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# Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry

Publication details, including instructions for authors and subscription information: http://www.tandfonline.com/loi/lsrt19

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To cite this article: M. Ali Deveci & Gazi İrez (1996) The Synthesis of Some Imino-Oxime Derivatives and Investigation of Their Ni(II), Co(II), Co(III) and Cu(II) Complexes, Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry, 26:5, 871-881, DOI: <u>10.1080/00945719608004340</u>

To link to this article: http://dx.doi.org/10.1080/00945719608004340

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# THE SYNTHESIS OF SOME IMINO-OXIME DERIVATIVES AND INVESTIGATION OF THEIR Ni(II), Co(II), Co(III) AND Cu(II) COMPLEXES

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# ABSTRACT

Two new ligands and their transition metal complexes were synthesized. These ligands are 1,2-bis(isonitrosophenylketimine)ethane (BPEH<sub>2</sub>) and 1,3bis(isonitrosophenylketimine)propane (BPPH<sub>2</sub>). With these ligands, complexes were synthesized using Ni(II), Co(II), Co(III) and Cu(II) salts with a metal: ligand ratio of 1:1. The structures of the above were determined by <sup>1</sup>H NMR and IR spectroscopy, elemental analyses and magnetic susceptibility.

#### **INTRODUCTION**

We reported earlier derivatives of monoglyoximes, diaminoglyoximes, heterocyclic <u>vic</u>-dioximes, tetraoximes and some of their transition metal complexes<sup>1-8</sup>. Complexes of Co(III) <u>vic</u>-dioximes have been investigated extensively due to their similarity to vitamin  $B_{12}^{9,10}$ . The Co(III) complexes (cobaloximes) readily undergo reduction with NaBH<sub>4</sub> to give blue Co(I) compounds which are capable of binding various alkyl or aryl groups<sup>18,19</sup>. Besides cobaloximes, which are regarded as vitamin  $B_{12}$  model compounds<sup>9-12</sup>, the Schiff

base complexes of cobalt and a number of tetra nitrogen macrocycles also serve as good models<sup>13-15</sup>.

In this article, we report the synthesis of the two new ligands, BPEH<sub>2</sub> and BPPH<sub>2</sub>, by reacting isonitrosoacetophenone with aliphatic diamines and their complexes with Ni(II), Co(II), Co(III) and Cu(II) ions.

# **RESULTS AND DISCUSSION**

The two new imino-oxime derivatives were synthesized by reacting aliphatic diamines with isonitrosoacetophenone. The structures of these ligands as well as that of their metal complexes were identified employing elemental analyses, <sup>1</sup>H NMR, IR spectroscopy, and magnetic susceptibility (Tables I, II and III). The general formula of the synthesized ligands is shown in Fig. 1.

The complexes were prepared by two methods, A and B, which are represented by the following general equations.

Method A

$$M(NO_3)_2 + LH_2 \xrightarrow{EtOH} [MLH] \cdot NO_3 + HNO_3$$
(1)  
M = Ni(II), Co(II), Cu(II) L = BPEH\_2, BPPH\_2

Method B

$$Cu(NO_3)_2 + LH_2 \xrightarrow{EtOH/H_2O} [CuL] + 2 HNO_3$$
(2)

$$M(NO_3)_2 + LH_2 \xrightarrow{\text{EtOH / H}_2O} [MLH] (OH)_n (H_2O)_m + 2 HNO_3 \quad (3)$$
  
M = Ni(II): (n = 1, m = 1); M = Co(III): (n = 2, m = 0)

#### <sup>1</sup><u>H NMR Spectra of Ligands and Complexes</u>

The <sup>1</sup>H NMR spectra of the synthesized ligands (BPEH<sub>2</sub>, BPPH<sub>2</sub>) and some of their metal complexes in DMSO-d<sub>6</sub> were investigated (Table II). The

Compounds	Color	M. p. (dec.)	Yield	Cal	culated.	(Found) % of		
-	_	° C	%	С	Н	N	М	
BPEH <sub>2</sub>	pale-yellow	(79)	60	67.05	5.63	17.39	-	
C <sub>18</sub> H <sub>18</sub> N <sub>4</sub> O <sub>2</sub>				(66.80)	(5.48)	(17.24)	-	
BPPH <sub>2</sub>	white	(115)	55	67.83	6.00	16.66	-	
$C_{19}H_{20}N_4O_2$				(67.68)	(5.87)	(16.42)	-	
BPEHNi NO3ª	brown	(143)	58	48.97	3.88	15.87	13.14	
C <sub>18</sub> H <sub>17</sub> N <sub>5</sub> O <sub>5</sub> Ni				(48.68)	(3.67)	(15.72)	(13.08)	
BPEHCu·NO3ª	dark-brown	(122)	65	48.42	3.84	15.70	14.10	
C <sub>18</sub> H <sub>17</sub> N <sub>5</sub> O <sub>5</sub> Cu				(48.36)	(3.76)	(15.48)	(14.00)	
BPEHCo NO3ª	orange	(142)	55	48.86	3.88	15.84	13.30	
C <sub>18</sub> H <sub>17</sub> N <sub>5</sub> O <sub>5</sub> Co				(48.74)	(3.68)	(15.72)	(13.25)	
Ni(BPEH)OH·H <sub>2</sub> O <sup>b</sup>	greenish-	(208)	68	52.16	4.87	13.53	13.99	
C <sub>18</sub> H <sub>20</sub> N <sub>4</sub> O <sub>4</sub> Ni	brown			(51.98)	(4.72)	(13.48)	(13.78)	
BPECu <sup>b</sup>	brown	(89)	70	56.39	4.21	14.62	16:43	
C <sub>18</sub> H <sub>16</sub> N <sub>4</sub> O <sub>2</sub> Cu				(56.22)	(4.08)	(14.54)	(16.28)	
Co(BPEH)(OH)2 <sup>b</sup>	brick-red	(123)	67	52,16	4.62	13.53	14.20	
C <sub>18</sub> H <sub>19</sub> N <sub>4</sub> O <sub>4</sub> Co				(52.04)	(4.48)	(13.48)	(14.22)	
BPPHNi·NO3 <sup>a</sup>	dark-red	(181)	58	50.10	4.21	15.39	12.73	
C <sub>19</sub> H <sub>19</sub> N <sub>5</sub> O <sub>5</sub> Ni				(49.87)	(4.08)	(15.28)	(12.64)	
BPPHCu·NO3ª	dark-brown	(182)	67	49.51	4.15	15.19	13.70	
C <sub>19</sub> H <sub>19</sub> N <sub>5</sub> O <sub>5</sub> Cu				(49.44)	(4.02)	(15.08)	(13.56)	
BPPHCo·NO3ª	brick-red	(154)	56	49.99	4.20	15.35	12.90	
C <sub>19</sub> H <sub>19</sub> N <sub>5</sub> O <sub>5</sub> Co				(49.86)	(4.12)	(15.28)	(12.82)	
Ni(BPPH)OH·H <sub>2</sub> O <sup>b</sup>	greenish-	(178)	70	53.18	5.17	13.06	13.68	
C <sub>19</sub> H <sub>22</sub> N <sub>4</sub> O <sub>4</sub> Ni	brown			(53.02)	(5.08)	(13.00)	(13.52)	
BPPCu <sup>b</sup>	walnut-	(152)	76	57.42	4.57	14.11	15.85	
C <sub>19</sub> H <sub>18</sub> N <sub>4</sub> O <sub>2</sub> Cu	green			(57.38)	(4.48)	(14.03)	(15.68)	
Co(BPPH)(OH)2 <sup>b</sup>	pale-brown	(175)	74	53.26	4.94	13.08	13.70	
$C_{19}H_{21}N_4O_4C_0$				(53.17)	(4.86)	(12.96)	(13.64)	

Table I. Color, Melting Points, Yields and Elemental Analytical of Results of Ligands and Complexes

a: The complexes were prepared according to Method A

b: The complexes were prepared according to Method B

Compounds	О-Н…О <sup>ь</sup>	<u>О-</u> Н <sup>ь</sup>	H - C = N	Harom	N - CH <sub>2</sub> -	- CH2-
BPEH <sub>2</sub>	-	12,53 (2H s)	8.04 (2H s)	7.70-7.49 (10H m)	2.50 (4H, t, J=4.0 Hz)	-
BPPH <sub>2</sub>	-	11,46 (2H s)	8.22 (2H s)	7.58-7.30 (10H m)	2.45 (4H, t, J=5.0 Hz)	1.96-1.58 (2H m)
BPPHNi NO3	18.34 (1H s)	-	7.26 (2H s)	7.54-7.48 (10H m)	3.57 (4H, t, J=5.2 Hz)	1.94-1.60 (2H m)
BPPHCu NO3	17.58 (1H s)	-	7.20 (2H s)	7.48-7.40 (10H m)	3.43 (4H, t, J=8.0 Hz)	1.85-1.65 (2H m)
BPPCu	-	-	7.18 (2H s)	7.47-7.38 (10H m)	3.42 (4H, t, J=6.0 Hz)	1.86-1.48 (2H m)
Ni(BPPH)OH·H <sub>2</sub> O	18.92 (1H s)	-	8.22 (2H s)	7.92-7.47 (10H m)	2.40 (2H, t, J=8.0 Hz)	1.69-1.46 (2H m)
BPPHCo·NO3	18.24 (1H s)	-	8.16 (2H s)	7.54-7.32 (10H m)	3.40 (4H, t, J= 8.0 Hz)	1.76-1.58 (2H m)
Co(BPPH)(OH)2	18.16 (1H s)	-	8.22 (2H s)	7.47-7.36 (10H m)	3.17 (4H, t, J=6.2 Hz)	1.62-1.48 (2H m)

Table IL <sup>1</sup>H NMR Spectra of Ligands<sup>a</sup> and of some their Complexes<sup>a</sup> in DMSO-d<sub>6</sub> [5 (ppm)]

a: s: singlet; t: triplet; m: multiplet,b: Disappears on D<sub>2</sub>O exchange.

Compounds	Magnetic Moment (B.M.)	0-н…0	0-н	C -H <sub>arom.</sub>	C - H <sub>alip.</sub>	C = N	C = C	N - O
BPEH	-	-	3300	3090	2960	1685-1650	1600	990
BPPH,	-	-	3160	3060	2890	1640-1620	1590	980
BPEHN: NO,	-	3400	•	3030	2945	1680-1640	1590	960
BPEHCu NO <sub>2</sub>	1.78	3440	-	3100	2950	1630-1600	1570	915
BPEHCo NO,	2.34	3500	-	3100	2895	1640-1590	1505	880
NI(BPEH)OH H,O	3.20	3310	-	3080	2920	1665-1630	1500	900
BPECu	1.87	-	-	3080	2935	1660-1620	1590	890
Co(BPEH)(OH),	-	3490	-	3100	2895	1660-1620	1520	900
BPPHNi NO,	-	3430	-	3030	2900	1640-1600	1520	945
BPPHCu NO,	1.73	3410	-	3090	2910	1665-1620	1525	945
BPPHCo NO,	2.42	3450	-	3095	2895	1680-1630	1500	950
Ni(ВРРН)ОН́∙Н,О	3.32	3450	-	3095	2925	1660-1630	1580	920
BPPCu	1.84	-	-	3070	2885	1660-1620	1500	940
Co(BPPH)(OH) <sub>2</sub>	-	3450	-	3100	2945	1640-1610	1500	930

Table III. Magnetic Moments and Characteristic IR Bands of the Ligands and Their Complexes as KBr Pellets (cm<sup>-1</sup>)



Fig. 1. Structure of the Ligands

chemical shift values of the N-OH protons in the ligands are seen as singlets at 12.53 ppm and 11.46 ppm, respectively. These values are in agreement with the chemical shift values of oxime protons<sup>1-8,13,17-20</sup>. The disappearance of these peaks after addition of D<sub>2</sub>O is evidence that these peaks belong to O-H protons. The shift of the C-H protons adjacent to the oxime groups is at 8.00 ppm<sup>1,21-23</sup>. The chemical shift values of H<sub>arom</sub>, N-CH<sub>2</sub> and CH<sub>2</sub> are 7.70-7.30 ppm, 3.54-2.46 ppm and 1.50 ppm, respectively. The CH<sub>2</sub> protons adjacent to the nitrogen atom are shifted downfield as compared to the normal chemical shift values of CH<sub>2</sub> protons<sup>2,3,13,21</sup>. When the <sup>1</sup>H NMR spectra of some of the complexes were examined, the disappearance of the peaks observed at about 11.00 ppm for the ligands and appearance of peaks at about 18.00 ppm for the complexes synthesized in absolute alcohol indicate that intermolecular hydrogen bonds (O-H-O) have formed<sup>16,17,20,24-26</sup> (Fig. 2). Furthermore, these peaks disappeared upon addition of D<sub>2</sub>O. When the <sup>1</sup>H NMR spectra of the complexes isolated in alcohol-water mixture were investigated, chemical shift values of the O-H-O protons appeared around 18.00 ppm in the Co(III) and Ni(II) complexes. But these chemical shift values were not observed in the Cu(II) complexes. This indicates that Ni(II) and Co(III) formed coordination bonds with a nitrogen atom (Fig. 3), whereas Cu(II) formed a bond with an oxygen atom.

# **IR Spectra of Ligands and Complexes**

The IR spectra of ligands and their complexes are given in Table III. When the IR spectral values of the ligands were examined, the OH stretching vibration of the oxime groups appeared<sup>1-8,13,17-20</sup> at 3300 cm<sup>-1</sup> and 3160 cm<sup>-1</sup>. The C=N stretching vibration of oxime and imine groups were observed at 1680 cm<sup>-1</sup> and



Fig. 2. The Square Planar Structure of Ni(II), Co(II) and Cu(II) Complexes Prepared According to Method A



 $R = -CH_2 - CH_2 -$  (BPEHM)  $R = -CH_2 - CH_2 - CH_2 -$  (BPPHM)  $M = Ni(II); A: OH, B: H_2O$ M = Co(III); A: OH, B: OH

Fig. 3. The Tetragonal Structure of Ni(II) and Co(III) Complexes Prepared According to Method B

1620 cm<sup>-1</sup> as two separate bands. This fact indicates the existence of two different C=N groups<sup>15,27,28</sup>. The stretching vibration bands of the N-O group<sup>1-8</sup> were observed at about 980 cm<sup>-1</sup> (Fig. 1).

When the IR spectra of the complexes were investigated, the OH stretching vibration bands were observed at about 3160 cm<sup>-1</sup> and 3300 cm<sup>-1</sup>, whereas these bands were not observed in the complexes prepared by either Method A or Method B. The hydrogen bridge (O-H…O) band was observed at about 3450 cm<sup>-1</sup> for the Ni(II), Co(II) and Cu(II) complexes isolated according to Method B (Fig. 2) and Ni(II) and Co(III) complexes isolated according to Method B (Fig. 3). The band around 3450 cm<sup>-1</sup> on the Cu(II) complexes prepared according to Method B was

not observed<sup>29,30</sup>. This fact indicates that the hydrogen bridge was not formed in the Cu(II) complexes (Fig. 4). The C=N stretching vibration bands of the Ni(II), Co(II) and Cu(II) complexes synthesized according to Method A and Ni(II) and Co(III) complexes synthesized according to Method B were shifted towards lower frequency as compared to C=N stretching vibration bands of the ligands which indicates that these ligands are coordinating with the nitrogen atoms of the oxime and imine groups. The observed broad bands around 3180-3300 cm<sup>-1</sup> of the Ni(II) complexes synthesized according to Method B belong to H<sub>2</sub>O. This indicates that water molecules participated in coordination in the Ni(II) complexes synthesized according to Method B.

### Magnetic Susceptibility of the Complexes

The magnetic susceptibility values of the complexes (Table III) show d<sup>8</sup> configuration for the Ni(II) complexes synthesized according to Method A and they are diamagnetic, while the d<sup>9</sup> Cu(II) complexes and d<sup>7</sup> Co(II) complexes are paramagnetic. The measured values for the Cu(II) and Co(II) complexes are 1.75 B.M. and 2.40 B.M., respectively, which corresponds to a single electron. These values also show that the Ni(II), Co(II) and Cu(II) complexes are of square-planar structure<sup>19,21,31</sup> (Fig. 2).

The d<sup>8</sup> Ni(II) complexes prepared according to Method B are paramagnetic. The measured values for the Ni(II) ion is around 3.30 B.M. which corresponds to two electrons. The magnetic susceptibility values as well as the observation of hydrogen bridges (O-H…O) in the <sup>1</sup>H NMR and IR spectra and H<sub>2</sub>O bands in the IR spectra and also the results of elemental analyses (Table I), indicate that the Ni(II) complexes are of a sp<sup>3</sup>d<sup>2</sup> hybrid tetragonal structure<sup>19,28,31,32</sup> (Fig. 3).

The d<sup>6</sup> complexes of Co(III) ion prepared according to Method B are diamagnetic, which indicates that the Co(II) ion was oxidized to Co(III) under these reaction conditions. The formation of hydrogen bridges (O-H…O) and the diamagnetic structure of the complexes indicate that the geometry is tetragonal<sup>31</sup> (Fig. 3).

The  $d^9$  Cu(II) complexes synthesized according to Method B are paramagnetic. The measured values for Cu(II) ion which is around 1.84 B.M.

(BPECu)



Fig. 4. The Tetrahedral Structure of Cu(II) Complexes Prepared According to Method B

corresponds to one electron. The OH protons and hydrogen bridge (O-H-O) bands were not observed in the <sup>1</sup>H NMR and IR spectra, which indicates that coordination took place via the oxygen atoms of the oxime group rather than nitrogen atoms. The complexes synthesized according to Method B are green, while the color of the Cu(II) complexes synthesized according to Method A is brown. The values of the magnetic susceptibility show that the Cu(II) complexes synthesized according to Method B are of tetrahedral structure<sup>31</sup> (Fig. 4).

### **EXPERIMENTAL**

The <sup>1</sup>H NMR spectra were recorded on a Varian T 100-A spectrometer while a Pye Unicam SP 1025 spectrophotometer was used for the IR spectra of the compounds pressed into KBr pellets. The elemental analyses were performed by the Laboratories of the Scientific and Technical Research Council of Turkey (TUBITAK). The magnetic moments of the complexes were measured according to the Gouy method on a Newport Instruments type D-104 magnet power supply. All reagents were of analytical grade.

The physical properties and spectral characteristics of the synthesized compounds are given in Tables I, II and III.

#### Preparation of the Compounds

Isonitrosoacetophenone was prepared according to published procedures<sup>33</sup>.

#### **IMINO-OXIME COMPLEXES**

**1.2-Bis(isonitrosophenylketimine)ethane (BPEH<sub>2</sub>).** A quantity of 0.02 mol (2.98 g) isonitrosoacetophenone was dissolved in 50 mL diisopropyl ether, then 0.01 mol (0.60 g) ethylenediamine was added dropwise to the mixture with stirring. After 30 minutes, a yellowish crystalline substance was formed. To the reaction mixture was added 0.5 mL absolute alcohol. The reaction mixture was stirred for another 4 hours. The substance formed was filtered, washed with diisopropyl ether and dried in vacuo. The product was soluble in absolute alcohol, DMSO, DMF, dioxane, acetone and chloroform. The yield was 1.932 g.

**1.3-Bis(isonitrosophenylketimine)propane (BPPH<sub>2</sub>).** A quantity of 0.02 mol (2.98 g) isonitrosoacetophenone was dissolved in the minimum amount of absolute alcohol (5 mL). A quantity of 0.01 mol (0.74 g) of 1,3-diaminopropane was added dropwise with stirring. The reaction mixture was stirred for 10 h at room temperature. The precipitated crystals were filtered, washed with alcohol and ether, and dried in vacuo. The synthesized product is soluble in DMSO, DMF and dioxane. The yield was 1.848 g.

The Synthesis of Ni(II), Co(II), [Co(III)] and Cu(II) Complexes of BPEH<sub>2</sub> and BPPH<sub>2</sub>. Method A: A quantity of 0.01 mol ligand (3.224 g BPEH<sub>2</sub> or 3.364 g BPPH<sub>2</sub>) was dissolved in 10 mL absolute ethanol. Then 0.01 mol of Ni(NO<sub>3</sub>)<sub>2</sub>  $6H_2O$ , Co(NO<sub>3</sub>)<sub>2</sub>  $6H_2O$  or Cu(NO<sub>3</sub>)<sub>2</sub>  $3H_2O$  dissolved in 10 mL absolute ethanol was added dropwise with stirring. In the course of the reaction the pH decreased to about 3.0-3.5. The formed dark colored solution was kept in a water bath at 70 °C for 30 min, then cooled to room temperature. The precipitate which formed upon addition of diethyl ether was filtered, washed with diethyl ether and dried in vacuo. The obtained complexes are soluble in water, ethyl alcohol, chloroform, DMSO and DMF.

Method B: A quantity of 0.01 mol ligand (3.224 g BPEH<sub>2</sub> or 3.364 g BPPH<sub>2</sub>) was dissolved in 15 mL hot ethyl alcohol, then 0.01 mol of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O, Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O or Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O dissolved in 15 mL water was added dropwise with stirring. The pH of the reaction mixture which dropped to 3.0-3.5 was adjusted to 5.0-5,5 with 1% NaOH solution. The formed complex was kept on a water bath at 70 °C for 30 min, then filtered, washed with alcohol and ether, and dried in vacuo. Cobalt(II) complexes are oxidized to Co(III) by this method of preparation. The obtained complexes are soluble in DMSO and DMF.

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Received: 4 May 1995 Accepted: 7 February 1996 Referee I: H. Po Referee II: A. O. Adeyemo Downloaded by [University of Waterloo] at 07:59 07 January 2015