–0.942)
3974 measured reflections
2348 independent reflections
2243 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.088
S = 1.19
2348 reflections
132 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—N1	2.089 (2)	Ni—S2	2.4509 (7)
Ni—S1	2.4110 (7)		
S1—Ni—S2	73.38 (2)		

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear* ; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

This work was supported by departmental research grant No. AX-0026 from the Robert A. Welch Foundation.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2592).

References

Brandenburg, K. (2006). *DIAMOND*. Release 3.1. Crystal Impact GbR, Bonn, Germany.
Cox, M. J. & Tiekink, E. R. T. (1999). *Z. Kristallogr.* **214**, 242–250.
Dakternieks, D., Duthie, A., Lai, C.-S. & Tiekink, E. R. T. (2006). *Acta Cryst.* E**62**, m3006–m3008.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Lai, C. S., Liu, S. & Tiekink, E. R. T. (2004). *CrystEngComm*, **6**, 221–226.
Rigaku (2005). *CrystalClear*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tiekink, E. R. T. & Haiduc, I. (2005). *Prog. Inorg. Chem.* **54**, 127–319.

  doi:10.1107/S1600536807051616

supplementary materials

Acta Cryst. (2007). E**63**, m2808 [**doi:10.1107/S1600536807051616**]

catena-Poly[[bis(*O*-isopropyl dithiocarbonato-$\kappa^2 S,S'$)nickel(II)]-μ-1,2-di-4-pyridylethane-$\kappa^2 N:N'$]

D. D. Hill and E. R. T. Tiekink

Comment

Nickel(II) xanthates, $Ni(S_2COR)_2$, are well known to form adducts with nitrogen-donors to generate aggregates of various topologies (Tiekink & Haiduc, 2005). In continuation of on-going studies in this area (Dakternieks *et al.*, 2006), the title compound, (I), was prepared and characterized. The octahedrally coordinated Ni(II) centre in (I), Fig. 1, exists within a *cis*-N_2S_4 donor set (Table 1); the Ni atom lies on a crystallographic 2-fold axis and the bridging ligand is disposed about a centre of inversion. The xanthate ligand ligand coordinates in a symmetric mode with the Ni—S1 and Ni—S2 bond distances being 2.4110 (7) & 2.4509 (7) Å. The structure is polymeric and adopts a zigzag topology as is often observed in such adducts (Tiekink & Haiduc, 2005), as shown in Fig. 2.

Experimental

The title compound was prepared by warming the parent nickel xanthate (Cox & Tiekink, 1999) with 1,2-bis(4-pyridyl)ethane in $CHCl_3$ (20 ml) following a literature procedure (Lai *et al.*, 2004). Green crystals of (I) were isolated by the slow evaporation of a of a 3:1 $CHCl_3$/acetonitrile solution of the compound; m.p. 423–427 K.

Refinement

The H atoms were included in the riding-model approximation with C—H distances = 0.95 to 1.00 Å, and with U_{iso}(methyl-H) = 1.5U_{eq}(methyl-C) and U_{iso}(H) = 1.2U_{eq}(remaining-C).

Figures

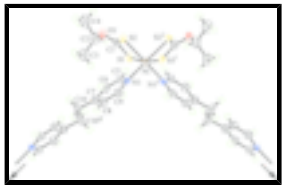


Fig. 1. Expanded view of the coordination geometry in (I) showing displacement ellipsoids for the non-hydrogen atoms at the 50% probability level. Symmetry codes: (i) $-x, y, 1/2-z$; (ii) $1-x, 1-y, 1-z$.

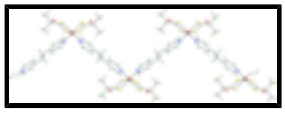


Fig. 2. Polymer topology in (I). Colour code: Ni (brown), S (yellow), O (red), N (blue), C (grey) & H (green).

catena-Poly[[bis(*O*-isopropyl dithiocarbonato-$\kappa^2 S,S'$)nickel(II)]-μ-1,2-di-4-pyridylethane-$\kappa^2 N:N'$]

Crystal data

$[Ni(C_4H_7OS_2)_2(C_{12}H_{12}N_2)]$ $F_{000} = 1072$

M_r = 513.38	D_x = 1.452 Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: -C 2yc	Cell parameters from 1905 reflections
a = 10.1460 (6) Å	θ = 2.9–30.0º
b = 15.8638 (10) Å	μ = 1.20 mm^{-1}
c = 15.0545 (9) Å	T = 98 (2) K
β = 104.256 (3)º	Fragment, green
V = 2348.5 (2) Å^3	0.30 × 0.11 × 0.05 mm
Z = 4	

Data collection

Rigaku AFC12κ/SATURN724 diffractometer	2348 independent reflections
Radiation source: fine-focus sealed tube	2243 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.019
T = 98(2) K	θ_{max} = 26.5º
ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	h = 0→12
T_{min} = 0.832, T_{max} = 1	k = −19→19
3974 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
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H-atom parameters constrained
$wR(F^2)$ = 0.088	$w = 1/[\sigma^2(F_o^2) + (0.0265P)^2 + 7.5757P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.19	$(\Delta/\sigma)_{max} < 0.001$
2348 reflections	$\Delta\rho_{max}$ = 0.57 e Å^{-3}
132 parameters	$\Delta\rho_{min}$ = −0.28 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}
Ni	0.0000	0.25754 (3)	0.2500	0.01712 (13)
S1	0.11332 (7)	0.24132 (4)	0.12782 (4)	0.02205 (16)
S2	0.17407 (7)	0.14873 (4)	0.29958 (4)	0.02095 (16)
O2	0.31745 (19)	0.13301 (12)	0.18192 (13)	0.0241 (4)
N1	0.1411 (2)	0.34583 (13)	0.31920 (14)	0.0193 (4)
C1	0.2118 (3)	0.17161 (17)	0.19961 (17)	0.0202 (5)
C2	0.3651 (3)	0.1563 (2)	0.10060 (19)	0.0287 (6)
H2	0.2861	0.1678	0.0474	0.034*
C3	0.4532 (3)	0.2335 (2)	0.1236 (2)	0.0348 (7)
H3A	0.3977	0.2813	0.1341	0.052*
H3B	0.4940	0.2466	0.0726	0.052*
H3C	0.5253	0.2229	0.1791	0.052*
C4	0.4439 (4)	0.0799 (2)	0.0814 (2)	0.0434 (8)
H4A	0.3822	0.0317	0.0657	0.065*
H4B	0.5160	0.0665	0.1360	0.065*
H4C	0.4845	0.0923	0.0301	0.065*
C5	0.1958 (3)	0.33574 (17)	0.40860 (17)	0.0204 (5)
H5	0.1530	0.2978	0.4415	0.024*
C6	0.3101 (3)	0.37706 (18)	0.45547 (18)	0.0234 (6)
H6	0.3445	0.3676	0.5193	0.028*
C7	0.3758 (3)	0.43277 (17)	0.40978 (19)	0.0241 (6)
C8	0.3171 (3)	0.44529 (17)	0.31804 (19)	0.0254 (6)
H8	0.3566	0.4839	0.2838	0.031*
C9	0.2010 (3)	0.40162 (17)	0.27593 (18)	0.0231 (6)
H9	0.1617	0.4119	0.2128	0.028*
C10	0.5056 (3)	0.4752 (2)	0.4583 (2)	0.0326 (7)
H10A	0.5343	0.5138	0.4148	0.039*
H10B	0.5772	0.4319	0.4771	0.039*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni	0.0166 (2)	0.0197 (3)	0.0142 (2)	0.000	0.00224 (17)	0.000
S1	0.0205 (3)	0.0290 (4)	0.0163 (3)	0.0013 (3)	0.0039 (2)	0.0028 (2)
S2	0.0229 (3)	0.0230 (3)	0.0178 (3)	0.0029 (3)	0.0066 (2)	0.0023 (2)
O2	0.0211 (10)	0.0291 (10)	0.0243 (10)	0.0007 (8)	0.0098 (8)	0.0000 (8)
N1	0.0199 (11)	0.0168 (11)	0.0196 (11)	0.0009 (9)	0.0019 (9)	−0.0001 (8)
C1	0.0179 (12)	0.0236 (13)	0.0188 (12)	−0.0007 (10)	0.0040 (10)	−0.0018 (10)
C2	0.0261 (14)	0.0390 (18)	0.0243 (14)	−0.0011 (13)	0.0126 (12)	−0.0005 (12)
C3	0.0298 (16)	0.0459 (19)	0.0285 (15)	−0.0035 (14)	0.0068 (13)	0.0048 (14)
C4	0.0361 (19)	0.055 (2)	0.046 (2)	−0.0044 (16)	0.0232 (16)	−0.0098 (17)
C5	0.0224 (13)	0.0205 (13)	0.0178 (12)	−0.0023 (11)	0.0040 (10)	0.0003 (10)

C6	0.0229 (14)	0.0271 (14)	0.0182 (13)	−0.0018 (11)	0.0016 (11)	−0.0009 (10)
C7	0.0233 (14)	0.0231 (14)	0.0263 (14)	−0.0037 (11)	0.0068 (11)	−0.0060 (11)
C8	0.0328 (15)	0.0208 (14)	0.0244 (14)	−0.0054 (12)	0.0103 (12)	0.0016 (11)
C9	0.0254 (14)	0.0219 (13)	0.0201 (13)	0.0000 (11)	0.0019 (11)	0.0024 (10)
C10	0.0261 (15)	0.0399 (18)	0.0326 (16)	−0.0139 (13)	0.0085 (12)	−0.0114 (14)

Geometric parameters (Å, °)

Ni—N1	2.089 (2)	C3—H3C	0.9800
Ni—N1^{i}	2.089 (2)	C4—H4A	0.9800
Ni—S1	2.4110 (7)	C4—H4B	0.9800
Ni—S1^{i}	2.4110 (7)	C4—H4C	0.9800
Ni—S2^{i}	2.4509 (7)	C5—C6	1.367 (4)
Ni—S2	2.4509 (7)	C5—H5	0.9500
S1—C1	1.690 (3)	C6—C7	1.387 (4)
S2—C1	1.681 (3)	C6—H6	0.9500
O2—C1	1.318 (3)	C7—C8	1.377 (4)
O2—C2	1.470 (3)	C7—C10	1.499 (4)
N1—C9	1.331 (3)	C8—C9	1.379 (4)
N1—C5	1.333 (3)	C8—H8	0.9500
C2—C3	1.505 (4)	C9—H9	0.9500
C2—C4	1.518 (4)	C10—C10^{ii}	1.510 (6)
C2—H2	1.0000	C10—H10A	0.9900
C3—H3A	0.9800	C10—H10B	0.9900
C3—H3B	0.9800		
N1—Ni—N1^{i}	95.78 (12)	H3A—C3—H3B	109.5
N1—Ni—S1	93.15 (6)	C2—C3—H3C	109.5
N1^{i}—Ni—S1	95.06 (6)	H3A—C3—H3C	109.5
N1—Ni—S1^{i}	95.06 (6)	H3B—C3—H3C	109.5
N1^{i}—Ni—S1^{i}	93.15 (6)	C2—C4—H4A	109.5
S1—Ni—S1^{i}	167.74 (4)	C2—C4—H4B	109.5
N1—Ni—S2^{i}	168.04 (6)	H4A—C4—H4B	109.5
N1^{i}—Ni—S2^{i}	88.07 (6)	C2—C4—H4C	109.5
S1—Ni—S2^{i}	97.80 (2)	H4A—C4—H4C	109.5
S1^{i}—Ni—S2^{i}	73.38 (2)	H4B—C4—H4C	109.5
N1—Ni—S2	88.07 (6)	N1—C5—C6	123.6 (2)
N1^{i}—Ni—S2	168.04 (6)	N1—C5—H5	118.2
S1—Ni—S2	73.38 (2)	C6—C5—H5	118.2
S1^{i}—Ni—S2	97.80 (2)	C5—C6—C7	120.0 (2)
S2^{i}—Ni—S2	90.45 (4)	C5—C6—H6	120.0
C1—S1—Ni	84.25 (9)	C7—C6—H6	120.0
C1—S2—Ni	83.17 (9)	C8—C7—C6	116.6 (2)
C1—O2—C2	120.1 (2)	C8—C7—C10	122.3 (3)
C9—N1—C5	116.6 (2)	C6—C7—C10	121.1 (3)
C9—N1—Ni	122.77 (18)	C7—C8—C9	119.9 (3)
C5—N1—Ni	119.13 (18)	C7—C8—H8	120.1

O2—C1—S2	117.2 (2)	C9—C8—H8	120.1
O2—C1—S1	123.9 (2)	N1—C9—C8	123.4 (2)
S2—C1—S1	118.98 (16)	N1—C9—H9	118.3
O2—C2—C3	108.2 (2)	C8—C9—H9	118.3
O2—C2—C4	104.7 (2)	C7—C10—C10^{ii}	113.7 (3)
C3—C2—C4	112.5 (3)	C7—C10—H10A	108.8
O2—C2—H2	110.4	C10^{ii}—C10—H10A	108.8
C3—C2—H2	110.4	C7—C10—H10B	108.8
C4—C2—H2	110.4	C10^{ii}—C10—H10B	108.8
C2—C3—H3A	109.5	H10A—C10—H10B	107.7
C2—C3—H3B	109.5		
N1—Ni—S1—C1	84.26 (11)	C2—O2—C1—S2	−173.59 (19)
N1^{i}—Ni—S1—C1	−179.65 (11)	C2—O2—C1—S1	6.8 (3)
S1^{i}—Ni—S1—C1	−47.77 (9)	Ni—S2—C1—O2	176.0 (2)
S2^{i}—Ni—S1—C1	−90.91 (9)	Ni—S2—C1—S1	−4.38 (15)
S2—Ni—S1—C1	−2.78 (9)	Ni—S1—C1—O2	−176.0 (2)
N1—Ni—S2—C1	−91.07 (11)	Ni—S1—C1—S2	4.44 (15)
N1^{i}—Ni—S2—C1	18.0 (3)	C1—O2—C2—C3	82.0 (3)
S1—Ni—S2—C1	2.80 (9)	C1—O2—C2—C4	−157.9 (2)
S1^{i}—Ni—S2—C1	174.09 (9)	C9—N1—C5—C6	−2.3 (4)
S2^{i}—Ni—S2—C1	100.81 (9)	Ni—N1—C5—C6	164.1 (2)
N1^{i}—Ni—N1—C9	−61.03 (19)	N1—C5—C6—C7	−0.2 (4)
S1—Ni—N1—C9	34.4 (2)	C5—C6—C7—C8	2.2 (4)
S1^{i}—Ni—N1—C9	−154.7 (2)	C5—C6—C7—C10	−176.5 (3)
S2^{i}—Ni—N1—C9	−169.4 (2)	C6—C7—C8—C9	−1.7 (4)
S2—Ni—N1—C9	107.6 (2)	C10—C7—C8—C9	177.0 (3)
N1^{i}—Ni—N1—C5	133.5 (2)	C5—N1—C9—C8	2.8 (4)
S1—Ni—N1—C5	−131.04 (19)	Ni—N1—C9—C8	−163.0 (2)
S1^{i}—Ni—N1—C5	39.9 (2)	C7—C8—C9—N1	−0.9 (4)
S2^{i}—Ni—N1—C5	25.2 (4)	C8—C7—C10—C10^{ii}	124.9 (4)
S2—Ni—N1—C5	−57.81 (19)	C6—C7—C10—C10^{ii}	−56.5 (5)

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

Fig. 1

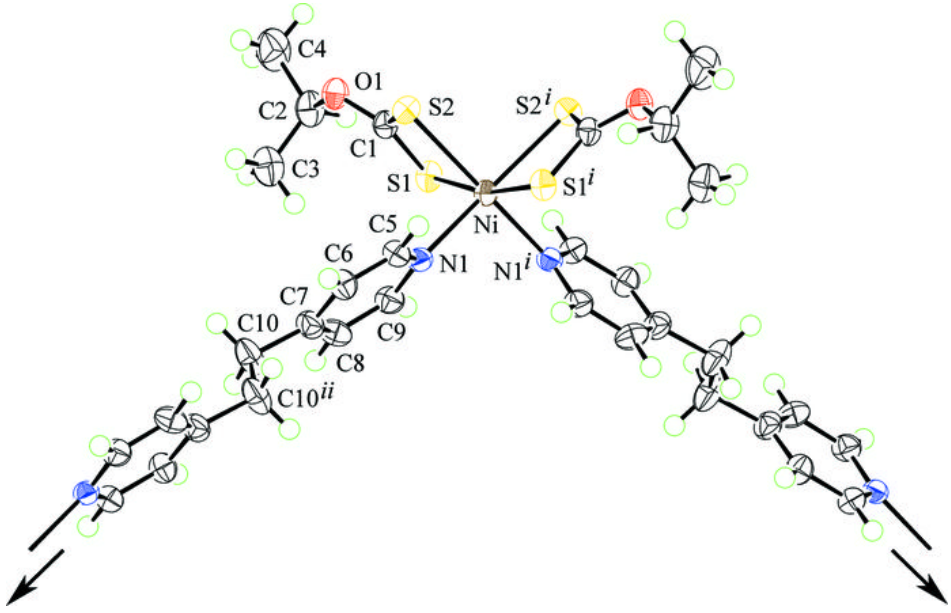


Fig. 2