Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Bis(*O*-ethyl dithiocarbonato-$\kappa^2S,S'$)bis(pyridine-3-carbonitrile-$\kappa N^1$)nickel(II)

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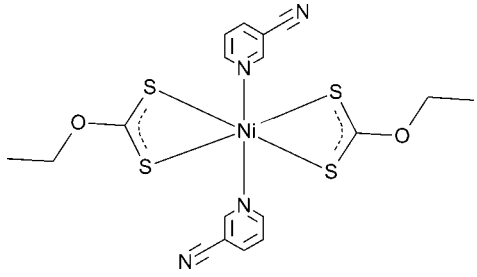
Received 28 November 2011; accepted 12 December 2011

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.025; $wR$ factor = 0.060; data-to-parameter ratio = 14.4.

The $Ni^{2+}$ ion in the title complex, $[Ni(C_3H_5OS_2)_2(C_6H_4N_2)_2]$, is in a strongly distorted octahedral coordination environment formed by an $N_2S_4$ donor set, with the $Ni^{2+}$ ion located on a centre of inversion. In the crystal, weak C—H···S and C—H···N interactions are observed.

## Related literature

For related structures, see: Tiekink & Haiduc (2005); Dakternieks *et al.* (2006); Hill & Tiekink (2007); Hogarth *et al.* (2009)




## Experimental

### Crystal data

$[Ni(C_3H_5OS_2)_2(C_6H_4N_2)_2]$
$M_r$ = 509.31
Monoclinic, $P2_1/c$
$a$ = 6.7302 (2) Å
$b$ = 18.8959 (5) Å
$c$ = 8.7242 (2) Å
$\beta$ = 95.916 (2)°
$V$ = 1103.58 (5) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 1.28 mm$^{-1}$
$T$ = 293 K
0.3 × 0.3 × 0.1 mm

### Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007)
$T_{min}$ = 0.728, $T_{max}$ = 1.000
18847 measured reflections
1930 independent reflections
1723 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.060
$S$ = 1.07
1930 reflections
134 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Ni1—N1 | 2.1273 (17) | Ni1—S2 | 2.4450 (6) |
| Ni1—S1 | 2.4335 (5) | | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C9—H9$A$···S2$^{i}$ | 0.97 | 2.85 | 3.455 (2) | 121 |
| C10—H10$C$···N2$^{ii}$ | 0.96 | 2.65 | 3.595 (4) | 169 |

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z+2$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009) and *PARST* (Nardelli, 1995).

RK acknowledges the Department of Science & Technology for the single-crystal X-ray diffractometer sanctioned as a National Facility under project No. SR/S2/CMP-47/2003. He is also thankful to the UGC for research funding under research project F.No. 37?4154/2009 (J&K) (SR).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2438).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, m58 [ **doi:10.1107/S1600536811053475** ]

# Bis(*O*-ethyl dithiocarbonato-$\kappa^2 S,S'$)bis(pyridine-3-carbonitrile-$\kappa N^1$)nickel(II)

**S. Kapoor, R. Kour, R. Sachar, R. Kant, V. K. Gupta and K. Kapoor**

## Comment

Xanthates (*O*-alkyl/aryl dithiocarbonates) have been known for a long time and many adducts of metal xanthates with different ligands have been prepared and studied in the last several decades. Adducts of transition metal xanthates with N-donor ligands are well represented in the literature, the most extensively studied being those of nickel(II). Nitrogen containing adducts of nickel(II) xanthates are known to adopt a variety of supramolecular assemblies (Tiekink & Haiduc, 2005). The Ni atom in (I) is located on a center of inversion and exists within a *trans*-N2S4 donor set that defines an approximately octahedral coordination geometry. The chelating xanthate ligand forms essentially equivalent Ni—S bond distances; this equivalence is reflected in the parameters defining the xanthate ligand. The bond angles around the Ni atom are in the range of 73.83 (2) to 180.00 (3)°. The Ni—S bond lengths, Ni1—S1 = 2.4335 (5); Ni1—S2 = 2.4450 (6) Å, are in good agreement with those reported for other Ni-dithiocarbonato complexes (Tiekink & Haiduc, 2005; Dakternieks *et al.*, 2006; Hill & Tiekink, 2007; Hogarth *et al.*, 2009). Molecules in the unit cell are packed together to form well defined layers. While no classical hydrogen bonds are present, the C—H···S and C—H···N hydrogen bonds (Table 1) play important role in stabilizing the crystal structure.

## Experimental

The title complex was prepared by stirring the parent nickel(II) ethylxanthate (0.781g,0.0026 mol.) with 3-cyanopyridine(0.541g, 0.0052 mol.) in acetone(60 ml) for one hour. Green crystals of (I) were isolated by the slow evaporation of the resulting solution of the complex.

## Refinement

All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.97 Å and with $U_{iso}$(H) = 1.2Ueq(C) or 1.5Ueq(methylC).

## Figures

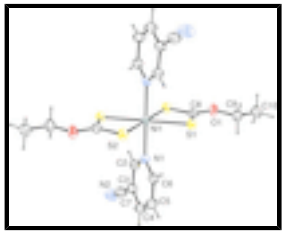

Fig. 1. *ORTEP* view of the molecule with displacement ellipsoids drawn at the 30% probability level. Unlabeled atoms are generated by the symmetry operation -*x*, 1-*y*, 1-*z*.

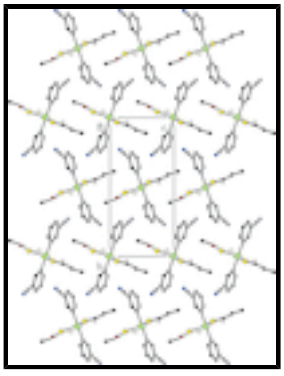

Fig. 2. The packing arrangement of molecules viewed down the *a* axis.

**Bis(*O*-ethyl dithiocarbonato-κ$^2$*S*,*S*')bis(pyridine-3-carbonitrile- κ*N*$^1$)nickel(II)**

*Crystal data*

| | |
|---|---|
| $[Ni(C_3H_5OS_2)_2(C_6H_4N_2)_2]$ | $F(000) = 524$ |
| $M_r = 509.31$ | $D_x = 1.533$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 10361 reflections |
| $a = 6.7302$ (2) Å | $\theta = 3.6–29.0°$ |
| $b = 18.8959$ (5) Å | $\mu = 1.28$ mm$^{-1}$ |
| $c = 8.7242$ (2) Å | $T = 293$ K |
| $\beta = 95.916$ (2)° | Hexagonal plate, green |
| $V = 1103.58$ (5) Å$^3$ | 0.3 × 0.3 × 0.1 mm |
| $Z = 2$ | |

*Data collection*

| | |
|---|---|
| Oxford Diffraction Xcalibur Sapphire3 diffractometer | 1930 independent reflections |
| Radiation source: fine-focus sealed tube | 1723 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.036$ |
| Detector resolution: 16.1049 pixels mm$^{-1}$ | $\theta_{max} = 25.0°$, $\theta_{min} = 3.7°$ |
| ω scans | $h = -8\rightarrow8$ |
| Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007) | $k = -22\rightarrow22$ |
| $T_{min} = 0.728$, $T_{max} = 1.000$ | $l = -10\rightarrow10$ |
| 18847 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.025$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.060$ | H-atom parameters constrained |

| | |
|---|---|
| $S$ = 1.07 | $w = 1/[\sigma^2(F_o^2) + (0.0208P)^2 + 0.7659P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 1930 reflections | $(\Delta/\sigma)_{max} = 0.001$ |
| 134 parameters | $\Delta\rho_{max} = 0.23$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.22$ e Å$^{-3}$ |

*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 | 0.0000 | 0.5000 | 0.5000 | 0.02943 (12) |
| S1 | 0.33453 (8) | 0.46692 (3) | 0.60894 (6) | 0.03356 (14) |
| S2 | 0.03241 (8) | 0.54100 (3) | 0.23794 (7) | 0.03737 (15) |
| O1 | 0.3099 (2) | 0.42097 (8) | 0.89474 (17) | 0.0404 (4) |
| N1 | 0.0499 (3) | 0.60555 (9) | 0.5796 (2) | 0.0340 (4) |
| N2 | −0.3300 (4) | 0.76898 (12) | 0.8455 (3) | 0.0691 (7) |
| C2 | −0.0808 (3) | 0.63914 (11) | 0.6579 (3) | 0.0368 (5) |
| H2 | −0.1910 | 0.6143 | 0.6856 | 0.044* |
| C3 | −0.0591 (3) | 0.71000 (12) | 0.6999 (3) | 0.0401 (5) |
| C4 | 0.1050 (4) | 0.74716 (13) | 0.6592 (3) | 0.0484 (6) |
| H4 | 0.1222 | 0.7947 | 0.6846 | 0.058* |
| C5 | 0.2413 (4) | 0.71212 (13) | 0.5805 (3) | 0.0469 (6) |
| H5 | 0.3542 | 0.7354 | 0.5532 | 0.056* |
| C6 | 0.2086 (3) | 0.64182 (12) | 0.5425 (3) | 0.0383 (5) |
| H6 | 0.3014 | 0.6186 | 0.4885 | 0.046* |
| C7 | −0.2097 (4) | 0.74323 (13) | 0.7818 (3) | 0.0497 (6) |
| C8 | 0.2162 (3) | 0.44638 (11) | 0.7649 (2) | 0.0319 (5) |
| C9 | 0.5252 (3) | 0.41173 (13) | 0.9032 (3) | 0.0446 (6) |
| H9A | 0.5889 | 0.4561 | 0.8807 | 0.054* |
| H9B | 0.5583 | 0.3767 | 0.8285 | 0.054* |
| C10 | 0.5962 (4) | 0.38776 (14) | 1.0625 (3) | 0.0520 (6) |
| H10A | 0.5554 | 0.4213 | 1.1357 | 0.078* |
| H10B | 0.7392 | 0.3842 | 1.0732 | 0.078* |
| H10C | 0.5395 | 0.3423 | 1.0810 | 0.078* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ni1 | 0.0264 (2) | 0.0265 (2) | 0.0358 (2) | 0.00092 (15) | 0.00515 (15) | 0.00119 (16) |
| S1 | 0.0274 (3) | 0.0369 (3) | 0.0373 (3) | 0.0017 (2) | 0.0078 (2) | 0.0051 (2) |
| S2 | 0.0275 (3) | 0.0441 (3) | 0.0418 (3) | 0.0007 (2) | 0.0097 (2) | 0.0045 (2) |
| O1 | 0.0312 (8) | 0.0509 (10) | 0.0392 (9) | 0.0036 (7) | 0.0040 (7) | 0.0093 (7) |
| N1 | 0.0334 (10) | 0.0296 (9) | 0.0387 (10) | 0.0001 (8) | 0.0028 (8) | 0.0021 (8) |
| N2 | 0.0700 (17) | 0.0489 (14) | 0.0917 (19) | 0.0032 (12) | 0.0249 (14) | −0.0198 (13) |
| C2 | 0.0360 (12) | 0.0332 (12) | 0.0412 (13) | −0.0003 (9) | 0.0035 (10) | −0.0004 (10) |
| C3 | 0.0443 (13) | 0.0338 (12) | 0.0412 (13) | 0.0033 (10) | 0.0001 (10) | −0.0040 (10) |
| C4 | 0.0590 (16) | 0.0314 (12) | 0.0535 (15) | −0.0084 (11) | −0.0004 (12) | −0.0019 (11) |
| C5 | 0.0466 (14) | 0.0411 (13) | 0.0531 (15) | −0.0124 (11) | 0.0061 (11) | 0.0036 (12) |
| C6 | 0.0351 (12) | 0.0371 (12) | 0.0424 (13) | −0.0012 (9) | 0.0033 (10) | 0.0038 (10) |
| C7 | 0.0561 (16) | 0.0361 (13) | 0.0565 (16) | −0.0024 (12) | 0.0042 (13) | −0.0096 (12) |
| C8 | 0.0306 (11) | 0.0294 (11) | 0.0357 (11) | −0.0003 (9) | 0.0028 (9) | 0.0021 (9) |
| C9 | 0.0341 (12) | 0.0503 (14) | 0.0488 (14) | 0.0072 (10) | 0.0013 (10) | 0.0061 (11) |
| C10 | 0.0525 (15) | 0.0469 (15) | 0.0536 (15) | 0.0075 (12) | −0.0082 (12) | 0.0037 (12) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Ni1—N1 | 2.1273 (17) | C3—C7 | 1.442 (3) |
| Ni1—S1 | 2.4335 (5) | C4—C5 | 1.372 (3) |
| Ni1—S2 | 2.4450 (6) | C4—H4 | 0.9300 |
| S1—C8 | 1.691 (2) | C5—C6 | 1.381 (3) |
| S2—C8$^{i}$ | 1.688 (2) | C5—H5 | 0.9300 |
| O1—C8 | 1.328 (2) | C6—H6 | 0.9300 |
| O1—C9 | 1.454 (3) | C9—C10 | 1.493 (3) |
| N1—C2 | 1.329 (3) | C9—H9A | 0.9700 |
| N1—C6 | 1.336 (3) | C9—H9B | 0.9700 |
| N2—C7 | 1.138 (3) | C10—H10A | 0.9600 |
| C2—C3 | 1.392 (3) | C10—H10B | 0.9600 |
| C2—H2 | 0.9300 | C10—H10C | 0.9600 |
| C3—C4 | 1.385 (3) | | |
| N1$^{i}$—Ni1—N1 | 180.00 (3) | C2—C3—C7 | 119.3 (2) |
| N1$^{i}$—Ni1—S1 | 89.73 (5) | C5—C4—C3 | 118.4 (2) |
| N1—Ni1—S1 | 90.27 (5) | C5—C4—H4 | 120.8 |
| N1$^{i}$—Ni1—S1$^{i}$ | 90.27 (5) | C3—C4—H4 | 120.8 |
| N1—Ni1—S1$^{i}$ | 89.73 (5) | C4—C5—C6 | 119.1 (2) |
| S1—Ni1—S1$^{i}$ | 180.0 | C4—C5—H5 | 120.5 |
| N1$^{i}$—Ni1—S2$^{i}$ | 88.96 (5) | C6—C5—H5 | 120.5 |
| N1—Ni1—S2$^{i}$ | 91.04 (5) | N1—C6—C5 | 123.1 (2) |
| S1—Ni1—S2$^{i}$ | 73.831 (18) | N1—C6—H6 | 118.4 |
| S1$^{i}$—Ni1—S2$^{i}$ | 106.169 (18) | C5—C6—H6 | 118.4 |
| N1$^{i}$—Ni1—S2 | 91.04 (5) | N2—C7—C3 | 179.3 (3) |

| | | | |
|---|---|---|---|
| N1—Ni1—S2 | 88.96 (5) | O1—C8—S2$^{i}$ | 116.53 (15) |
| S1—Ni1—S2 | 106.169 (18) | O1—C8—S1 | 123.21 (15) |
| S1$^{i}$—Ni1—S2 | 73.831 (18) | S2$^{i}$—C8—S1 | 120.26 (12) |
| S2$^{i}$—Ni1—S2 | 180.0 | O1—C9—C10 | 107.76 (19) |
| C8—S1—Ni1 | 83.10 (7) | O1—C9—H9A | 110.2 |
| C8$^{i}$—S2—Ni1 | 82.80 (7) | C10—C9—H9A | 110.2 |
| C8—O1—C9 | 118.02 (17) | O1—C9—H9B | 110.2 |
| C2—N1—C6 | 117.86 (19) | C10—C9—H9B | 110.2 |
| C2—N1—Ni1 | 121.65 (14) | H9A—C9—H9B | 108.5 |
| C6—N1—Ni1 | 120.33 (15) | C9—C10—H10A | 109.5 |
| N1—C2—C3 | 122.5 (2) | C9—C10—H10B | 109.5 |
| N1—C2—H2 | 118.7 | H10A—C10—H10B | 109.5 |
| C3—C2—H2 | 118.7 | C9—C10—H10C | 109.5 |
| C4—C3—C2 | 119.0 (2) | H10A—C10—H10C | 109.5 |
| C4—C3—C7 | 121.7 (2) | H10B—C10—H10C | 109.5 |
| N1$^{i}$—Ni1—S1—C8 | −88.58 (9) | C6—N1—C2—C3 | 0.8 (3) |
| N1—Ni1—S1—C8 | 91.42 (9) | Ni1—N1—C2—C3 | −174.57 (16) |
| S2$^{i}$—Ni1—S1—C8 | 0.42 (7) | N1—C2—C3—C4 | −0.1 (3) |
| S2—Ni1—S1—C8 | −179.58 (7) | N1—C2—C3—C7 | 178.6 (2) |
| N1$^{i}$—Ni1—S2—C8$^{i}$ | 90.39 (9) | C2—C3—C4—C5 | −0.9 (4) |
| N1—Ni1—S2—C8$^{i}$ | −89.61 (9) | C7—C3—C4—C5 | −179.6 (2) |
| S1—Ni1—S2—C8$^{i}$ | −179.58 (7) | C3—C4—C5—C6 | 1.2 (4) |
| S1$^{i}$—Ni1—S2—C8$^{i}$ | 0.42 (7) | C2—N1—C6—C5 | −0.5 (3) |
| S1—Ni1—N1—C2 | −130.63 (16) | Ni1—N1—C6—C5 | 174.93 (18) |
| S1$^{i}$—Ni1—N1—C2 | 49.37 (16) | C4—C5—C6—N1 | −0.5 (4) |
| S2$^{i}$—Ni1—N1—C2 | −56.80 (16) | C9—O1—C8—S2$^{i}$ | −177.93 (16) |
| S2—Ni1—N1—C2 | 123.20 (16) | C9—O1—C8—S1 | 1.2 (3) |
| S1—Ni1—N1—C6 | 54.10 (16) | Ni1—S1—C8—O1 | −179.72 (18) |
| S1$^{i}$—Ni1—N1—C6 | −125.90 (16) | Ni1—S1—C8—S2$^{i}$ | −0.67 (12) |
| S2$^{i}$—Ni1—N1—C6 | 127.93 (16) | C8—O1—C9—C10 | 176.01 (19) |
| S2—Ni1—N1—C6 | −52.07 (16) | | |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C9—H9A···S2$^{ii}$ | 0.97 | 2.85 | 3.455 (2) | 121 |
| C10—H10C···N2$^{iii}$ | 0.96 | 2.65 | 3.595 (4) | 169 |

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

**Fig. 1**

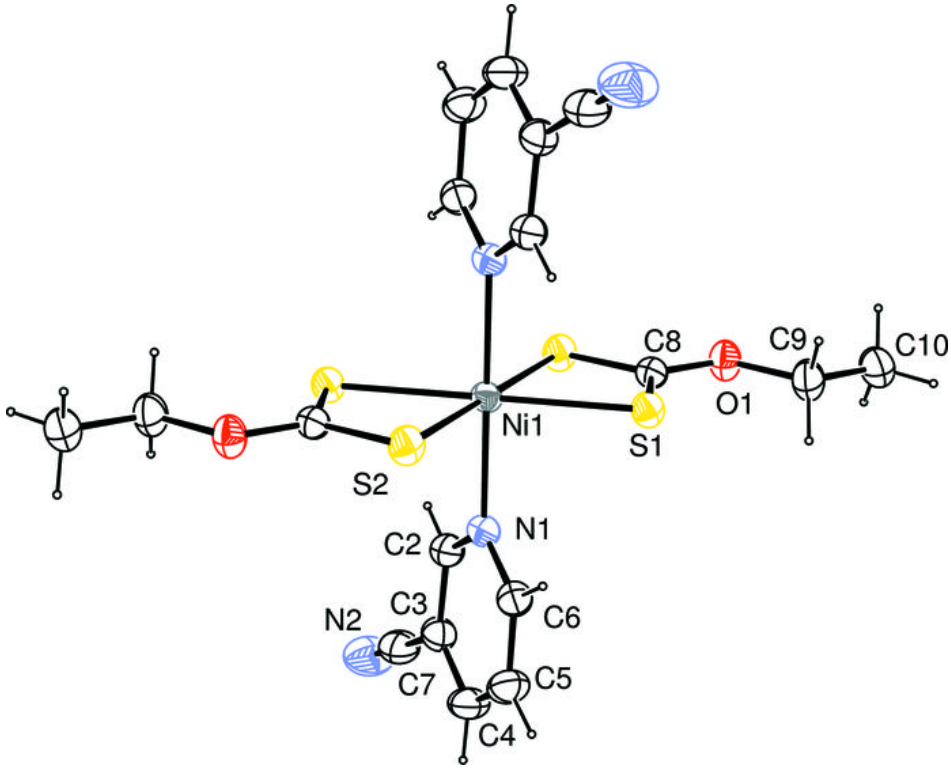

**Fig. 2**