

## Nickel (II)-complex ligand as host for Potassium ion guest. Spectroscopic characterization and X-ray structure determination

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**Abstract:** The title complex,  $\{[Ni(hab)]K[(hab)Ni]\} \cdot SCN$  (**H<sub>2</sub>hab** is bis(2-hydroxy-3-methoxybenzylidene)-1,2-diaminobenzene) crystallizes in the Monoclinic space group  $P2_1/c$  with unit cell dimensions  $a = 11.5891(13) \text{ \AA}$ ,  $b = 17.307(2) \text{ \AA}$ ,  $c = 19.781(2) \text{ \AA}$ ,  $\beta = 92.368(10)^\circ$ ,  $V = 3964.2(8) \text{ \AA}^3$ ,  $Z = 4$ ,  $R_1 = 0.106$  and  $wR_2 = 0.305$ . The  $Ni^{II}$  cation is in a  $N_2O_2$  inner and has a distorted square-planar coordination environment. The  $Ni^{II}$  cation is linked by two imino nitrogen atoms and two bridged phenolate oxygen atoms. The potassium cation is octacoordinated in  $O_8$  donor site and linked by four bridged phenolate oxygen atoms and four methoxy oxygen atoms. In the crystal, the  $\{[Ni(hab)]K[(hab)Ni]\}^+$  cations form layers parallel to the  $ab$  plane. The  $SCN^-$  anions are located in the interlayer space and connect the layers via  $C-H \cdots N$  and  $C-H \cdots S$  hydrogen bonds, forming three dimensional network.

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### I. Introduction

In the past decades a numerous heteronuclear coordination compounds derived from ligand having two compartments of different sizes have been reported [1-4]. These types of compounds can be synthesized after isolating the mononuclear complex in which the metal ion occupies the smaller cage. The reactions of these mononuclear species with metallic centers of different natures can lead compounds with very varied structures [5-8]. The control of the experimental conditions during the synthesis reactions made it possible to construct structures with interesting physico-chemical properties [9, 10]. Both polynuclears and co-crystals were obtained with salen type ligands [11-13]. Complexes with  $Na^+$  ions are widely studied because of their implications for molecular recognition of biological systems [14-16]. The study of the structures of these complexes shows that the  $Na^+$  ion systematically occupies the open site while the transition metal is in the closed  $N_2O_2$  site of smaller size [17-20]. The **H<sub>2</sub>hab** (bis (2-hydroxy-3-methoxybenzylidene)-1,2-diaminobenzene) Schiff base obtained by condensation of 3-methoxysalicylaldehyde and o-phenylenediamine allows to generate several types of 3d/4f and 3d/Na heteronuclear complexes in which the 3d metal invariably occupies the  $N_2O_2$  site [17], [21-24]. It is in this context that we report the synthesis of a complex  $Ni^{2+}/K^+$  formulated as follows  $\{[Ni(hab)]K[Ni(hab)]\} \cdot NCS$ . The compound is obtained by initially isolating the mononuclear nickel (II) complex before reacting the latter with potassium thiocyanate to obtain a heterotrinnuclear complex.

### II. Experimental section

#### 2.1. Starting materials and Instrumentations

Commercially available 1,2-diaminobenzene, 3-methoxysalicylaldehyde,  $NiCl_2 \cdot 6H_2O$  and  $KSCN$  were purchased from Aldrich and used without further purification. The analyses for carbon, hydrogen and nitrogen were carried out using a LECO CHNS-932 instrument. The IR spectra were recorded as KBr discs on a Perkin Elmer Spectrum Two spectrophotometer (4000–400  $cm^{-1}$ ). UV–VIS spectra were recorded in methanol solution concentration of  $10^{-3} \text{ M}$  at  $25^\circ C$  and wavelength was reported in nm using a Perkin Elmer Lambda 365 UV/VIS

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spectrophotometer. The molar conductance of  $10^{-3}$  M solution of the metal complex in DMF was measured at 25 °C using a WTW LF-330 conductivity meter with a WTW conductivity cell. Room temperature magnetic susceptibility of the powdered sample was measured using a Johnson Matthey scientific magnetic susceptibility balance (calibrant  $\text{Hg}[\text{Co}(\text{SCN})_4]$ ).

## 2.2. Synthesis of the metal-ligand complex

The complex  $[(\text{NiL})\cdot(\text{H}_2\text{O})]$  was prepared according to the experimental process described in the literature with slight modification. To a solution of o-vanillin (0.7602 g, 5 mmol) in 10 mL of ethanol was added 5 mL aqueous solution of  $\text{Na}_2\text{CO}_3$  0.2 M and 10 mL of an ethanolic solution of  $\text{NiCl}_2\cdot 6\text{H}_2\text{O}$  (0.1185g, 5 mmol). The mixture is refluxed for 30 mn and a solution of 1,2-diaminobenzene (0.250 g, 2.5 mmol) in 10 mL of ethanol was added. The resulting mixture was refluxed for 120 min. On cooling a brown precipitate which appears was filtered off, washed with  $3\times 10$  mL of ether and dried in air yielding an orange compound formulated as  $[(\text{NiL})\cdot\text{H}_2\text{O}]$ . Yield: 80%. M.p.: 245 °C. FT-IR (KBr,  $\nu$ ,  $\text{cm}^{-1}$ ): 3373 (OH) (br, water), 1606 (C=N) 1586 (C=C), 1543 (C=C), 1441 (C=C), 1439, 1340, 1243, 1111, 733. UV-visible ( $\lambda$ , nm): 511. Anal. calcd. for  $\text{C}_{22}\text{H}_{20}\text{NiN}_2\text{O}_5$ : C, 58.58; H, 4.47; N, 6.21. Found: C, 55.49; H, 4.39; N, 6.18 %.  $\Lambda_M$  ( $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$ ): 10.

## 2.3. Synthesis of the trinuclear complex

The filtrate of a mixture of KSCN (0.0972 g, 1 mmol) in 20 mL of absolute ethanol was added to a DMF solution (5 mL) of the  $[(\text{NiL})\cdot\text{H}_2\text{O}]$  (0.4511 g, 2 mmol). The resulting solution was heated under reflux for 2 hours. After cooling the solution was filtered and the filtrate was kept at 298 K. After two weeks crystals suitable for X-ray diffraction were collected and formulated as  $\{[(\text{NiL})_2\text{K}]\cdot(\text{SCN})\}$ . Yield: 85%. FT-IR (KBr,  $\nu$  ( $\text{cm}^{-1}$ )): 2053 (S=C=N), 1606 (C=N), 1584 (C=C), 1545 (C=C), 1463 (C=C), 1440, 1238, 1191, 731. Anal. calcd. for  $\text{C}_{45}\text{H}_{36}\text{Ni}_2\text{KN}_5\text{O}_8\text{S}$ : C, 56.10; H, 3.77; N, 7.27; S, 3.33 %. Found: C, 56.07; H, 3.73; N, 7.22; S, 3.30 %.  $\Lambda_M$  ( $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$ ): 128.

## 2.4. X-ray data collection, structure determination and refinement

Diffraction: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright Mo-K $\alpha$  rotating anode generator ( $\lambda = 0.71075$  Å) with VHF Varimax optics (100  $\mu\text{m}$  focus). Cell determination, data collection, data reduction, cell refinement and absorption correction: *CrysAlisPro* 1.171.39.34b. [25]. Gaussian absorption correction was applied. Structure solution and refinement were performed using *SHELXT* [26] and *SHELXL-2014/7* [27]. All hydrogen atoms were added in calculated positions and refined in riding mode on the parent atom. Molecular graphics were generated using *ORTEP-3* [28].

**Table-1.** Crystallographic data and refinement parameters for the complex.

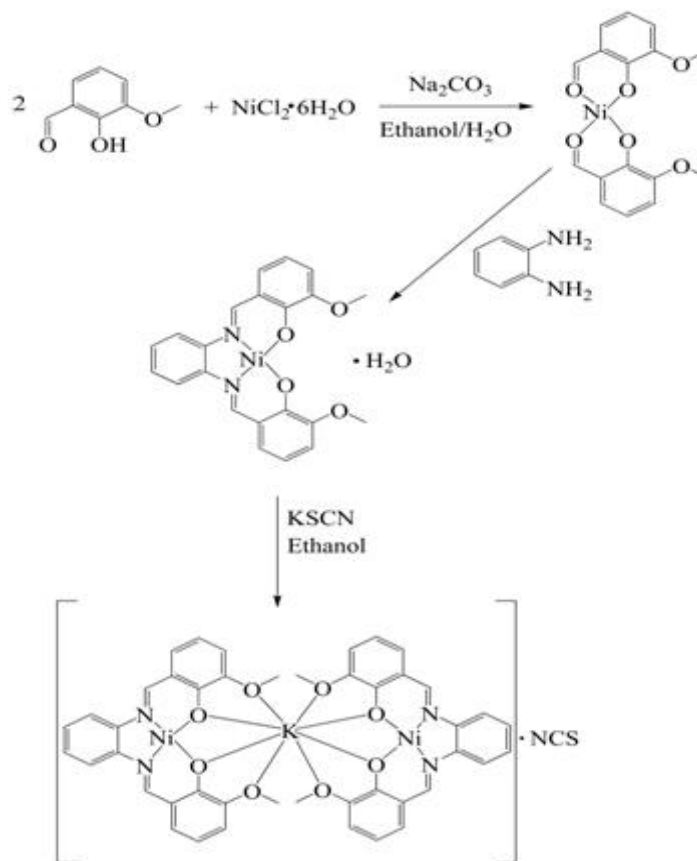
Formula of refinement	$\text{C}_{44}\text{H}_{36}\text{KN}_4\text{Ni}_2\text{O}_8\cdot\text{CNS}$
Molecular weight (g/mol)	963.37
Crystal system	Monoclinic
Space group	P21/c
<i>a</i> (Å)	11.5891 (13)
<i>b</i> (Å)	17.307 (2)
<i>c</i> (Å)	19.781 (2)
$\beta$ (°)	92.368 (10)
<i>V</i> (Å <sup>3</sup> )	3964.2 (8)
<i>Z</i>	4
<i>D<sub>c</sub></i> (g cm <sup>-3</sup> )	1.694
Crystal size (mm)	0.28 × 0.27 × 0.18
Crystal color	metallic dark red
Crystal habit	block
Mo <i>K</i> $\alpha$ (Å)	0.71073
$\mu$ (mm <sup>-1</sup> )	1.17
<i>T</i> (K)	293
$\theta_{\text{min}}$ , $\theta_{\text{max}}$	2.569, 25.350
<i>hkl</i> ranges	-10, 13 ; -14, 20 ; -23; 23
Measured reflections	14247
Independent reflections	7230
Reflections with $[I > 2\sigma(I)]$	4563
No. of parameters	563
<i>R</i> <sub>int</sub>	0.064
<i>R</i> <sub>1</sub> (F) [ $I > 2\sigma(I)$ ]	0.106
<i>wR</i> <sub>2</sub> ( <i>F</i> <sup>2</sup> )	0.305

Goodness-of-fit	1.09
$\Delta\rho_{\max}, \Delta\rho_{\min}$ (e $\text{\AA}^{-3}$ )	0.99, -1.00

### III. Result and discussion

#### 3.1. General Studies

The aim of the present work was to study the coordination of nickel(II)-ligand to potassium cations in order to synthesize a trinuclear complex. The condensation of 3-methoxysalicylaldehyde with 1,2-diaminobenzene in the presence of  $\text{Ni}^{\text{II}}$  afforded a metal-ligand which can act as a chelating ligand with the cation  $\text{K}^{\text{I}}$ , to produce a trinuclear complex (Scheme 1). Accordingly, the dimethylformamide solution of metal-ligand was treated with KSCN solution in absolute ethanol in 2:1 molar ratio. The resulting solution was refluxed during two hours before filtered-off and left under slow evaporation. Crystals suitable for X-ray diffraction were isolated after two weeks. The elemental analysis results are in good agreement with the calculated values for the metal-ligand and the trinuclear complex which are non-hygroscopic and stable in air. Both metal-ligand and trinuclear complexes are diamagnetic. The metal ligand molar conductivity  $\Lambda$  is  $5 \Omega^{-1} \text{cm}^2 \text{mol}^{-1}$  indicating a non-electrolytic nature in DMF solution, while the trinuclear complex gives  $128 \Omega^{-1} \text{cm}^2 \text{mol}^{-1}$  in accordance with a 1:1 electrolyte in DMF [29]. The infrared spectra of the metal-ligand exhibits a band at  $1606 \text{ cm}^{-1}$  characteristic of C=N chromophore coordinated to the metal center through the azomethine atom. Additional bands attributable to C=C of the aromatic rings are also pointed. On comparison the spectra of the trinuclear complex exhibits additional band at  $2053 \text{ cm}^{-1}$  which is attributable to the S=C=N uncoordinated moiety [30]. Both bands characteristic of C=C and C=N remain unchanged. The UV-Vis spectrum of both nickel(II)-ligand and the trinuclear compounds are recorded in methanol solution in the range of 200–1000 nm. The two spectra are comparable and show one absorption band attributable to the  $\pi \rightarrow \pi^*$  transition of the aromatic rings in the range 280–295 nm and an additional band in the range 310–330 nm which can be attributed to the  $n \rightarrow \pi^*$  transitions of the C=N chromophore. The high intensity bands due to LMCT appeared near 450 nm and the band at 511 nm are consistent with the observed d-d transitions of a diamagnetic square planar nickel(II) center complex [31].



Scheme 1. Synthetic scheme for metal-ligand and the heterotrinuclear complex preparation

**Table-2:** Selected bonds lengths (Å).

Ni1—O3	1.832 (8)	K1—O4	2.654 (9)
Ni1—N1	1.853 (8)	K1—O1	2.596 (7)
Ni1—O2	1.854 (7)	K1—O2	2.673 (7)
Ni1—O3	1.832 (8)	K1—O3	2.707 (8)
Ni1—N2	1.866 (9)	K1—O4	2.654 (9)
Ni2—N3	1.868 (9)	K1—O5	2.630 (8)
Ni2—N4	1.844 (9)	K1—O6	2.725 (7)
Ni2—O6	1.839 (7)	K1—O7	2.706 (7)
Ni2—O7	1.842 (7)	K1—O8	2.604 (8)
C1—O1	1.443 (11)	S1—C45	1.63 (2)
O3—Ni1—N1	175.1 (3)	O2—K1—O3	54.5 (2)
O3—Ni1—O2	83.8 (3)	O2—K1—O6	144.8 (2)
N1—Ni1—O2	95.4 (3)	O7—K1—O6	53.6 (2)
O3—Ni1—N2	96.7 (4)	O6—Ni2—O7	83.4 (3)
N1—Ni1—N2	84.2 (4)	O6—Ni2—N4	175.8 (3)
O2—Ni1—N2	178.9 (4)	O7—Ni2—N4	95.0 (4)
O8—K1—O5	167.2 (3)	O6—Ni2—N3	95.1 (4)
O1—K1—O4	171.3 (3)	O7—Ni2—N3	178.3 (4)
O4—K1—O7	110.4 (3)	N4—Ni2—N3	86.6 (5)

**Table 3:** Hydrogen bonds

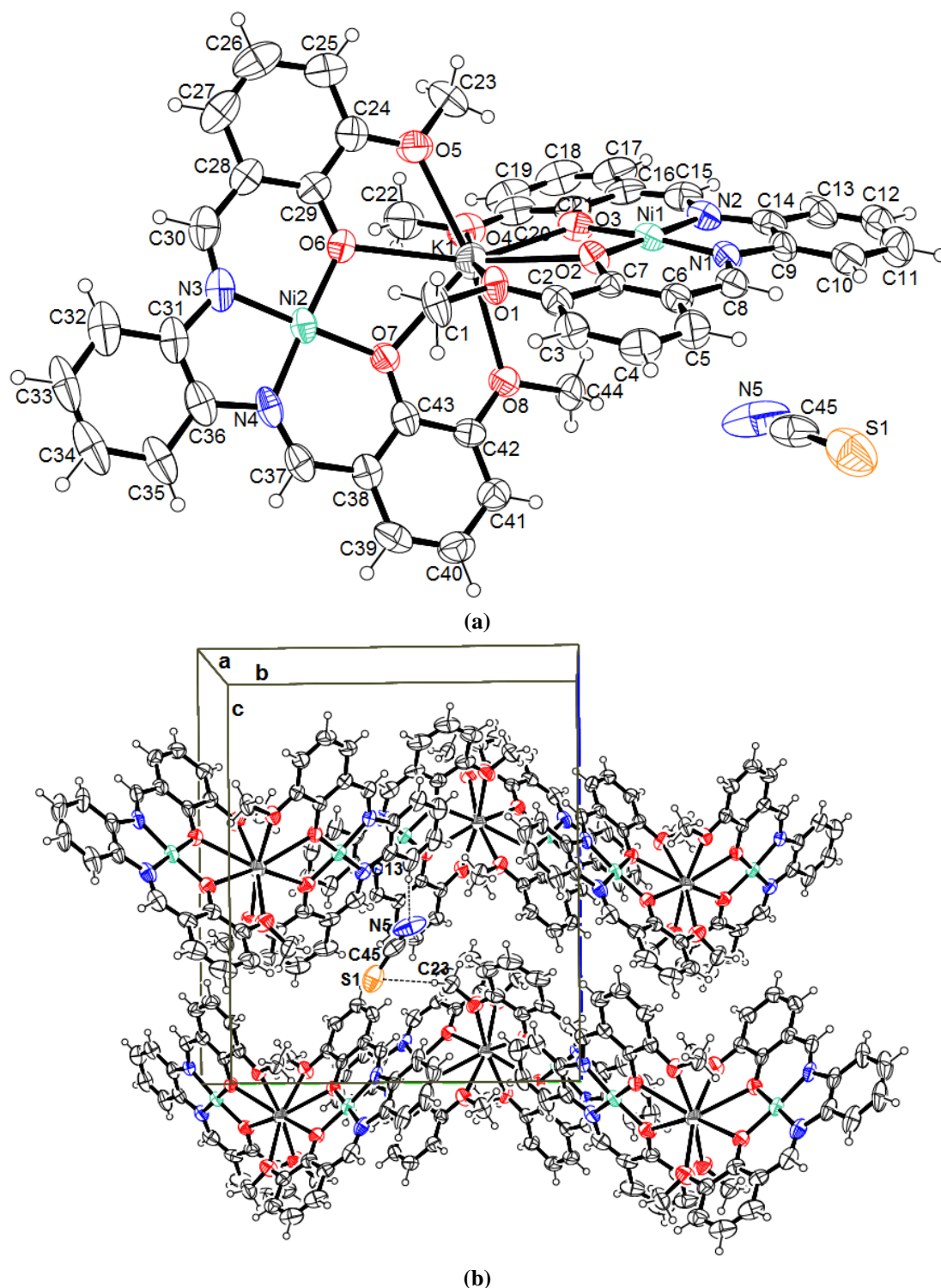
C13—H13...N5 <sup>i</sup>	0.93	2.58	3.50 (2)	169.1
C22—H22A...S1 <sup>ii</sup>	0.96	3.03	3.978 (15)	169.7
C23—H23A...S1 <sup>iii</sup>	0.96	2.99	3.936 (15)	170.5

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

### 3.2. Crystal structure of the complex {[Ni(hab)]K[(hab)Ni]}·SCN

The complex formulated as {[Ni(hab)]K[(hab)Ni]}·SCN (**H<sub>2</sub>hab** is bis(2-hydroxy-3-methoxybenzylidene)-1,2-diaminobenzene) crystallizes in the monoclinic space group P2<sub>1</sub>/c. Details for the structure solution and refinement are summarized in Table 1. The selected bond distances and angles are listed in Tables 2. The structure is showed in Fig. 1a. In the complex each of the two nickel(II) ions occupies the N(imino)<sub>2</sub>O(phenoxo)<sub>2</sub> compartment of on [hab]<sup>2-</sup> moiety, while the potassium(I) ions is coordinated by the two open O(phenoxo)<sub>2</sub>O(methoxy)<sub>2</sub> compartment of the two [hab]<sup>2-</sup> moieties. In the title complex, C<sub>44</sub>H<sub>36</sub>KN<sub>4</sub>Ni<sub>2</sub>O<sub>8</sub>·SCN, each Ni atom located in N<sub>2</sub>O<sub>2</sub> inner is tetracoordinated. The Ni<sup>II</sup> cations are in a slightly distorted square planar geometry with all *transoid* N—Ni—O angles being 175.1 (3)° and 178.9 (4)° for Ni1 and 175.8 (3)° and 178.3 (4)° for Ni2. The values of the *cisoid* angles for the two Ni atoms are in the range [83.4 (3)–96.7 (4) °]. The values of the angle sum around each Ni is 360.1° proving the slight distortion of the environment around the Ni atoms. The Ni1—O2 and Ni1—O3 distances of 1.854 (7) Å and 1.832 (8) Å and Ni2—O6 and Ni2—O7 distances of 1.839 (7) Å and 1.842 (7) Å are comparable and are longer than those found in a similar complex [32]. The Ni—N distances in the complex which fall in the range [1.844 (9)–1.868 (9) Å] are comparable to Ni—N distances found in a reported complex [33]. The potassium cation has an O<sub>8</sub> donor set made up by four  $\mu_2$ -bridging phenolate oxygen atoms and four methoxy oxygen atoms from the ligand molecules acting in unidentate fashion and forming a distorted polyhedron. The K<sup>+</sup> cation share four phenolate oxygen atoms with Ni atoms.

The cations Ni<sup>II</sup> and K<sup>I</sup> are arranged in the following sequence: Ni—K—Ni. The metal atoms are found at non-linear positions [Ni—K—Ni, 162.44 (9)°] as shown in Fig. 1a. The Ni—K distances of 3.759 (3) Å and 3.788 (3) Å distances are found to be longer than those found in heteronuclear Ni/K complexes [34, 35]. The molecules complex form a zigzag layers parallel to the *ab*-plane Fig. 1b. Within the zigzag chain, the cations SCN<sup>-</sup> which are located in the interlayer space, connect the layers via weak hydrogen bond of types C—H...N and C—H...S resulting in three dimensional network.



**Figure 1:** ORTEP plot (30% probability ellipsoids) showing the molecule structure of the heterotrinnuclear complex (a) and the hydrogen bonds (b) in the complex.

#### IV. Conclusion

The Schiff base which is widely used to prepare dinuclear complexes is also capable of giving polynuclear structures. The main characteristic in the case of the use of a transition metal and an element of the block seems to be the systematic occupation of the  $N_2O_2$  site by the transition metal. All examples to our knowledge give this type of behavior. Using this ligand, we have isolated and determined the X-ray diffraction structure a hetero-tri-nuclear Ni-K-Ni complex. As expected, the  $Ni^{II}$  ion occupies the  $N_2O_2$  site while the  $K^I$  cation occupies the  $O_8$  site. The three dimensional network is formed with zigzag layers parallel to the *ab*-plane. The  $SCN^-$  anion is located in the interlayer and connect layers via weak hydrogen bond.

## V. Supplementary material

CCDC 1861725 contains the supplementary crystallographic data for the reported complex. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44)-1223-336-033; e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk). or [www: http:// www.ccdc.cam.ac.uk](http://www.ccdc.cam.ac.uk)).

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