

# Syntheses, characterisation and structural analysis of new cobalt(II,III) complexes incorporating a pyridoxal-semicarbazone ligand

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## Abstract

The reaction between 3-hydroxy-5-hydroxymethyl-2-methyl-4-pyridinecarboxaldehydesemicarbazide (pyridoxal-semicarbazide, PLSC, H<sub>2</sub>L) and appropriate sulphate, chloride and acetate Co(II) salts in water mixtures, resulted in the formation of new cobalt(II,III) complexes: [Co(H<sub>2</sub>L)(SO<sub>4</sub>)(H<sub>2</sub>O)<sub>2</sub>](**1**), [Co(HL)<sub>2</sub>]NCS·H<sub>2</sub>O(**2**) and [Co(HL)<sub>2</sub>]·H<sub>2</sub>O(**3**). The complexes were characterized by elemental analysis, conductometric and magnetochemical measurements, IR spectra and X-ray diffraction.

*Keywords: Cobalt(II,III) complexes, Pyridoxal semicarbazone, Synthesis, Structural*

## 1. Introduction

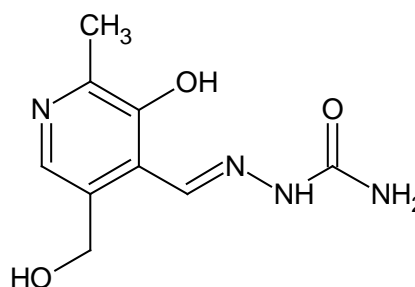
Cancer is one of the primary targets regarding medicinal chemistry. Even though platinum-based complexes had been the primary focus of the research on chemotherapy agents [1-3], the interests in this field have shifted to non-platinum-based agents [4-11], in order to find different metal complexes with less side effects and similar, or better, cytotoxicity.

A wide variety of metal-complexes based on titanium, gallium, germanium, palladium, gold, copper, ruthenium and tin are being intensively studied as platinum replacements [4-11]. Cobalt is another option for the central metal in complex compound. Cobalt (II, III)-based complexes appear to be very promising candidates for anticancer therapy, an idea supported by a considerable number of research articles describing the synthesis and cytotoxic activities of numerous cobalt complexes [12].

On the other hand it is very important role of the ligand in the complex, which can increase the biological activity of the complex. Semicarbazones (SC) can act as biologically active antibacterial agents and are excellent chelating ligands of different denticity resulting in the synthesis of a great number of transition metal complexes containing these ligands [13,14]. Several of these complexes, due to their stability and intense colour, have been suggested as analytical reagents [15]. Furthermore, complexes incorporating either SC-based ligand exhibit a wide variety

of biologically important properties, such as antiviral, antitumor and anti-inflammatory activities. Dehydration

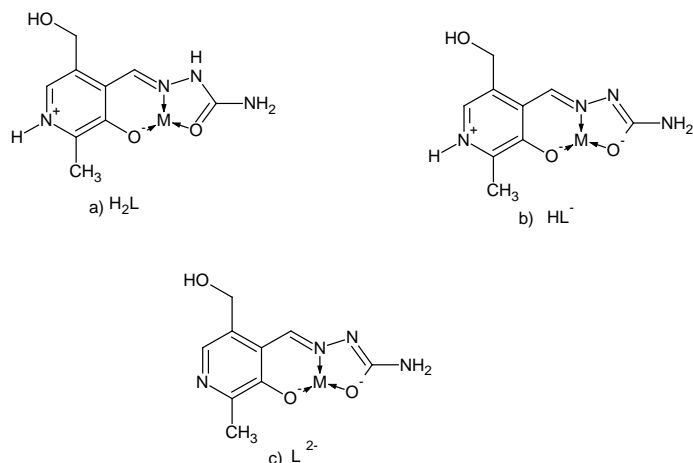
of SC with pyridoxal moiety (3-hydroxy-5-hydroxymethyl-2-methyl-pyridine-4-carbaldehyde) results in the formation of Schiff base ligands PLSC (see Scheme 1), respectively [16].



**Scheme 1.** Structural formula of PLSC ligand

The choice of the coordinated ligand(s) because besides being the integral part of biologically active complexes these organic molecules (ligands) can exert biological activity on their own [17]. Several research articles have been published reporting the synthesis and biological activity of transition metal complexes incorporating PLSC ligand [18-28], including a review article [24] and monograph [16].

Coordination chemistry of PLSC-based complexes proved to be very interesting as this ligand can exist in neutral, mono- and dianionic forms depending on pH while its most predominant tridentate coordination mode, achieved through hydrazine nitrogen, phenolic and carbonyl oxygen atoms, allows it to be an excellent chelating ligand [16]. The synthesis, physical properties, structural analysis, as well as the biological activities, of several transition metal complexes incorporating PLSC [16] have already been described. The tridentate coordination mode is predominant for PLSC and it can adopt three different forms in the coordination sphere of a transition metal, namely neutral (but zwitterionic), monoanionic (hydrazine deprotonation) and dianionic (both pyridinium and hydrazine deprotonation) forms (see Scheme 2).



Scheme 2. Coordination modes and ligand forms: a) neutral, b) mono- and c) dianionic

Furthermore, this ligand, based on semicarbazone and pyridoxal moieties (forms found in vitamin B6), has an enormous potential as a biologically active reagent as it has been demonstrated that transition metal complexes incorporating semicarbazones show biological activity. In particular, with the regard to biological importance, cooper (II) complexes with PLSC ligands show anticancer activity [29].

Given all the above, we have the desire to give contribute with this work, regarding further research into the synthesis of complexes with ligand PLSC. Thus, we wish to report the synthesis, characterisation and X-ray analyses of several Co (II, III) complexes incorporating PLSC ligand namely  $[Co(H_2L)(SO_4)(H_2O)_2]$  (1),  $[Co(HL)_2]NCS \cdot H_2O$  (2) and  $[Co(HL)_2] \cdot H_2O$  (3).

## 2. Experimental procedure

All commercially obtained reagent-grade chemicals were used without further purification, except for the ligand, which was prepared according to the previously described procedure [16].

### 2.1.Synthesis of complexes

#### Synthesis of $[Co(H_2L)(SO_4)(H_2O)_2]$ (1)

0.26 g (1 mmol) of PLSC was dissolved in  $H_2O$  ( $10 \text{ cm}^3$ ) by heating followed by the addition of 0.15g (1 mmol) of  $CoSO_4$  into the warm solution. After a couple of hours, separation of purple-coloured crystals suitable for the X-ray analysis was observed. The crystals were filtered and washed with EtOH. Yield: 0.19 g (78%).

#### Synthesis of $[Co(HL)_2]NCS \cdot H_2O$ (2)

0.26 g (1 mmol) of PLSC and 0.28 g (1 mmol) of  $CoCl_2 \cdot 6H_2O$  was dissolved in  $10 \text{ cm}^3$  MeOH and warmed up until all of the reactants dissolved. To the resulting brown-purple coloured solution, 0.12 g (1 mmol) of  $NH_4NCS$  was added. The warm mixture was filtrated and set aside for 10 h at room temperature, until the separation of violet, single crystals was completed. The crystalls were filtered and wahsed with EtOH. Yield: 0.16 g (65%).

#### Synthesis of $[Co(HL)_2] \cdot H_2O$ (3)

0.26 g (1 mmol) of PLSC was dissolved in  $H_2O$  ( $10 \text{ cm}^3$ ) by heating followed by the addition of 0.17g (1 mmol) of  $Co(OAc)_2$  into the warm solution. After a couple of hours, separation of purple-coloured crystals suitable for the X-ray analysis was observed. The crystals were filtered and washed with EtOH. Yield: 0.16 g (71%).

## 2.2. Quantitative analysis

Anal. Calcd for :

Complex (1):  $C_9H_{16}CoN_4O_9S$ : C 26.03, H 3.88, N 13.49.  
Found: C 25.87, H 3.65, N13.40.

Complex(2):  $C_{19}H_{30}Co_1N_9O_{10}S_1$ :C45.35, H 4.75, N 19.83. Found: C 45.15, H 4.56,N 19.70.

Complex(3):  $C_{18}H_{26}Co_1N_8O_8$ : C 39.93, H 4.84, N 20.69.  
Found: C 39.60, H 4.64, N 19.90.

## 2.3. Spectral Data for Complex

Complex(1):Selected IR data for  $[Co(PLSC)(SO_4)(H_2O)_2]$  (KBr pellet): 2800, 1680, 1573, 1498, 622.

Complex(2):Selected IR data for  $[Co(PLSC)(PLSC-2H)]NCS \cdot H_2O$  (KBr pellet): 2800,1642.61, 1605, 1507.86, 2054.

Complex(3): Selected IR data for  $[Co(PLSC-H)_2] \cdot H_2O$  (KBr pellet): 2800, 1624.60, 1599, 1501.

## 2.4. Conductometric and Magnetochemical measurements

Complex (1):  $\lambda_M(H_2O)$ :  $183 \text{ S cm}^2 \text{ mol}^{-1}$ .  $\mu_{\text{eff}} = 4.15 \text{ BM}$ .

Complex(2): $\lambda_M(H_2O)$ : $105 \text{ S cm}^2 \text{ mol}^{-1}$ . $\mu_{\text{eff}} =$  (diamagnetic).

Complex (3):  $\lambda_M(H_2O)$ :  $154 \text{ S cm}^2 \text{ mol}^{-1}$ .  $\mu_{\text{eff}} = 4.61 \text{ BM}$ .

## 2.5. Analytical and physical measurement

Magnetic susceptibilities were measured at room temperature using a magnetic susceptibility balance (Johnson Matthey Chemicals Limited, England), Inorganic Chemistry, University of Oxford. Molar conductivities of the freshly prepared  $1 \times 10^{-3}$  M solution were measured on a Jenway 4010 conductivity meter, and IR spectra (KBr disk) were recorded on a Thermo Nicolet (NEXUS 670 FT-IR) instrument. Department of Chemistry, University of Novi Sad. Elemental (C,H,N) analysis of air-dried samples was carried out by standard micromethods in the Center for Instrumental Analysis, Faculty of Chemistry, Belgrade. The X-ray analysis was performed at the University of Oxford Chemical Crystallography Service.

## 2.6. Crystal structure determination

A single crystal of was mounted on a glass fiber in a random orientation. Data collection was performed at temperature 150 K on a computing data collection Nonius Kappa CCD diffractometer using graphite monochromated MoK $\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ). Data collection and cell refinement were carried out using DENZO and COLLECT [30, 31] The structures were solved with SIR-92 and refined using CRYSTALS [32,33]. In general, the hydrogen atoms were visible in the difference map. Therefore, they were positioned geometrically and refined in a separate hydrogen cycle (with soft restraints) before inclusion in the refinement using a riding model. Crystallographic data have been deposited at the Cambridge Crystallographic Data Centre. Copies of the data can be obtained free of charge by writing to the CCDC, 12 Union Road, Cambridge CB2 IEZ, UK; e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk). The CCDC deposition numbers are 795759, 795760 and 795761 for complexes (1), (2) and (3), respectively. Crystal data and details concerning data collection and structure refinement are given in Table 1.

**Table 1.** Crystal data and structure refinement details of complexes

Identification code	(1)	(2)	(3)
Empirical formula	C <sub>9</sub> H <sub>16</sub> Co <sub>1</sub> N <sub>4</sub> O <sub>9</sub> S <sub>1</sub>	C <sub>19</sub> H <sub>30</sub> Co <sub>1</sub> N <sub>9</sub> O <sub>10</sub> S <sub>1</sub>	C <sub>18</sub> H <sub>26</sub> Co <sub>1</sub> N <sub>8</sub> O <sub>8</sub>
Formula weight	415.25	635.50	541.39
Temperature	150K	150K	150K
Wavelength	0.71073 Å	0.71073 Å	0.71073 Å
Crystal system	Monoclinic	Triclinic	Monoclinic
Space group	P 1 21/n 1	P-1	C 1 2/c 1
a (Å)	9.0717(3)	10.5245(5)	16.1908(5)
b (Å)	9.3621(3)	12.1824(6)	13.4392(5)
c (Å)	17.7394(6)	12.3526(8)	12.5129(4)
$\alpha$ (°)	90	107.503(2)	90
$\beta$ (°)	102.2482(14)	95.833(2)	126.6585(18)

$\gamma$ (°)	90	11.542(3)	90
V(Å <sup>3</sup> )	1472.32(8)	1364.20(14)	2184.17(14)
Z	4	2	4

## 3. Results and discussion

### 3.1. Synthesis and characterization of the complexes

All the reported complexes (1 through 3) are crystalline solids, stable in air and at the room temperature, and of characteristic purple color for Co (II, III) compounds with this class of ligands [34,35]. They are all soluble in the water.

The magnetic moment measurements confirmed that all of the reported complexes incorporate Co(II) d<sup>7</sup> (paramagnetic) and Co(III) d<sup>6</sup> (diamagnetic), cation.

The molar conductivity value for complex 1 ( $\lambda_M(\text{H}_2\text{O})$ : 183 S cm<sup>2</sup> mol<sup>-1</sup>) substantially greater than for a 1:1 electrolyte, which can be explained by doubly charged cations and anions formed in solutions. But, the molar conductivity value for complex 2 is in accordance with the molar conductivity value for a 1:1 electrolyte. A little more molar conductivity in complex 3, than of the electrolyte 1: 1 is explained by the existence of the monoanionic form of the ligand PLSC in the crystal structure of the complex 3.

In the IR spectra of all three complexes there exist one peak at about 2800 cm<sup>-1</sup> belonging to  $\nu(\text{NH}^+)$  of pyridine nitrogen [36]. The carbonyl stretching frequency for the coordinated ligand for all three complexes is found to be lower for about 20 cm<sup>-1</sup> in comparison with the free ligand ( $\sim 1680 \text{ cm}^{-1}$ ), which is consistent with the coordination of the ligand through this carbonyl group [21]. So, the tridentate coordination mode of the ligands in each complex is confirmed by characteristic vibrational frequency for carbonyl ( $\nu(\text{C}=\text{O})$  between 1640-1680 cm<sup>-1</sup>) and phenolic ( $\nu(\text{C}-\text{O})$  between 1496-1508 cm<sup>-1</sup>) oxygen atoms as well as for hydrazine nitrogen ( $\nu(\text{C}=\text{N})$  between 1568-1625 cm<sup>-1</sup>) atom in their respective IR spectra. The peak observed at 622 cm<sup>-1</sup> in the IR spectrum of 1 is characteristic of a coordinated SO<sub>4</sub> group while the peak at 2054 cm<sup>-1</sup> found in the IR spectrum of 2 represents the non-coordinated thiocyanide group.

### 3.2. Description of the structure of complexes

Complex 1 is a new complex Co(II) and PLSC ligand. The molecular structure 1 (see Fig. 1) containing a single PLSC, (H<sub>2</sub>L) ligand in its neutral form, such as (H<sub>2</sub>L)CuBr<sub>2</sub>[18], (H<sub>2</sub>L)FeCl<sub>2</sub>(H<sub>2</sub>O)[23], (H<sub>2</sub>L)Ni(NCS)<sub>2</sub>[22], [Ni(PLSC)<sub>2</sub>](NO<sub>3</sub>)<sub>2</sub>[25], and [Cu(PLSC)Cl<sub>2</sub>][Cu(PLSC

)(H<sub>2</sub>O)(SO<sub>4</sub>)<sub>2</sub>], [Cu<sub>2</sub>(PLSC)<sub>2</sub>(NCS)<sub>2</sub>](NCS)<sub>2</sub>, [Cu(PLSC)(NO<sub>3</sub>)<sub>2</sub>(CH<sub>3</sub>OH)](27) are the most predominant complexes characterized thus far.

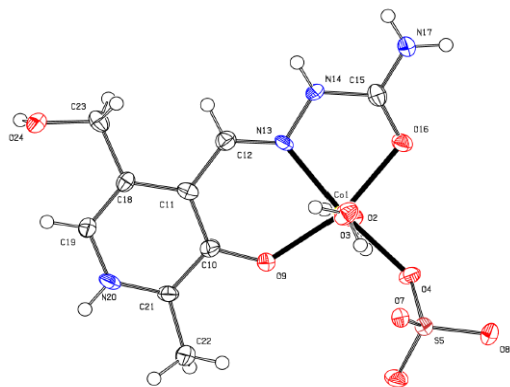


Figure 1. Molecular structure of **1** ([Co(H<sub>2</sub>L)(SO<sub>4</sub>)(H<sub>2</sub>O)<sub>2</sub>]).

However, the ligand PLSC in structure 1, in its neutral but zwitterionic form, coordinates in the usual tridentate fashion creating, together with two molecules of water and one SO<sub>4</sub> group, a slightly disordered octahedral geometry around the central cobalt cation. The structural parameters regarding value of Pyridine angles for **1** confirm that the PLSC ligand is in its neutral form (Table 2).

Table 2. The values of the pyridine and hydrazine angles in the complexes

complex	Form of ligand	Pyridine angles	hydrazine angles
(1)	H <sub>2</sub> L	124.27°	116.90°
(2)	HL	123.15°	108.25°
		123.68°	108.87°
(3)	HL	121.40°	109.99°
		121.46°	

The Co1- O2 and Co1 – O3 bond distances (2.073(3), 2.103(3) Å, respectively) (Table 3) are the similar, as well as Co1-O4 and Co1-N13 (2.087(3), 2.084(3) Å, respectively). The longest bond around the cobalt is the bond with oxygen of the carbonyl group O16 (2.111(3) Å). Angle values are almost in line with the values of the corners of the octahedron (Table 4). Finally, the value of 124. 27° for the C21-N20-C19 (Table 2) bond angle indicates the presence of proton on N20, and confirms the zwitterionic form of the ligand PLSC.

Table 3. The values of the angles around the central atom in the complexes of cobalt

Co(1)-O(2)	2.073(3)	Co(1)-O(2)	1.889(4)	Co(1)-O(2)#1	1.944(3)
Co(1)-O(3)	2.103(3)	Co(1)-N(5)	1.871(5)	Co(1)-N(5)#1	1.908(3)
Co(1)-O(4)	2.087(3)	Co(1)-O(9)	1.870(4)	Co(1)-O(9)#1	1.880(2)
Co(1)-O(9)	2.035(3)	Co(1)-O(18)	1.896(4)	Co(1)-O(2)	1.944(3)
Co(1)-N(13)	2.084(3)	Co(1)-N(21)	1.871(5)	Co(1)-N(5)	1.908(3)
Co(1)-O(16)	2.111(3)	Co(1)-O(29)	1.870(0)	Co(1)-O(9)	1.880(2)

Table 4. Selected angles (°) for cobalt complexes

O(2)-Co(1)-O(3)	171.15(10)	O(2)-Co(1)-N(5)	82.49(19)	O(2)#1-Co(1)-N(5)#1	81.99(11)
O(2)-Co(1)-O(4)	83.63(11)	O(2)-Co(1)-O(9)	176.48(18)	O(2)#1-Co(1)-O(9)#1	175.60(11)
O(3)-Co(1)-O(4)	88.23(10)	N(5)-Co(1)-O(9)	94.79(19)	N(5)#1-Co(1)-O(9)#1	93.74(11)
O(2)-Co(1)-O(9)	89.75(11)	O(2)-Co(1)-O(18)	87.35(17)	O(2)#1-Co(1)-O(2)	87.85(15)
O(3)-Co(1)-O(9)	95.34(11)	N(5)-Co(1)-O(18)	92.66(18)	N(5)#1-Co(1)-O(2)	93.06(10)
O(4)-Co(1)-O(9)	101.75(10)	O(9)-Co(1)-O(18)	90.56(17)	O(9)#1-Co(1)-O(2)	91.30(11)
O(2)-Co(1)-N(13)	99.74(12)	O(2)-Co(1)-N(21)	92.27(19)	O(2)#1-Co(1)-N(5)	93.06(10)
O(3)-Co(1)-N(13)	87.85(12)	N(5)-Co(1)-N(21)	173.25(19)	N(5)#1-Co(1)-N(5)	173.16(16)
O(4)-Co(1)-N(13)	171.46(11)	O(9)-Co(1)-N(21)	90.27(19)	O(9)#1-Co(1)-N(5)	91.10(11)
O(9)-Co(1)-N(13)	86.17(11)	O(18)-Co(1)-N(21)	82.82(18)	O(2)-Co(1)-N(5)	81.99(11)
O(2)-Co(1)-O(16)	87.64(11)	O(2)-Co(1)-O(29)	92.34(17)	O(2)#1-Co(1)-O(9)	91.30(11)
O(3)-Co(1)-O(16)	89.63(11)	N(5)-Co(1)-O(29)	88.71(17)	N(5)#1-Co(1)-O(9)	91.10(11)
O(4)-Co(1)-O(16)	95.13(10)	O(9)-Co(1)-O(29)	89.82(17)	O(9)#1-Co(1)-O(9)	89.86(16)
O(9)-Co(1)-O(16)	162.52(11)	O(18)-Co(1)-O(29)	178.55(18)	O(2)-Co(1)-O(9)	175.60(11)

In other hand, several complexes containing PLSC ligand in its mononionic (e.g. (PLSC-H)PtCl<sub>3</sub> [19], [Ni(PLSC-H)<sub>2</sub>]<sub>2</sub>H<sub>2</sub>O, [Ni(PLSC-H)N<sub>3</sub>]<sub>2</sub>H<sub>2</sub>O [25] and dianionic form (e.g. [Ni(PLSC-2H)(NH<sub>3</sub>)]·1.5H<sub>2</sub>O [22], NH<sub>4</sub>[VO<sub>2</sub>(PLSC-2H)]·H<sub>2</sub>L·2H<sub>2</sub>O [17]). Interesting for this work, so far only a single cobalt II complex incorporating PLSC ligand has been reported ([Co(PLSC)<sub>2</sub>](NO<sub>3</sub>)<sub>2</sub>·2H<sub>2</sub>O) [26]. So, structure cobalt III (see Fig. 2 and Fig. 3), were first synthesized with monoanionic form of ligand PLSC now.

X-ray analysis proved to be an extremely useful tool in identifying if any deprotonation occurred in pyridoxal-semicarbazone type ligands such as PLSC. It is generally established that the deprotonation of the hydrazine nitrogen precedes the deprotonation of the pyridine nitrogen in the formation of the doubly deprotonated form

( $L^{2-}$ ) of the PLSC ligand (example  $NH_4[VO_2(L)] \cdot H_2L \cdot 2H_2O$  [17]).

Deprotonation at the pyridine nitrogen is manifested by decrease in the C-N-C (N is the pyridine nitrogen) angle from 121-125 to 117-119 [17], while hydrazine nitrogen deprotonation is evident by angle reduction. It is generally established that the deprotonation of the hydrazine nitrogen precedes the deprotonation of the pyridine nitrogen in the formation of first the singly ( $HL^-$ ) and then the doubly ( $L^{2-}$ ) deprotonated forms of the PLSC ligand [16].

Thus, complexes **2** and **3** follow the general trend as the monoanionic forms of the ligand are formed by hydrazine nitrogen. The structural parameters for complexes (**2**) and (**3**) confirm that the PLSC ligand is in its monoanionic form (Table 2 and Table 4).

Figures 1 through 3 represent the crystal structures of the corresponding complexes, while Table 3 and 4, besides the bond angles around each cobalt centre, includes the important structural parameters of the bound PLSC ligands.

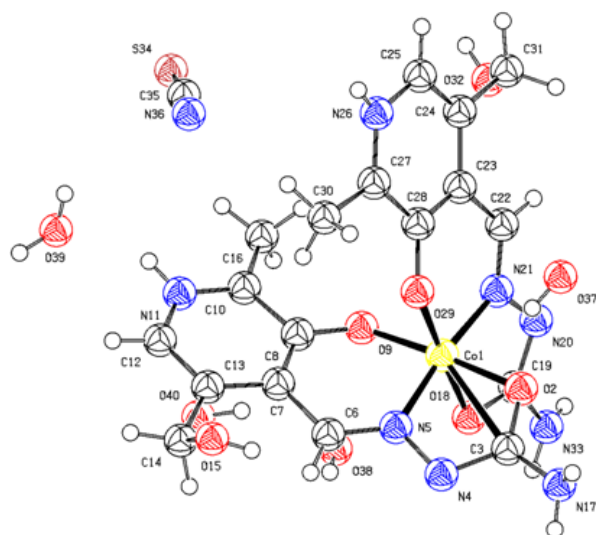


Figure 2. Molecular structure of **2** ( $[Co(HL)_2]NCS \cdot H_2O$ )

Therefore, the values of the pyridine angles (C21-N20-C19) of  $124.3(3)^\circ$  and the values of the hydrazine angles (C15-N14-N13) of  $116.9(3)^\circ$  for structure **1** confirm the presence of a hydrogen atom, resulting in the neutral form of the PLSC ligand. The values of this bond angle for the rest of the synthesized complexes (complex **2**: C27(12)-N26(11)-C25(10) is  $123.6(5)^\circ$  ( $123.7(5)^\circ$ ) and complex **3**: C12(12a)-N11(11a)-C10(10a) is  $121.4(3)^\circ$  ( $121.4^\circ$ ) confirm the presence of a hydrogen atom in pyridine angle also, but the values of the hydrazine angles (complex **2**: N5(21)-N4(20)-C3(19) is  $108.9(4)^\circ$  ( $108.3(4)^\circ$ ) and

(complex **3**: N5(5a)-N4(4a)-C3(3a)) indicate the deprotonation H atom and exist  $HL^-$  (monoanionic form) of the ligand (Table 2).

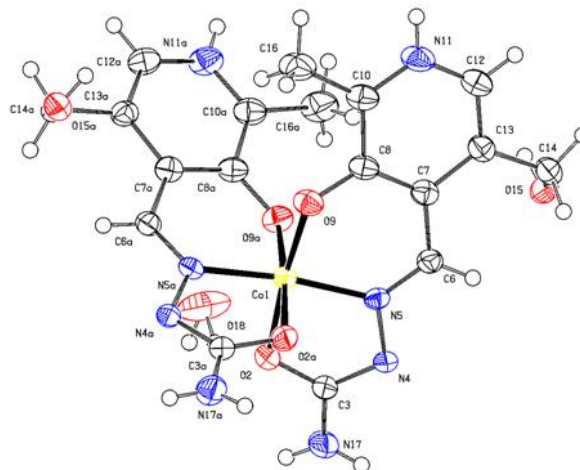


Figure 3 Molecular structure of **3** ( $[Co(HL)_2] \cdot H_2O$ )

Comparison of the bond lengths of the synthesized complexes revealed that in general the longest bond the ligand forms with the cobalt centre is through the carbonyl oxygen (1: O16-Co are  $2.111(3) \text{ \AA}$ ; 2: Co(1)-O(2(18)) is  $1.889(4); 1.896(4) \text{ \AA}$ ; 3: Co(1)-O(2)#1 is  $1.944(3) \text{ \AA}$ ; while the shortest bond is formed through the phenolic oxygen atom in each complex (1: Co(1)-O(9) is  $2.035(3)$ ; 2: Co(1)-O(9(29)) are  $1.870(4)$  and  $1.870(4) \text{ \AA}$ , 3: Co(1)-O(9)#1 is  $1.880(2) \text{ \AA}$ ). Other important bond lengths are summarized in Table 3. The octahedral geometry around each cobalt center is slightly disordered and all-important bond lengths and angles around the cobalt center in each complex are summarized in Table 3.

All the characterized complexes are octahedral, complexes (**2**) and (**3**) is bis(ligand) complexes. The bis-PLSC containing complexes form the octahedral geometry by tridentate ligation (phenolic or carbonyl oxygen and hydrazine nitrogen atoms) of each ligand.

#### 4. Conclusions

The synthesis, characterization and structural analysis of three cobalt complexes incorporating PLSC ligand are described. Even though in each synthetic procedure a Co(II) starting material was used two Co(II) and one Co(III) complexes were isolated. All three complexes are octahedral regardless of the number (one or two) of PLSC ligands a complex incorporates.

## 5. Acknowledgments

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### Appendix and Supplementary Material

CCDC 795759, 795760 and 795761 contains the supplementary crystallographic data for the complex. Copies of this information may be obtained free of charge from CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax:+44-1223-336033; e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk) or www:<http://www.ccdc.cam.ac.uk>)

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