

Synthesis and Characterization of 2-Ethoxy Isobutryl Acid Hydrazone and 2-Ethoxy Isobutryl Aceto Hydrazone and Its Metal Complexes with Co(II), Ni(II) and Cu(II)

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Abstract

Complexes of Co(II), Ni(II) and Cu(II) derived from 2-ethoxy isobutryl acid hydrazone (EIAH¹) and 2-ethoxy isobutryl aceto hydrazone (EIAH²) have been synthesized and characterized by elemental analysis, FT-IR, molar conductance, UV – visible spectra, ESR, thermal analysis and magnetic susceptibility. The complexes are coloured, stable in air and exhibit coordination number of 4 or 6. Analytical data showed that all the complexes exhibited 1:2 (metal:ligand) ratio. The changes observed between the FT-IR and UV – visible spectra of the ligands and of the complexes allowed us to establish the coordination mode of the metal in complexes. Presence of coordinated water molecules in Co (II) and Ni (II) complexes is confirmed by TGA studies. The conductivity data show that all these complexes are non electrolytes. The solubility test on the ligands and its metal(II) complexes revealed their solubility in dimethylsulphoxide (DMSO). The ligands and metal complexes show a good activity against the bacteria; *B. Subtilis*, *E.coli* and *S.aureus* and fungi *A.niger*, *A.flavus* and *C.albicans*. The antimicrobial results also indicate that the metal complexes are better antimicrobial agents as compared to the ligands.

Keywords: Synthesis, hydrazine, hydrazone, metal complexes, characterization

Introduction

Study of metal complexes of hydrazide/hydrazone had and has made much progress in recent time ¹. Due to the fact that, hydrazide/hydrazone complexes were known for their semiconducting catalytic properties, waste water treatment for metal recovery, in protective coating, in solvent extraction, and anti fungal properties ^{1, 2}. Co-ordination of metal hydrazide/hydrazone complexes are mostly derived from bi-chelating ligands in which metal ions and chelating agents are arrayed alternatively.

In fact, the rapidly developing field of bioinorganic chemistry is centred on the presence of metal complexes in the biological systems ³. Moreover, the study of co-ordination compounds has enabled the chemists to make significant progress in refining the concept of chemical bonding and also to explain the influence, that bonding has on the various properties of the compounds.

A transition metal complexes which usually contain nitrogen, sulphur or oxygen as ligands atoms are becoming increasingly important because these ligands can bind with different metal centres involving various co-ordination sites and allow successful synthesis of metallic complexes with interesting stereochemistry. Heterocyclic compounds are widely distributed in the nature and essential to many biochemical, analytical and industrial processes. Compound containing these heterocyclic have important properties in the field of material science and biological systems ⁴.

Hydrazones compounds are not only intermediates but they are also very effective organic compounds. They can be used as intermediates to synthesize coupling products by using the active hydrogen of – CONHN=CH– azomethine group. Various effective compounds for example: iproniazide and isocarboxazide are synthesized by reduction of hydrazide/hydrazones ⁵. The primary aims of this work was to synthesis new metal complexes of Co(II), Ni(II) and Cu(II) of hydrazide/hydrazones that was obtained from 2 – ethoxy isobutryl acid hydride and 2 – ethoxy isobutryl aceto hydrazone. These complexes could be used in waste water treatment to minimised water born diseases especially for the developing countries.

Materials and Methods

Reagents / solvents and Instrumentation: CoSO₄.7H₂O, CuSO₄.5H₂O, and NiSO₄.7H₂O, were of analytical grade reagents and obtained from commercial sources (Aldrich Chemical Co. and BDH) and used without further purification. Elemental analyses were performed on an Elemental Vario EL III Carlo Erba1108 analyzer. FT–IR spectra were recorded in KBr medium on a Perkin Elmer RX1 spectrophotometer in wave number 4000 – 400 cm⁻¹. Electronic spectra (in DMSO) were recorded using perkin Elmer Lambda – 2B – spectrophotometer. ESR spectra were recorded on a Varian E – 112 spectrometer using TCNE as the internal standard. Molar conductance measurements were conducted using 10⁻³ M solutions of the complexes in DMSO on Elico – CM 82 Conductivity Bridge at room temperature. Melting point /decomposition temperatures of the ligands and complexes were determined using a Gallenkamp melting point apparatus. The magnetic susceptibilities measurement were taken on a Gouy balance at room temperature using mercuric tetrathiocyanatocobaltate(II) as

the calibrant.

Synthesis of the hydrazide: The hydrazide was prepared according to the method described in the literature⁶. The hydrazide [EIAH¹] was synthesized by adding ethanolic solution of 2-ethoxy ethyl isobutyrate with ethanolic solution of hydrazine hydrate in equimolar ratio. The reaction mixture was then refluxed on water bath for about 6 hours. The condensation product was filtered, thoroughly washed with ethanol and ether, re-crystallised and dry in a vacuum desiccator for 3 days, The purity of the synthesised compound was monitored by TLC using silica gel G (% yield= 74.63).

Synthesis of the hydrazone: The hydrazones [EIAH²] were prepared via the acid hydrazide. The hydrazides were refluxed with excess acetone for 4 hours on water bath and allowed to cool; the condensation product gave the hydrazone. The hydrazones were filtered, re-crystallised from ethanol and ether, dried over calcium chloride in a vacuum desiccator. The purity of the synthesised compound was monitored by TLC using silica gel G (% yield= 84.20).

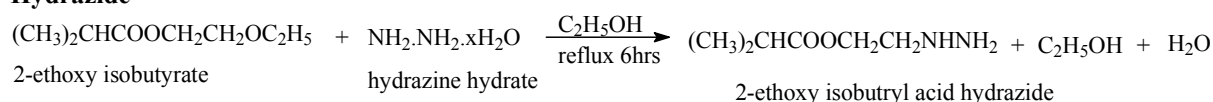
Preparation of metal complexes: The metal complexes have been prepared by mixing the solution of CoSO₄.7H₂O, CuSO₄.5H₂O, and NiSO₄.7H₂O, to the ethanolic solution of the hydrazide [EIAH¹] and hydrazone [EIAH²] in 1:2 molar ratios. The resulting mixture was then refluxed on water bath for about 7 hours. A coloured product appeared on standing and cooling the above solution. The complexes were filtered, washed with ether and dried under reduced pressure over anhydrous CaCl₂ in a desiccator. They were further dried in an electric oven at 60⁰C⁷.

Antibacterial activities: Antibacterial activity of hydrazide/hydrazone (ligands) and its metal complexes were studied against gram – positive bacteria (*Bacillus subtilis* and *Staphylococcus aureus*) and gram-negative bacteria (*E.coli*) at a concentration of 50µg/mL by nutrient agar disk method. Dimethyl sulfoxide (DMSO) system was used as control in this method and the area of inhibition of zone measured in mm. The antifungal activities of the compounds were also tested by the well diffusion method against the fungi: *A. Niger*, *A. flavus* and *C. albicans*, on potato dextrose agar as the medium and Griseofulvin as control. The stock solution was prepared by dissolving the compound in DMSO. In a typical procedure, as well was made on a agar medium inoculated with the fungi. The well was filled with the test solution using a micro-pipette and the plate was incubated 72 hours at 30⁰C. During this period, the test solution diffused and growth of the inoculated fungi was affected. The inhibition zone was developed, at which the concentration was noted⁸.

Results and Discussion

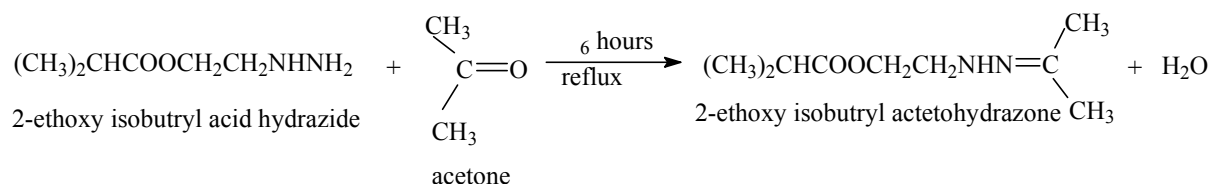
The preparation of the hydrazide and hydrazone as ligands from the ester and hydrazine hydrate were represented by the following equations below:

Hydrazide



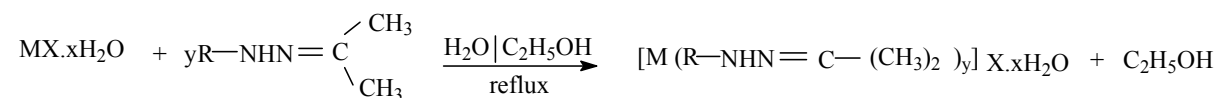
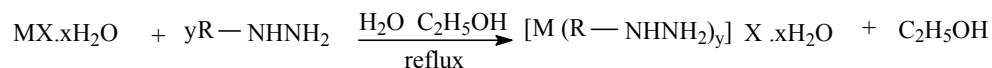
.....1

Hydrazone



.....2

The reaction of the metal (II) salts and the hydrazide/ hydrazone from equations 1 and 2 above, to form metal complexes can be represented in equation 3 as follow:



Where M = Cu, Ni, Co, x = 5/7, y = 2, X = SO₄

.....3

All the metal complexes are coloured, solid and stable towards air or moisture at room temperature. They decompose on heating at high temperature, more or less soluble in common organic solvents. Analytical data of the compounds, together with their physical properties are consistent with proposed molecular formula given in **Table 1**. All the metal complexes have 1:2 (metal:ligand) stoichiometry. The molar conductance in DMSO of the complexes are 23.4, 33.6 and 48.1 Scm² mol⁻¹ respectively, for Co(II), Ni(II) and Cu(II) complexes of (EIAH¹). Similarly, the molar conductance for Co(II), Ni(II) and Cu(II) complexes of (EIAH²) are 32.8, 21.2 and 23.5 Scm² mol⁻¹ respectively. These indicate that the complexes are non – electrolytic.

Table 1: Analytical and physical data of Ligands and metal complexes

Molecular formula/ Mol. Wgt(Colour)	Yield (%)	Elemental Analysis Found/Calculated (%)				*Λ _m mol ⁻¹	M.Pt/Dec °C
		C	H	N	M		
C ₆ H ₁₄ N ₂ O ₂ [EIAH ¹] 146 (Off White)	75	49.32 (49.00)	9.67 (10.01)	19.19 (18.79)	-	-	201
[Cu (C ₆ H ₁₄ N ₂ O ₂) ₂]SO ₄ 452 (Black)	66	31.89 (31.85)	6.20 (5.97)	12.40 (12.10)	14.06 (14.13)	23.4	220
[Ni (C ₆ H ₁₄ N ₂ O ₂) ₂]SO ₄ ·2H ₂ O 483 (Green)	82	29.84 (29.83)	5.84 (5.81)	11.60 (11.63)	12.15 (12.10)	33.6	270
[Co (C ₆ H ₁₄ N ₂ O ₂) ₂] SO ₄ · H ₂ O 401 (Bitter almond)	87	35.94 (35.59)	7.04 (6.99)	13.97 (13.97)	15.96 (15.66)	48.1	210
C ₉ H ₁₈ N ₂ O ₂ [EIAH ²] 186 (Off White)	84	58.10 (58.89)	9.67 (9.25)	15.05 (15.00)	-	-	230
[Cu (C ₉ H ₁₈ N ₂ O ₂) ₂] SO ₄ 532 (Black)	74	20.32 (19.97)	3.41 (3.00)	5.27 (5.22)	11.94 (11.67)	32.8	220
[Ni (C ₉ H ₁₈ N ₂ O ₂) ₂] SO ₄ ·H ₂ O 545 (Green)	65	19.83 (20.05)	3.30 (3.23)	5.14 (5.65)	10.77 (10.70)	21.2	270
[Co (C ₉ H ₁₈ N ₂ O ₂) ₂]SO ₄ ·2H ₂ O 563 (Black)	87	19.20 (19.20)	3.22 (3.21)	4.98 (4.67)	10.47 (10.45)	23.5	230

IR spectra: The data of the IR spectra of the ligands and its metal complexes are listed in **Table 2**. The IR spectra of the complexes were compared with those of the free ligands in order to determine the involvement of coordination sites in chelation. Characteristic peaks in the spectra of the ligands and complexes were considered and compared. The IR spectra of (EIAH¹) and (EIAH²) exhibit a strong band at 1630 cm⁻¹ due to ν(C=N) azomethine group. This band shifts to lower energy region by 11 – 30 cm⁻¹ in the complexes. It suggests bonding through azomethine nitrogen⁹. The coordination of nitrogen to the metal atom would be expected to reduce the electron density on the azomethine link and thus cause a shift in the C=N band. A strong band was observed at 1663, 1669 cm⁻¹ assignable to (ν(C=O/C-O)) of carbonyl group of the ligands. The band position of (ν(C=O/ C-O)) has shifted to lower side by 18–28 cm⁻¹ in the complex on coordination of metal ions with oxygen of ethoxy group. The appearances of broad band around 3313–3363 cm⁻¹, in the spectra of complexes have been assigned to associated water molecule. In all complexes new bands at 490 ± 8 cm⁻¹ and 404 ± 2 cm⁻¹ are due to the formation of ν(M-N) and ν(M-O) bands respectively. The appearance of broad band at 3368 – 3397 cm⁻¹ in complexes have been assigned to ν(OH) water molecules⁶. In the FT-IR spectra the band due to N – H stretching in the free ligands occurs in the 3175 – 3084 cm⁻¹ region and remains unaffected after complexation. This precludes the possibility of coordination through the hydrazine nitrogen atom. The small shift to higher frequency of the band due to ν(N-N) can be taken as additional evidence of the participation of the azomethine group in bonding. The spectra of the complexes show a very sharp non-ligand band around 770 cm⁻¹ which are due to the ν₃ and ν₂ vibrational modes of uncoordinated sulphate group, since the stretching vibrations of the sulphate ion are usually observed as a sharp singlet around 1100 cm⁻¹ and a medium band around 680 cm⁻¹. The sulphate ions are therefore probably in the outer coordination sphere for the complexes⁷.

Table 2. IR bands of ligands and their complexes

Compounds	$\nu(\text{C}=\text{N})$	$\nu(\text{N}-\text{H})$	$\nu(\text{C}-\text{O}/\text{C}=\text{O})$	$\nu(\text{N}-\text{N})$	$\nu(\text{M}-\text{N})$	$\nu(\text{M}-\text{O})$	$\nu(\text{H}_2\text{O})$	$\nu(\text{SO}_4)$
[EIAH ¹]	1630	3084	1669	1003	-	-	-	-
[Cu(EIAH ¹) ₂]SO ₄	1603	3100	1641	1024	486	402	3313	750
[Ni(EIAH ¹) ₂]SO ₄ .2H ₂ O	1608	3100	1651	1055	490	408	3340	700
[Co (EIAH ¹) ₂] SO ₄ .H ₂ O	1610	3170	1649	1033	488	410	3360	650
[EIAH ²]	1626	3080	1663	1003	-	-	-	-
[Cu(EIAH ²) ₂] SO ₄	1600	3175	1635	1020	488	410	3365	770
[Ni(EIAH ²) ₂] SO ₄ .H ₂ O	1598	3150	1630	1030	485	408	3350	720
[Co(EIAH ²) ₂]SO ₄ .2H ₂ O	1605	3155	1640	1025	482	406	3345	710

Electronic spectra and magnetic moment: The electronic spectral data of the metal complexes in DMSO solution are given in **Table 3**. The nature of the ligand field around the metal ion has been deduced from the electronic spectra. The electronic spectrum of Co(II) complex of (EIAH¹) shows two bands of appreciable intensity at 15503 cm⁻¹ and 20125 cm⁻¹ which have tentatively been assigned to ⁴A₂→⁴T₁(F)(v₂) and ⁴A₂→⁴T₁(P)(v₃) transitions. The magnetic moment is 4.35 B.M. Thus the tetrahedral geometry has been suggested for this complex. The electronic spectrum of Ni(II) complex of (EIAH¹), shows three bands at 11070 cm⁻¹, 18816 cm⁻¹ and 24125 cm⁻¹ corresponding to transition ³A_{2g}(F)→³T_{2g}(F) (v₁), ³A_{2g}(F)→³T_{1g}(F) (v₂) ³A_{2g}(F)→³T_{2g}(P) (v₃) respectively. The magnetic moment is 3.19 B.M. These findings are in favour of an octahedral geometry for the Ni(II) complex. For Cu(II) complex of (EIAH¹), a single broad band at 13320 cm⁻¹ has been observed, this attributes to ²E_g→²T_{2g} transition. Its magnetic moment is 1.85 B.M. Thus the octahedral geometry has been suggested for complex. The electronic spectrum of Co(II) complex of (EIAH²) shows two bands of appreciable intensity at 12360 cm⁻¹ and 19806 cm⁻¹ which have tentatively been assigned to ⁴A₂→⁴T₁(F) (v₂) and ⁴A₂→⁴T₁(P) (v₃) transitions. The magnetic moment is 4.67 B.M. Thus, the tetrahedral geometry has been suggested for this complex. The electronic spectrum of Ni(II) complex of (EIAH²) exhibits three bands at 11220 cm⁻¹, 20212 cm⁻¹ and 23645 cm⁻¹ which are assignable to ³A_{2g}(F)→³T_{2g}(F) (v₁), ³A_{2g}(F)→³T_{1g}(F) (v₂) and ³A_{2g}(F)→³T_{1g}(P) (v₃) transition respectively¹⁰. The magnetic moment is 3.14 B.M. These findings are in favour of an octahedral geometry for the Ni(II) complex. In the Cu(II) complex of (EIAH²) a single broad band at 16750 cm⁻¹ has been observed, this attributes to 2E_g→2T_{2g} transition. Its magnetic moment is 1.94 B.M. Thus the octahedral geometry has been suggested for this complex^{1, 6, 7}.

Table 3: Electronic spectral/Magnetic moment data for the complexes

Molecular formular	λcm^{-1}	Assignment	Geometry	M.M (B.M)
[Cu (C ₆ H ₁₄ N ₂ O ₂) ₂]SO ₄	13320	² E _g → ² T _{2g}	Tetrahedral	1.85
[Ni (C ₆ H ₁₄ N ₂ O ₂) ₂]SO ₄ .2H ₂ O	11070	³ A _{2g} (F)→ ³ T _{2g} (F)	Octahedral	3.19
	18816	³ A _{2g} (F)→ ³ T _{1g} (F)		
	24125	² g(F)→ ³ T _{2g} (P)		
[Co (C ₆ H ₁₄ N ₂ O ₂) ₂]SO ₄ .H ₂ O	15503	⁴ A ₂ → ⁴ T ₁ (F)	Octahedral	4.35
	20125	⁴ A ₂ → ⁴ T ₁ (P)		
[Cu (C ₉ H ₁₈ N ₂ O ₂) ₂] SO ₄	16750	² E _g → ² T _{2g}	Tetrahedral	1.94
[Ni (C ₉ H ₁₈ N ₂ O ₂) ₂] SO ₄ .H ₂ O	11220	³ A _{2g} (F)→ ³ T _{2g} (F)	Octahedral	3.14
	20212	³ A _{2g} (F)→ ³ T _{1g} (F)		
	23645	³ A _{2g} (F)→ ³ T _{1g} (P)		
[Co (C ₉ H ₁₈ N ₂ O ₂) ₂]SO ₄ .2H ₂ O	12360	⁴ A ₂ → ⁴ T ₁ (F)	Octahedral	4.67
	19806	⁴ A ₂ → ⁴ T ₁ (P)		

Thermal analysis: The thermogram of Ni (II) and co (II) complexes shows the coordination of two moles of hydrated water where as there is absence of coordination of such hydrated water molecule in case of Cu (II) complexes. Hence from TGA, it is clear that the complex under study contains two water molecules which are coordinated to central metal ion¹¹.

Electron spin resonance: The ESR spectra of Cu (II) complexes in the polycrystalline state shows two peaks, one of intense absorption at high field and the other of less intensity at low field. From these spectra the values of g_{\parallel} and g_{\perp} have been calculated by Kneubehls method¹². The observed g-values point to the presence of the unpaired electron in the dx²-y² orbital with $g_{\parallel} > g_{\perp}$ characteristic of octahedral geometry. The g_{\parallel} obtained for the Cu (II) complexes is less than 2.3 indicating covalent character of the metal-ligand bond¹². The axial symmetry parameter (G) for the complexes is found to be greater than 4. This shows absence of interaction between copper centres in the solid state^{12, 13}.

Antimicrobial activities: The bactericidal and fungicidal investigation data of the compounds are summarized in **Table 4**. The results of the investigations account for the antipathogenic behaviour of the compounds and this

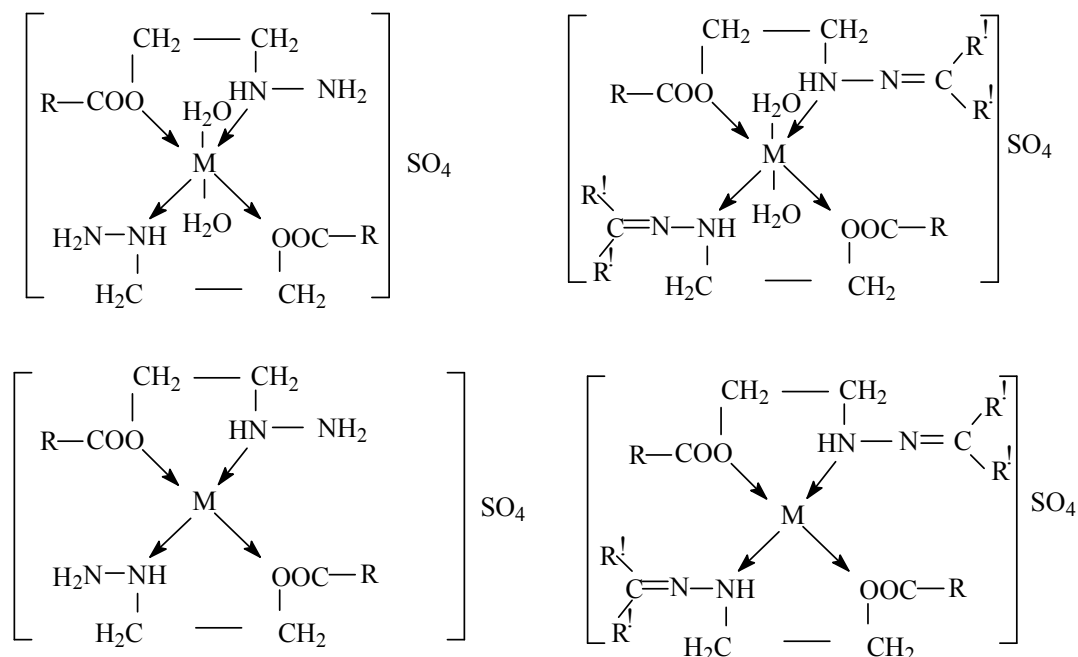
efficacy is positively modified on complexation. Overtone's Concept and Chelation Theory explain well this drug action¹⁴.

Table 4. In-vitro antibacterial activity of compounds and their inhibition zone (%).

Compounds	Antibacterial activity			Antifungal activity		
	<i>B. subtilis</i>	<i>E. coli</i>	<i>S. aureus</i>	<i>A. niger</i>	<i>A. flavus</i>	<i>C. albicans</i>
[EIAH ¹]	10	21	14	10	15	18
[Cu(EIAH ¹) ₂]SO ₄	23	34	22	20	22	32
[Ni(EIAH ¹) ₂]SO ₄ .2H ₂ O	25	33	20	21	20	31
[Co (EIAH ¹) ₂] SO ₄ .H ₂ O	22	30	22	20	22	31
[EIAH ²]	15	21	12	15	14	22
[Cu(EIAH ²) ₂] SO ₄	22	35	19	18	20	34
[Ni(EIAH ²) ₂]SO ₄ .2H ₂ O	20	35	20	19	18	33
[Co(EIAH ²) ₂]SO ₄ .H ₂ O	24	36	21	20	17	35
Streptomycin	28	37	26	-	-	-
Griseofulvin	-	-	-	22	20	-

Conclusion

In the present research studies, we were able to successful synthesised three newly complexes {Co (II), Ni (II) and Cu (II)} and they were characterized by various physicochemical and spectral analyses. The synthesized ligands bind with metal ions in a bi-dentate manner, with ON donor sites of the ethoxy-O and azomethine-N. The antimicrobial data show that the metal complexes to be more biological active compared to the parent ligands against all phatogenic species. The compounds also inhibit the growth fungi bacteria to a greater extent as the concentration increased. TGA analysis showed that the complexes Co(II) and Ni(II) contains two water molecules which are coordinated to the central metal ion. From all the available data, the probable structures for the complexes have been proposed as shown in **Scheme 1**.



where R = (CH₃)₂CH, M = Co(II) and Ni(II) for octahedral geometry

R' = CH₃, M = Cu(II) for tetrahedral geometry

Scheme I: Proposed structures of Co (II), Ni (II) for octahedral geometry and Cu (II) for tetrahedral geometry.

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