

## Research Article

# Synthesis, Characterization, and X-Ray Crystal Structure of Bis(O-amyl dithiocarbonato- $\kappa^2$ S,S')bis(4-cyanopyridine- $\kappa$ N)nickel(II)

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Received 24 August 2013; Accepted 23 December 2013; Published 17 February 2014

Academic Editor: Xian-He Bu

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The  $[\text{Ni}(\text{S}_2\text{CO}-n\text{-C}_5\text{H}_{11})_2(\text{C}_6\text{H}_4\text{N}_2)_2]$  adduct of 4-cyanopyridine with  $[\text{Ni}(\text{S}_2\text{CO}-n\text{-C}_5\text{H}_{11})_2]$  was synthesized and characterized by elemental analysis, magnetic susceptibility measurement, IR, electronic spectral data, and X-ray diffraction analysis. The Ni atom in the title complex is octahedrally coordinated within a trans- $\text{N}_2\text{S}_4$  donor set, with the Ni atom located on a centre of inversion. The title compound exhibits magnetic moment value (3.20 B.M) which is in agreement with magnetic moment values observed for paramagnetic octahedral complexes of nickel(II). The title complex crystallizes in the orthorhombic space group Pbca with unit cell parameters  $a = 11.455(5)$ ,  $b = 9.602(4)$ , and  $c = 26.374(1)$  Å. Crystal structure was solved by direct methods and refined by full matrix least-squares procedures to a final  $R$  value of 0.0499 for 2004 observed reflections. The amyl chain is disordered over two sets of sites, with occupancy ratios of 0.595 : 0.405. Infinite long chains of molecules are formed with the help of C-H···N hydrogen bond.

## 1. Introduction

The coordination compounds in which the dithio ligands form a ring with the metal ion have been reported for a great number of metal ions and display interesting and often quite novel properties and structures. O-alkyldithiocarbonates (Xanthates) are among the more frequently considered sulphur containing ligands that have been studied from past many years and many adducts of metal Xanthates with different ligands which have been prepared and studied in the last several decades [1, 2]. Xanthates are formed by the reaction between a metal alkoxide and carbon disulphide. A variety of alcohols have been Xanthated in this manner. Xanthates have ability to bind to various metals [3] and form chelates with virtually all transition elements. They have been proved to be versatile chelating agents for the separation and extraction of metals in analytical chemistry and mineral floating [4, 5]. Xanthates are used in curing and vulcanization of rubber, as collectors of sulphides ores

or minerals in froth-floatation process, as fungicides and pesticides in agriculture and in the manufacture of synthetic textiles [6, 7], and as corrosion inhibitors and quite recently in therapy for HIV infections. Metal xanthate complexes and their reaction products with a variety of Lewis bases have been extensively studied [8–10]. The soluble alkali metal xanthates are widely used in extraction and separation of Hg, Ag, Cd, and so forth [11–13]. Sodium and potassium ethyl xanthate have antidotal effects in acute mercury poisoning. Transition metal xanthate complexes have been investigated for nonlinear optical applications [14].

To the best of our knowledge, the reaction products of Ni(II) xanthates with substituted pyridine ligand as nitrogen donor (Lewis base) have not been studied that extensively, especially their crystallographic aspects. Therefore, synthesis and characterization, including the X-ray crystal structure of bis(O-amyl dithiocarbonato- $\kappa^2$ S,S')bis(4-cyanopyridine- $\kappa$ N)nickel(II), are reported here.

## 2. Experimental Section

**2.1. Materials and Measurements.** All reactions were carried out at room temperature. All reagent and chemicals were purchased from commercial sources and used as received. The O-amyl xanthate ligand was generated by the reaction between amyl alcohol and CS<sub>2</sub> in the presence of KOH and crystallized from ethanol. Elemental analysis for C, H, N and S was carried out on an elemental vario EL III, Carlo Erba 1108 elemental analyzer. Molar conductance was determined on the millimolar solution in DMF using century CC 601 Digital conductivity meter and a conductivity cell with a cell constant 1. Magnetic moment of the complex was determined at room temperature by VSM technique. Infrared spectra of the complex over the region 4000–400 cm<sup>-1</sup> were recorded on Perkin Elmer FT-IR spectrophotometer using KBr disc. The electronic spectra of the title complex were recorded in DMF on systronics 119 UV-visible spectrophotometer.

**2.2. Synthesis of Potassium Salt of O-Amyldithiocarbonate.** The potassium salt of O-amyl dithiocarbonate was prepared by the standard published method [15]. 42 g (0.75 mol) of KOH Pellets were placed in a 500 mL flask and 229 g (278 mL) of amyl alcohol was added. The resulting solution was heated under reflux for 1 hour and then allowed to cool. From the reaction mixture, liquid portion was carefully decanted off. The residual solid was transferred into a dry 500 mL flask to which 57 g (45 mL, 0.75 mol) of CS<sub>2</sub> was added slowly with constant shaking. The resulting solid yellow mass was filtered after cooling in ice on a sintered glass funnel at the pump and it was washed with three 25 mL portions of ether. The potassium O-amyl dithiocarbonate obtained was dried in a vacuum desiccator over silica gel. It was then recrystallised from absolute ethanol.

**2.3. Procedure for the Synthesis of Bis(O-amyl dithiocarbonato- $\kappa^2$ S,S')bis(4-cyanopyridine)nickel(II).** The complex bis(O-amyl dithiocarbonato)nickel(II) also known as parent compound was first prepared by adding an aqueous solution of potassium salt of O-amyl dithiocarbonate (4.0 g, 0.02 mol) to an aqueous solution of NiCl<sub>2</sub>·6H<sub>2</sub>O (2.37 g, 0.01 mol) with stirring. Dark green precipitates of the complex were obtained. The complex was filtered, washed several times with cold distilled water, and dried in a vacuum desiccator over anhydrous calcium chloride.

The title complex was then obtained by dissolving bis(O-amyl dithiocarbonato)nickel(II) (1.0 g, 0.0026 mol) prepared above in about 60 mL of acetone followed by the addition of 4-cyanopyridine (0.541 g, 0.0052 mol) and the resulting solution was allowed to be stirred for one hour. The solution was then kept undisturbed for about twenty-four hours for slow evaporation. Shining dark green crystals of the adduct were obtained which were filtered and dried in a vacuum desiccator over the anhydrous calcium chloride. Yield: 86%. Anal. Calcd. for C<sub>24</sub>H<sub>30</sub>N<sub>4</sub>O<sub>4</sub>S<sub>4</sub>Ni: C, 48.57; H, 5.09; N, 9.44; S, 21.61, found: C, 48.21; H, 4.86; N, 9.23; S, 21.32.  $\Lambda_m$  (ohm<sup>-1</sup> mol<sup>-1</sup> cm<sup>2</sup>): 22.92.  $\mu_{\text{eff}}$  (B.M.): 3.20. IR (cm<sup>-1</sup>): 1272

( $\nu_{\text{as}}\text{C}-\text{O}-\text{C}$ ), 1140 ( $\nu_s\text{C}-\text{O}-\text{C}$ ), 1043 ( $\nu\text{C}-\text{S}$ ). UV (cm<sup>-1</sup>): 10512 ( $\nu_1$ ), 17921 ( $\nu_2$ ), 27720 ( $\nu_3$ ).

**2.4. X-Ray Crystal Analysis.** Detailed structural information on the conformation of the molecule in the crystalline state was obtained by employing X-ray crystallographic techniques. A dark green block-shaped single crystal of the title compound measuring 0.30 × 0.20 × 0.20 mm was picked up for X-ray intensity data collection on a CCD area-detector diffractometer (*X'calibur system-Oxford diffraction make*, UK) which is equipped with graphite monochromated MoK $\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ). A total number 18668 of reflections were collected, of which 2004 reflections were treated as observed ( $I > 2\sigma(I)$ ). Data were reduced using CrysAlis RED software [16]. Data were corrected for Lorentz-polarization and absorption factors. The structure was solved by direct methods using SHELXS97 and refined by SHELXL97 [17]. All the hydrogen atoms were located from a difference electron density map and their positional and isotropic thermal parameters were included in the refinement. The final refinement cycle yielded an *R*-factor of 0.0499 and wR ( $F^2$ ) = 0.0919 for the observed data. The residual electron density range is  $-0.263 < \Delta\rho < 0.358 \text{ e\AA}^{-3}$ . The CIF for this structure has been deposited at Cambridge Crystal Data Centre (CCDC number 893165).

## 3. Results and Discussion

The synthetic methodology followed for preparing the complex was developed in our laboratory. The complex was prepared by stirring the parent compound with 4-cyanopyridine in acetone. The complex of bis(O-amyl dithiocarbonato)nickel(II) with 4-cyanopyridine is a shining dark green crystalline solid. The adduct is soluble in common organic solvents such as acetone, chloroform, DMF, and DMSO. The complex obtained in this study gave analytical result which is concordant with the formula assigned. On the basis of elemental analysis, the adduct isolated having 1:2 stoichiometry is assigned the formula Ni(S<sub>2</sub>CO-n-C<sub>5</sub>H<sub>11</sub>)L<sub>2</sub> (where L = 4-cyanopyridine).

**3.1. Conductance and Magnetic Measurements.** The molar conductivity value calculated from the conductivity measured on millimolar solution of the title complex in DMF supports the neutral and nonionic nature of the complex [18]. The 1:2 adduct of bis(O-amyl dithiocarbonato)nickel(II) with 4-cyanopyridine exhibits magnetic moment value (3.20 B.M.) which is in agreement with magnetic moment values observed for paramagnetic octahedral complexes of nickel(II) [19, 20].

**3.2. IR Spectra.** The assignment of the significant IR spectral bands of the adduct is also presented. Infrared spectra of the present complex were recorded in order to investigate the mode of coordination of the Xanthate and heterocyclic ligand with the central metal ion. The complex exhibits characteristic bands in the range 1280–996 cm<sup>-1</sup> that are related to the vibrations of S<sub>2</sub>COR group [21, 22]. The bands

at 1272, 1140, and 1043 cm<sup>-1</sup> are the characteristic absorption of  $\nu_{as}$ (C—O—C),  $\nu_s$ (C—O—C), and  $\nu$ (C≡S), respectively. The band occurring below 400 cm<sup>-1</sup> may be assigned to  $\nu$ (Ni—S) stretching mode. In order to decide the coordination site in cyanopyridine ligand, a study of vibrations of pyridine ring and C≡N stretching frequency can be of much help. It is established that there should be an increase in  $\nu$ (C≡N) by at least 30 cm<sup>-1</sup> if a cyanopyridine coordinates with the metal using nitrile nitrogen atom [23]. But on the other hand, if —CN group coordinates using  $\pi$ -electrons,  $\nu$ (C≡N) must show a negative shift [24, 25]. The C≡N stretching frequency in free 4-cyanopyridine ligand falls at 2225 cm<sup>-1</sup> but in the present Complex, the C≡N stretching vibration occurs around 2220 cm<sup>-1</sup> showing no appreciable negative shift in  $\nu$ (C≡N). Thus the possibility of coordination of 4-cyanopyridine through its cyano nitrogen atom with metal is ruled out and it can be concluded that 4-cyanopyridine coordinates with metal through its ring nitrogen atom. This has been further confirmed by examining ring breathing vibrations for 4-cyanopyridine which have been found to shift to higher energy position on complexation indicating that ligand-metal coordination occurs through ring nitrogen atom.

**3.3. Electronic Spectra.** The electronic spectral measurement was used for assigning the stereochemistry of the title complex based on the positions and number of d-d transition peaks. The electronic spectra of 1:2 adduct of bis(O-amylidithiocarbonato)nickel(II) with 4-cyanopyridine were recorded in DMF in the range 10,000–50,000 cm<sup>-1</sup> at room temperature. It shows three broad bands  $\nu_1$ ,  $\nu_2$ , and  $\nu_3$  around 10,000, 18,000, and 28,000 cm<sup>-1</sup>, respectively. These bands can be assigned due to  $^3A_{2g} \rightarrow ^3T_{2g}(F)$ ,  $^3A_{2g} \rightarrow ^3T_{1g}(F)$  and  $^3A_{2g} \rightarrow ^3T_{1g}(P)$  transitions, respectively. Appearance of these three broad bands shows that adduct is having trans octahedral geometry around nickel(II) metal ion [26]. In addition absorption bands observed in the region from 35,000 to 40,000 cm<sup>-1</sup> can be assigned to M → L charge transfer transitions.

**3.4. Description of the Crystal Structure.** The crystallography data for bis(O-amylidithiocarbonato- $\kappa^2$ S,S')bis(4-cyanopyridine- $\kappa$ N)nickel(II) are presented in Table 1. Selected bond distances and bond angles for nonhydrogen atoms are listed in Table 2. A general view of the molecule indicating atom numbering scheme is shown in Figure 1. *Ortep-3* software [27] was used for making the thermal ellipsoids. The geometry of the molecule was calculated using *PLATON* [28] and *PARST* [29] software.

The asymmetric unit comprises half molecule and Nickel(II) cation lies on an inversion centre. The Ni(II) atom is coordinated by a distorted octahedral arrangement of four sulphur atoms from two chelating xanthate ligands and two nitrogen atoms from two cyanopyridines ligands. The Ni1—N10 bond length is 2.121(3) Å. The Ni—S bond lengths involving the xanthate ligands are 2.440(9) Å for Ni1—S1 and 2.433(1) Å for Ni1—S2. The bond angles in the pyridine

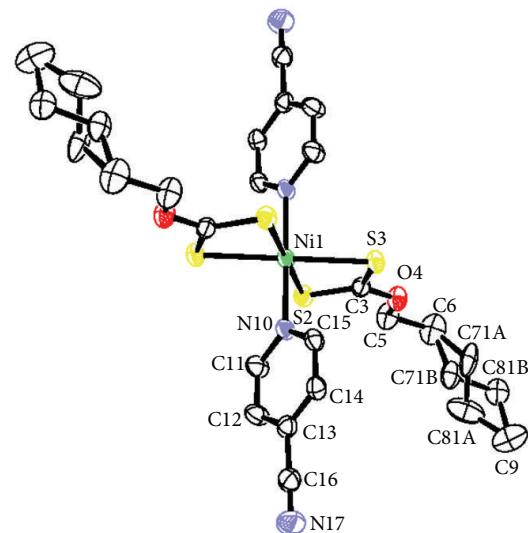


FIGURE 1: ORTEP view of the molecule with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radii.

ring vary from 117.0(3) to 123.8(3) $^\circ$ , with the average value being 119.9(8) $^\circ$ , and these values of bond angles and bond distances are in good agreement with those reported for other analogous Ni-dithiocarbonato complexes [30–32]. The C—S bond lengths involving the xanthate ligands are S1—C3 = 1.691(4) and S2—C3 = 1.691(4) Å. These values show double bond character due to the delocalization over the two C—S bonds [33]. The plane formed by two sulfur, nickel and carbon atoms (Ni1, S1, C3, and S2) is planer and the dihedral angle between this plane and the plane of cyanopyridine ring is 89.14(8) $^\circ$  meaning thereby that both these planes are held almost perpendicular to each other. The cyanogroup bound to C13 atom of the pyridine ring lies in the same plane as that of pyridine ring. It is evident from the least-squares plane calculation of the pyridine ring where the deviation of C16 atom of cyano group from the plane of pyridine ring is — 0.0767(4). The amyl chain attached to the dithiocarbonato group contains disorder over two sets of sites with occupancy ratios of 0.595 : 0.405. The O4—C3 bond distance [1.320(4) Å] is shorter than O4—C5 [1.453(5) Å] as a consequence of the hybridization of the carbon atom. The short value of bond distance for O4—C3 is consistent with a significant contribution of the resonance form of the xanthate anion that features a formal C=O and a negative charge on each of S atom [34].

The packing of the molecule within the unit cell viewed down the *y*-axis is shown in Figure 2. Molecules in the unit cell are arranged in the form of layers and within the layers the molecules are parallel to each other and each molecule is linked to the adjacent molecule with the help of C—H $\cdots$ N hydrogen bond to form infinite chains. Chains of the hydrogen-bonded molecules are packed with van der Waals contact.

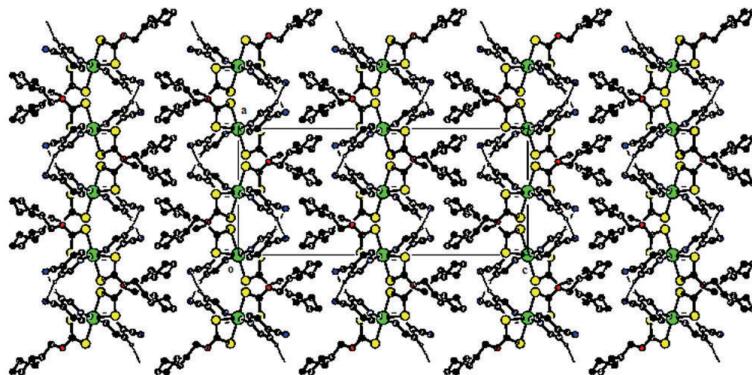


FIGURE 2: Molecular packing along the *b*-axis, indicating the formation of C14–H14···N17 intermolecular hydrogen bond (symmetry: 3/2-*x*, 1/2-*y*, *z*).

TABLE 1: Crystallographic data for the title compound.

CCDC number	893165
Empirical formula	C <sub>24</sub> H <sub>30</sub> N <sub>4</sub> O <sub>2</sub> S <sub>4</sub> Ni
Formula weight	593.47
Temperature (K)	293(2)
Shape	Block shaped
Color	Dark green
Crystal system	Orthorhombic
Space group	P b c a
Unit cell dimensions (Å, °)	
<i>a</i>	11.455(5)
<i>b</i>	9.602(4)
<i>c</i>	26.374(1)
$\alpha$	90.00
$\beta$	90.00
$\gamma$	90.00
Volume (Å <sup>3</sup> ), Z	2900.8(2), 4
Dimension (mm <sup>3</sup> )	0.30 × 0.20 × 0.20
$D_c$ (gcm <sup>-3</sup> )	1.359
$\mu$ (mm <sup>-1</sup> )	0.984
$F(000)$	1240
2θ range for data collection (°)	3.56–26.00
Limiting indices	$-14 < h < 14$ $-11 < k < 11$ $-32 < l < 32$
Reflection collected	18668
Independent reflection	2850
Goodness-of-fit on $F^2$	1.104
Data/restraint/parameter	2850/6/179
Final <i>R</i> indices [ $I > 2\sigma(I)$ ]	0.0499
<i>R</i> indices (all data)	0.0781
Largest difference peak and hole (e nm <sup>-3</sup> )	358 and -263

#### 4. Conclusion

The characterization data as reported in this paper consists of conductivity measurement; spectral and magnetic studies

TABLE 2: Selected bond distances (Å) and bond angles (°).

Bond distances			
Ni1–Ni0	2.121(3)	Ni1–S2	2.433(1)
Ni1–S1	2.441(9)	S1–C3	1.691(4)
S2–C3	1.691(4)	C3–O4	1.320(4)
O4–C5	1.453(5)	C5–C6	1.482(6)
N10–C11	1.338(4)	N10–C15	1.341(4)
Bond angles			
N10–Ni1–S2	91.10(8)	N10–Ni1–S1	90.29(8)
S2–Ni1–S1	105.70(3)	C3–S1–Ni1	82.22(1)
C3–S2–Ni1	82.47(1)	O4–C3–S2	122.9(3)
O4–C3–S1	116.1(3)	S2–C3–S1	121.0(2)
N17–C16–C13	177.6(5)		

reveal that the adduct is octahedral, nonelectrolytic, and paramagnetic in nature. There exists half molecule of bis(*O*-amyldithiocarbonato- $\kappa^2$ S,S')bis(4-cyanopyridine- $\kappa$ N) nickel(II) in the asymmetric unit cell. The dithio and cyanopyridine rings are held at right angles to each other. Molecules within the unit cell form layers. CCDC number 837852 contains the supplementary crystallographic data for the compound bis(*O*-amyldithiocarbonato- $\kappa^2$ S,S')bis(4-cyanopyridine- $\kappa$ N) nickel(II). The data can be obtained free of charge via [http://www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif) by e-mailing [data\\_request@ccdc.cam.ac.uk](mailto:data_request@ccdc.cam.ac.uk), or by contacting The Cambridge Crystallography Data Centre, 12 Union Road, Cambridge.

#### Conflict of Interests

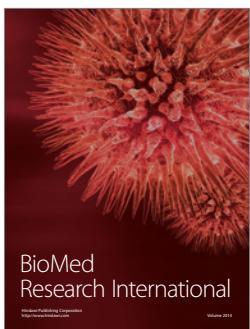
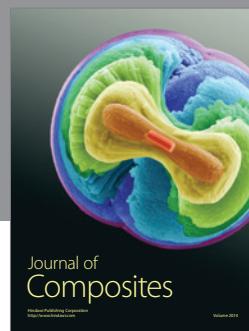
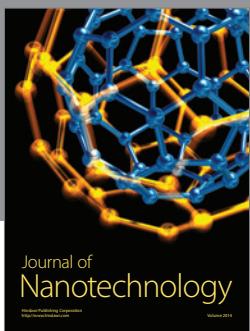
The authors declare that there is no conflict of interests regarding the publication of this paper.

#### Acknowledgment

One of the authors (R. Kant) acknowledges the Department of Science and Technology, Government of India, for sanctioning Single Crystal X-ray diffractometer as a National Facility under research Project no. SR/S2/CMP-47/2003.

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