

Coenzyme B₁₂ Model Studies: Synthesis and Characterization of Thiocyanato Bridged Binuclear Cobaloximes

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Abstract: The $\text{RCo}(\text{DH})_2\text{NCSCo}(\text{DH})_2\text{L}$ has been synthesized from the corresponding monomeric $[\text{CH}_3\text{Co}(\text{DH})_2\text{L}]$ and $[\text{SCNCo}(\text{DH})_2\text{L}]$ where DH = Dimethyl glyoximate ion, L = (N or O or S) donor ligand. Studies by infrared, Electronic and ¹HNMR spectroscopy indicates that the binuclear Cobaloximes contains a –SCN– linkage, where one Cobalt (III) is coordinated to alkyl (CH_3) group and N of SCN, the other Cobalt (III) to donor ligand (L) and S of SCN bridge. Evidence for formation of thiocyanato ligand bridges was obtained from infrared data which show 20 - 45 cm^{-1} increase in $\nu\text{C}\equiv\text{N}$ upon formation of the binuclear Cobaloxime from the corresponding terminal thiocyanato Cobaloxime, $[\text{SCNCo}(\text{DH})_2(\text{L})]$. The importance of binuclear complexes is one can study properties of the whole molecule in addition to the properties related to each metal
Key words: Binuclear Cobaloximes, Thiocyanato and spectroscopic characterization.

Introduction

Study on the mononuclear transition metal complexes in perspective of their interesting electrochemical, photophysical and photochemical studies [1]. A suitable choice of the mononuclear building block, the bridging ligand and an appropriate design of the structure of the binuclear complex can in fact allow the elucidation of the structural properties of the complex [2]. Therefore numerous bimetallic complexes have been reported in the literature. In recent years, much attention has been devoted to complexes with pseudohalogen bridges CN^- , NCS^- , because of their interesting magneto structural properties [3].

Cobaloximes (Models for Coenzyme B₁₂) [4,5] have become the center of increasing chemical interest, since the most important property of cobaloximes is the ability to form very stable Co-C bonds. Coordination compounds containing O & S donor ligands and their complexes are of considerable importance perhaps due to their antibacterial activity [6 -8]. In all the reactions it is believed that the homolysis [9] of Co-C bond may play a significant role in the kinetic and thermodynamic stability of this bond and also be influenced by the nature of the ligand in the trans position, steric crowding and distortion of the *trans* ligand may play a crucial role in the activation of coenzyme B₁₂.

The synthesis, characterization, and X-ray structure of binuclear complex, $[\text{CH}_3\text{Co}(\text{DH})[\text{DB}(\text{OCH}_3)_3(3\text{-Py})]_2; \text{trans}-[(\text{L})\text{Co}(\text{DH})_2\text{XCo}(\text{DH})_2\text{X}]$ where $\text{L} = \text{SbPh}_3, \text{AsPh}_3, \text{PPh}_3, \text{SbBu}_3$; $\text{X} = \text{SeCN}^-, \text{SCN}^-$; $\text{Z} = \text{Cl}^-, \text{Br}^-$), X in a variety of solvents (DH = monoanion of dimethylglyoxime) [10, 11]. Several of the terminal SCN⁻ groups in the mononuclear Cobaloximes were N-bound. Few or none of the binuclear Cobaloximes could be produced from N-bound complexes, regardless of solvent dielectric constant. These results are in accordance with the dissociative mechanism proposed for the formation of the binuclear Cobaloximes.

Burkhardt[12] synthesized complexes of the type $\text{trans-[Co(dmgH)}_2\text{(L)SeCN]}$ and $\text{trans-[(L)Co(dmgH)}_2\text{-SeCN-Co(dmgH)}_2\text{SeCN]}$ in a variety of solvents and possible mechanism for the formation of the binuclear complexes was presented, [$\text{dmgH}_2 = \text{dimethylglyoxime}$ and $\text{L}=4\text{-acetylpyridine, PPh}_3, \text{PBu}_3, \text{PMePh}_2, \text{PBuPh}_2, \text{P(OPh)}_3, \text{P(OBu)}_3, \text{P(OPr-iso)}_3, \text{P(OMe)}_3$]. The entire terminal SeCN^- groups exhibit coordination via the Se atom, regardless of the nature of L, both in the solid state and in solution. These results demonstrate symbiotic electronic control of the selenocyanate's bonding mode by the other ligands present in the coordination sphere, and are consistent with the considerable π -donor capability of the Se-bound selenocyanate, as reflected in its low position in the spectrochemical series.

The ligand-bridged complexes are of interest in view of their role as reaction intermediates in inner-sphere electron-transfer process [2] and this synthetic strategy was often utilized to provide a general route to synthesize cationic dinuclear cyano-bridged complexes [13]. It is therefore, interesting and useful to study the thiocyanato bridged binuclear inorganic Cobaloximes of the type $[\text{CH}_3\text{Co(DH)}_2\text{SCNCo(DH)}_2\text{(L)}]$ where (L=urea, formamide, acetamide, thiocetamide, semicarbazide, thiosemicarbazide, Alanine, alanine methyl ester, pyrazole and dimethyl pyrazole) and that should be useful to compare with the corresponding mononuclear Cobaloximes $[\text{SCNCo(DH)}_2\text{(L)}]$.

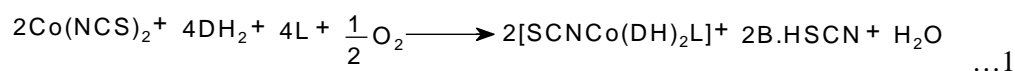
Experimental

$\text{Co(NO}_3)_2 \cdot 6\text{H}_2\text{O}$, KSCN and CH_3Br were purchased from Aldrich Chemicals. All other Chemicals used were of analytical grade and were used without further purification unless otherwise noted. The complexes of the type $\text{SCN Co(DH)}_2\text{(L)}$ and $\text{CH}_3\text{Co(DH)}_2\text{NCSCo(DH)}_2\text{L}$ were prepared by using the Following Procedures.

Preparation of $\text{K[NCSCO(DH)}_2\text{SCN]}$

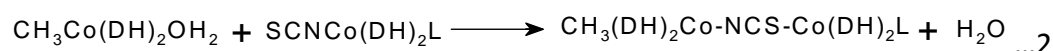
The synthetic route used in this work for the synthesis of thiocyanato bridged binuclear Cobaloximes takes the advantage of the lability of the coordinated water in the alkyl(aqua)cobaloxime [14] which easily allows for the substitution by the nitrogen of coordinated thiocyanate of the thiocyanato Cobaloxime as shown in equation 2. Alkyl(aqua)Cobaloximes were prepared by the procedure of Brown et al [15]. To prepare thiocyanato bridged binuclear Cobaloximes of the type $\text{RCo(DH)}_2\text{SCNCo(DH)}_2\text{L}$, first the monoacido Cobaloximes of the type $\text{SCNCo(DH)}_2\text{L}$ (L=ALA, AME, PY, DMPY, FA, AC, U, SC, TAC, TU & TSC) were synthesized by bubbling air (direct air oxidation method) [16] for 2 to 6 hrs through a methanolic solution of Cobalt(II)thiocyanate, $[\text{Co(NCS)}_2]$ (The thiocyanate was synthesized by metathetic reaction of hydrated $\text{Co(NO}_3)_2$ & KCNS in methanolic medium. The precipitated KNO_3 was filtered off and the solution is cobalt (II) thiocyanate) DMG and the ligand taken in 1:2:2 proportions. This solution was allowed to stand and resulting crystalline solid was washed with methanol and ether, finally dried *in vacuo*.

The reaction presumably proceeds in the following manner



Preparation of Thiocyanato Bridged Bicoaloxime

The thiocyanato bridged Binuclear Cobaloxime was synthesized as follows: 2×10^{-4} moles of $\text{RCo(DH)}_2\text{OH}_2$ (where $\text{R}=\text{CH}_3$) was dissolved in minimum amount of chloroform at 40°C to give an orange solution, to this an equimolar concentration of the complex $\text{SCNCo(DH)}_2\text{L}$ dissolved separately in minimum amount of chloroform at 40°C is added. The two solutions were mixed and stirred constantly at $40\text{-}50^\circ\text{C}$ for 1 hour. The solvent was removed under reduced pressure to give yellow powder, which was washed with water, 95% methanol, ether and dried *in vacuo*.



Results and Discussion

The Infra-Red spectra of $\text{SCNCo(DH)}_2\text{L}$, shows main bands due to the coordinated dimethyl glyoxime, the base ligand and the ambidentate thiocyanate ligand. The prominent bands IR data are given in **table.1**. The ambidentate SCN group may coordinate to Cobalt (III) through N or the S or both ($\text{M-NCS-M}'$). In general class a or hard metal acids are expected to coordinate through the N-end of the SCN group and form Cobalt (III) – N bond [17]; whereas class b or soft metals bond through the S-end of the SCN group and form Cobalt (III)–S

bond [18]. However, others factors such as the oxidation state of metal ion, nature of other ligands present in the complex and steric considerations will influence the mode of coordination. Cobalt (III) is an example of a hard acid [19] should attach through the N-end of the SCN group. But the dimethylglyoxime, a soft ligand, modifies the typical class a or hard Cobalt (III) centre to class b or soft which is often observed in $\text{Co}(\text{DH})_2^+$ [20]. The thiocyanate group is usually bonded to Cobalt (III) centre in Cobaloximes through the 'S' end [19]. The $\nu(\text{C}=\text{N})$ in these complexes was usually obtained as a very intense and sharp peak at 2110-2140 cm^{-1} indicating the coordination of the thiocyanate group.

Caulton et al [21, 22] argued that the potential π donor ligand X [$\text{Co}(\text{Me}_2\text{en})(\text{B})$] (X), where X = SCN, SeCN] can confer stability by forming not only M - X σ bond but also an X \rightarrow M π bond via the lone pairs on X.

Table.1: IR Data.[§] of $\text{SCNCo}(\text{DH})_2\text{L}^*$.

S. No	Complex	$\nu(\text{Co-N})$	DH			SCN		Ligand		
			N (CH_3)	$\nu(\text{C}=\text{N})$	N (NO)	$\nu(\text{CS})$	$\nu(\text{CN})$	$\nu(\text{C}=\text{S})$	$\nu(\text{C}=\text{O})$	$\nu(\text{N-H})$
1	$\text{SCNCo}(\text{DH})_2\text{U}$	511.9	1364.2	557.5	1090.0	738.4	2108.1	-	1695.2	3205.9
			1436.0		1229.6					
2	$\text{SCNCo}(\text{DH})_2\text{FA}$	512.9	1324.2	1560.6	1091.2	739.8	2108.3	-	1631.1	3201.3
			1410.9		1232.8					
3	$\text{SCNCo}(\text{DH})_2\text{TU}$	514.2	1396.4	1559.8	1089.2	740.6	2102.3	708.9	-	3326.2
			1468.1		1235.4					
4	$\text{SCNCo}(\text{DH})_2\text{AC}$	513.8	1364.0	1558.3	1114.4	749.0	2108.0	-	1684.1	3206.6
			1436.7		1232.5					
5	$\text{SCNCo}(\text{DH})_2\text{TAC}$	514.2	1364.2	1558.0	1088.6	742.3	2107.6	708.9	-	3206.5
			1436.1		1232.3					
6	$\text{SCNCo}(\text{DH})_2\text{TSC}$	514.0	1364.2	1560.1	1092.1	745.3	2109.5	709.4	-	3204.3
			1438.3		1237.7					
7	$\text{SCNCo}(\text{DH})_2\text{SC}$	551.7	1374.4	1555.9	1104.2	764.2	-	1651.7	3228.6	
			1430.0		1283.7					

§ : Recoded as KBr discs and values in cm^{-1} *: L, U=Urea, FA=Formamide, TU=Thiourea, AC=Acetamide, TAC=Thioacetamide, TSC=Thiosemicarbazide & SC=Semicarbazide.

Infrared Spectral DATA Analysis

The thiocyanato bridged binuclear Cobaloximes were characterized by electronic, IR and ^1H NMR techniques. In IR spectra, the thiocyanato binuclear Cobaloximes show the main bands due to the coordinated dimethylglyoxime, the base ligands and the vibrations of the SCN axial ligand. The infrared spectra of the dimer complex shows no absorptions attributable to coordinated H_2O , this proves that it is the H_2O ligand in $\text{CH}_3\text{Co}(\text{DH})_2\text{H}_2\text{O}$ that is substituted. The disappearance of IR peak at 3072cm^{-1} indicates the replacement of aquo group from $\text{CH}_3\text{Co}(\text{DH})_2\text{H}_2\text{O}$ resulting in the formation of bridged complexes [$\text{CH}_3(\text{DH})_2\text{Co-NCS-Co}(\text{DH})_2\text{L}$].

The major difference in the IR spectra of the reactants and products of equation 2 is that the CN stretching frequency of the SCN ligand is at higher energy ($30 - 41\text{ cm}^{-1}$) in the thiocyanato bridged binuclear Cobaloximes than in the thiocyanato(ligand)Cobaloximes. This type of increase in CN stretching frequency of SCN upon forming complexes containing bridged thiocyanato ligands from terminal thiocyanato complexes has been documented by Wilmarth et al[23, 24].

Infrared spectra of thiocyanato bridged Bicobaloxime complexes exhibit a weak and broad absorption in the region $\sim 2180\text{ cm}^{-1}$ due to $\nu(\text{C}=\text{N})$ stretching of the bridged thiocyanate. A decrease in the σ electron donor strength of the trans ligand in $\text{SCNCo}(\text{DH})_2\text{L}$ (Table 1 & 2) complexes results in a regular increase in $\nu(\text{C}=\text{N})$. As a result of coordination of the thiocyanate ligand (anti-bonding π orbital) there is an increase in the CN stretching frequency over the value for ionic cyanide ($\nu(\text{C}=\text{N})$ for ionic cyanide is 2080 cm^{-1}). An examination of the IR data shows that the $\nu(\text{C}=\text{N})$ is highest for the complexes $[\text{CH}_3(\text{DH})_2\text{Co}-\text{NCS}-\text{Co}(\text{DH})_2\text{L}]$ when the ligands $-\text{CH}_3$ and L are the weakest σ donors. In addition, a regular decrease in the $\nu(\text{C}=\text{N})$ is expected as the basicity of L increases. The increase in $\nu(\text{C}=\text{N})$ may be due to steric hindrance of the L. The increase is precisely obtained for the bridged Cobaloximes, where the bridged thiocyanate $\nu(\text{C}=\text{N})$ is seen to be $30 - 40\text{ cm}^{-1}$ higher than in the parent monothiocyanato Cobaloximes, $\text{SCNCo}(\text{DH})_2\text{L}$ as given in Table 3.

Table.2: IR Data.[§] of $\text{SCNCo}(\text{DH})_2\text{L}^*$.

S. No	Complex	$\nu(\text{Co-N})$	DH			SCN		$\nu(\text{Co-N})$	Ligand	
			$\nu(\text{CH}_3)$	$\nu(\text{C}=\text{N})$	N (NO)	$\nu(\text{CS})$	$\nu(\text{CN})$		$\nu(\text{C}=\text{N})$	$\nu(-\text{NH})$
1	$\text{SCNCo}(\text{DH})_2\text{PY}$	511.9	1364.2	1557.5	1090.0	738.4	2143.1	509.8	606.0	3130.8
			1436.0		1229.6				2111.9	
2	$\text{SCNCo}(\text{DH})_2\text{DMPY}$	512.9	1324.2	1560.6	1091.2	739.8	2108.3	507.2	709.0	3205.9
			1410.9		1232.8				2149.2	
3	$\text{SCNCo}(\text{DH})_2\text{ALA}$	514.2	1396.4	1559.8	1089.2	740.6	2102.3	507.6	-	3205.9
			1468.1		1235.4					
4	$\text{SCNCo}(\text{DH})_2\text{AME}$	513.8	1364.0	1558.3	1114.4	749.0	2108.0	510.6	-	3133.8
			1436.7		1232.5					

§ : Recorded as KBr discs and values in cm^{-1}

* : L, PY=Pyrazole, DMPY=Dimethyl pyrazole, ALA=Alanine & AME=Alanine methylester.

Table.3. IR Spectral Data* of SCN stretching frequencies for the bridged[†] and terminally[#] bound Cobaloximes.

S.No	Ligand L in Bridged Cobaloxime	$\nu(\text{C}=\text{N})^{\$}$	$\nu(\text{C}=\text{N})^{**}$
1	Urea	2108.1	2154.1
2	Formamide	2108.3	2173.1
3	Thiourea	2102.3	2120.1
4	Acetamide	2108.3	2136.6
5	Thioacetamide	2107.3	2152.2
6	Thiosemicarbazide	2109.5	2137.2
7	Semicarbazide	2108.3	2127.1
8	Pyrazole	2111.9	2171.0
9	Dimethyl pyrazole	2108.0	2155.0
10	Alanine	2119.9	2139.0
11	Alanine methyl ester	2110.0	2151.9

* : recorded as KBr discs and values in cm^{-1} , †: $[\text{CH}_3(\text{DH})_2\text{Co}-\text{SCN}-\text{Co}(\text{DH})_2\text{L}]$

: $[\text{SCNCo}(\text{DH})_2\text{L}]$ (L = U, FA, TU, AC, TAC, TSC, SC, PY, DMPY, ALA & AME)

§ : $\nu(\text{C}=\text{N})$ in $[\text{SCNCo}(\text{DH})_2\text{L}]$

** : $\nu(\text{C}=\text{N})$ in $[\text{CH}_3(\text{DH})_2\text{Co}-\text{NCS}-\text{Co}(\text{DH})_2\text{L}]$

¹H NMR Data Analysis

¹H NMR data (table 4) of the thiocyanato bridged binuclear Cobaloximes show absorptions corresponding to methyl protons of $\text{Co}-\text{CH}_3$ in bridged binuclear Cobaloxime and methyls of dimethyl glyoximate $(\text{DH})_2$. Absorption due to the 12 equivalent protons of the equatorial dimethylglyoxime ligand at

each of the cobalt center of the thiocyanato bridged binuclear Cobaloximes is observed. The δ (DH)₂ of the dimethylglyoxime bonded to the cobalt which is bonded to methyl (CH₃) ligand of CH₃Co(DH)₂H₂O and δ (DH)₂ of the dimethylglyoxime bonded to the cobalt which is bonded to donor ligands (L) that is SCNCo(DH)₂L are observed as two distinctively different peaks.

Electronic Spectral Data Analysis

Electronic data of binuclear Cobaloximes (Table.5) have been determined in CHCl₃ or MeOH solution made from crystalline samples of the pure bridged binuclear Cobaloximes exhibits a significant feature that the monomeric alkyl(ligand)cobaloxime, CH₃Co(DH)₂L display a weak absorption at 441nm region, that is not present in the electronic spectra of SCNCo(DH)₂L but, a strong absorption appears at 441nm in the CH₃Co(DH)₂H₂O. The disappearance of peak at 441nm clearly confirms the replacement of H₂O during the bridged binuclear cobaloxime formation.

UV –Visible data of all the monomeric CH₃Co(DH)₂L exhibit a visible absorption maximum at 441nm. This is assigned to R⁻ → Cobalt charge transition. A similar visible absorption maximum is present in the electronic spectra of the thiocyanato bridged binuclearcobaloximes at 441nm with very low intensity. The monomeric Alkyl(aquo)Cobaloxime, CH₃Co(DH)₂L display a weak absorption at 441nm region, that is not present in the electronic spectra of SCNCo(DH)₂L, disappearance of this in the Binuclear Cobaloxime clearly confirms the replacement of H₂O during the bridged binuclear Cobaloxime formation.

Thus it is concluded that one of the cobalt centers in the thiocyanato bridged binuclear Cobaloximes must simultaneously be coordinated by CH₃ and a trans donor ligand (L). This can only be true if these compounds possess the –SCN- linkage isomeric structure 1, where one Cobalt is coordinated to CH₃, the other to atom of (N or O or S) donor ligand (L) and both the cobalt's bridged through the thiocyanate. Therefore one would expect the spectrum of the bridged Cobaloxime to be approximately the sum of the monomeric [CH₃Co(DH)₂L] and SCNCo(DH)₂L. In addition in the electronic spectra a peak around ~310nm and 380nm are present.

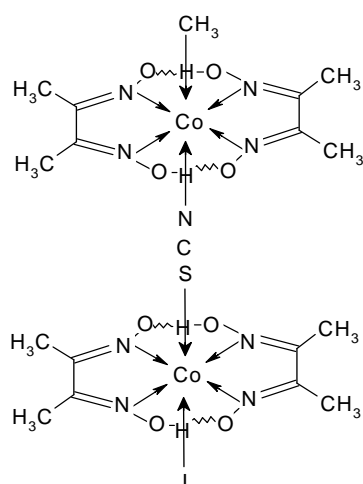
Table.4: ¹H NMR Data CH₃[§](DH)₂*Co-NCS-Co(DH)₂[#]L

S.No	Complex	(DH) ₂ [*]	(DH) ₂ [#]	CH ₃ [§]	L
1	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ FA	2.1	2.0	0.5	7.7
2	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ AC	2.2	2.1	0.5	4.2, 2.3
3	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ TSC	2.2	2.1	0.6	7.5
4	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ PY	2.2	2.1	0.5	6.0
5	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ DMPY	2.2	2.0	0.5	5.8, 2.5
6	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ ALA	2.2	2.10	0.4	2.4, 1.8
7	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ AME	2.2	2.1	0.5	0.8, 3.1

Table.5: Electronic Spectroscopy (UV-Visible Data)* for CH₃(DH)₂Co-NCS-Co(DH)₂L

UV-VISIBLE DATA				
S.No	Complex	Peak I	Peak II	Peak III
1	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ U	431.0	313.5	256
2.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ FA	-	314.0	-
3.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ TU	430.5	318.5	258.5
4.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ AC	438.5	-	-
5.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ TAC	441.0	378.5	303.5
6.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ TSC	440.0	-	-
7.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ SC	431.0	-	-
8.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ PY	431.0	331.0	231.0
9.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ DMPY	423.5	328.5	246.0
10.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ ALA	-	318.5	226.0
11.	CH ₃ (DH) ₂ Co-NCS-Co(DH) ₂ AME	-	313.5	231.0

*:nm

Structure.1: Thiocyanato bridged Bicobaloxime

L = U, TU, FA, AC, TAC, TSC, SC, PY, DMPY, ALA and AME

Conclusion

Binuclear Cobaloximes are good tools to study the Alkyl(aqua) Cobaloximes. The stability of these complexes can be explained on the basis of steric hindrance, HSAB principle and $d\pi-p\pi$ back bonding. CN stretching frequencies of SCN ligand shifts to 40 - 50 cm^{-1} higher compared to monomers, which supports the formation of dimers.

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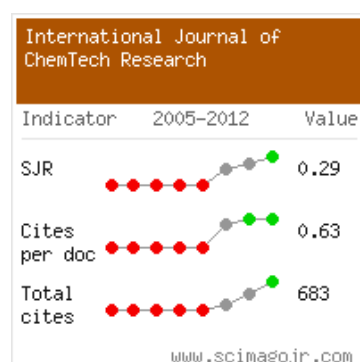
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