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Reactivity of Monochlorotetrakis2-Isopropylphenoxoniobium(V), [NBCL($OC_6H_4CH(CH_3)_2$-2)$_4$] Towards Chelating Ligands

Mala Sharma*

Abstract

The niobium(V) complexes with the formula [NbCl($OC_6H_4CH(CH_3)_2$-2)$_4$] were successfully synthesized in high yields through the reaction of niobium pentachloride with four equivalent amounts of 2-isopropylphenol in carbon tetrachloride. The synthesized complexes were characterized and confirmed by elemental analysis, molar conductance measurements, infrared (IR) spectroscopy, ^1H and ^13C NMR spectroscopy, as well as mass spectrometric analysis. The spectroscopic studies have shown these to exhibit dimeric structures bridging through isopropylphenoxo groups. The X-ray diffraction study of complexes has suggested their amorphous nature. The methanolic solution of newly synthesized complex monochlorotetrakis2-isopropylphenoxoniobium(v), [NbCl($OC_6H_4CH(CH_3)_2$-2)$_4$] react with equimolar amounts of chelating ligands such as β-Diketone, α-hydroxyketone and α-hydroxyaldehydes viz. Acetylacetone(acacH), Benzoin(benzH) and Salicylaldehyde(salH) (LH) in benzene to yield 1:1 coordination compounds authenticated by physicochemical, IR and ^{1}H NMR spectral studies. The spectral data demonstrate that these chelating agents such as Acetylacetone(acacH), Benzoin(benzH) and Salicylaldehyde(salH), are effective in isopropylphenoxo bridging bond-scission and yield unimolecular compounds.

Keywords: Chelating agents, coordination compounds, niobium(v) 2-isopropylphenoxide, spectroscopic studies

INTRODUCTION

The chemistry of niobium alkoxides [1-3] and aryloxides [4-6] continues to be an area of immense research interest over the years owing to their notable complex geometries [7], potential catalytic properties [8] and many valuable applications in the field of chemical synthesizers of oxide and mixed metal oxides [9-11] in material science. The alkoxides readily react with the protons of a large number of organic hydroxyl compounds such as alcohols, glycols, carboxylic acids, β-diketones, alkanolamines etc. containing reactive hydroxyl groups with the replacement of the alkoxo group by the new organic ligand. Niobium ethoxide and niobium butoxide were coordinated with the bidentate ligands chloroacetic acid and dichloroacetic acid in 1:1 and 1:2 molar ratios, employing dry toluene as the solvent. Upon hydrolysis, followed by condensation and subsequent sol–gel processing, both the original and modified niobium alkoxides served as effective precursors for the formation of nanosized niobium oxide particles [12–13]. Furthermore, the chemical modification of metal alkoxides using organic additives such as β-diketones and β-ketoesters has been widely recognized as an efficient strategy for regulating their reactivity and tailoring the structural and physicochemical properties of the resulting gels [14].

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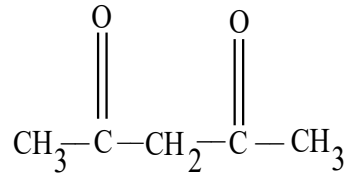
In view of above observations, it is therefore of interest to investigate the reactions of [$NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4$] with chelating ligands such as β-Diketone, α-hydroxyketone and α-hydroxyaldehydes viz. Acetylacetone(acacH), Benzoin(benzH) and Salicylaldehyde(salH).

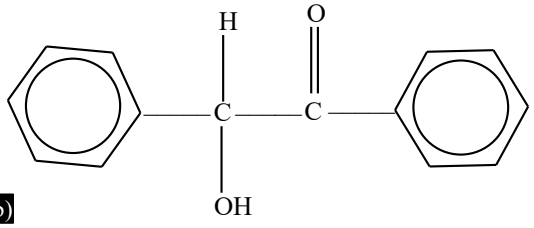
β-Diketone



(a)

Acetylacetone (acacH)

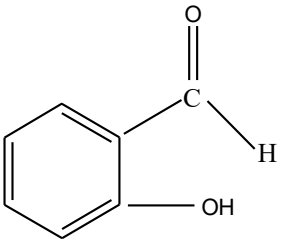
α-Hydroxyketone



(b)

Benzoin (benzH)

α-Hydroxyaldehyde



(c)

Salicylaldehyde (salH)

EXPERIMENTAL

Materials and physical measurements:

$NbCl_5$ (Fluka) was utilized as received without additional purification, and its purity was verified through chlorine analysis. 2-Isopropylphenol (Merck; b.p. 210 °C) was purified by vacuum distillation prior to use. All solvents were rendered anhydrous using standard drying procedures. The niobium content in the synthesized complexes was determined gravimetrically as Nb_2O_5 after decomposing the complexes with a concentrated H_2SO_4–HNO_3 mixture, followed by ignition at 650–700 °C. Chlorine content was estimated by Volhard's method.

Elemental (C and H) microanalyses were carried out using an Eager 300 NCH System Elemental Analyzer. The molar conductance of 10^{-3} M solutions of the complexes in nitrobenzene was measured at 25 ± 0.1 °C with an Elico conductivity bridge (CM-82T). Molecular weights were determined by the cryoscopic method in benzene (0.0015–0.0020 M) employing a Beckmann thermometer. Infrared spectra (KBr pellets) were recorded on a Nicolet-5700 FTIR spectrophotometer, while ^1H NMR spectra were obtained on a BRUKER AVANCE II 400 spectrometer using $CDCl_3$ as the solvent.

Synthesis of [$NbCl\ (OC_6H_4CH(CH_3)_2\text{-}2)_4$]

The niobium(V) complexes with the composition [$NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4$] were synthesized in quantitative yields through the reaction of niobium pentachloride with the corresponding substituted phenols, taken in suitable molar ratios, in carbon tetrachloride under reflux conditions [15].

Reactions of [$NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4$] with β-Diketone, α-Hydroxyketone and α-hydroxyaldehydes viz. Acetylacetone, Benzoin and Salicylaldehyde (LH)

To benzene solution of parent complex, were added methanolic solution of equimolar amount of the ligands viz. acetylacetone, benzoin and salicylaldehyde (LH) in separate experiments. These were stirred at room temperature for 3-4 h. The reaction mixture was subsequently heated under reflux, during which a noticeable color change occurred along with the release of hydrogen chloride (HCl) gas. Upon completion of the reaction, when no more gas evolved, it was concentrated to ¾th of its original volume,

when the solids separated out. They were subsequently filtered, rinsed with petroleum ether and hexane, and dried under vacuum. In the case of reaction of complex with salicylaldehyde, acetonitrile was added to obtain complex as solid. Anal. Calcd. for $Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4.acac$, $C_{41}H_{52}O_6Nb$ (%): C, 67.10; H, 7.05; Cl, 0; Nb, 12.65. Found: C, 67.12; H, 7.09; Cl, 0; Nb, 12.69. Λ_m, ($PhNO_2$): 0.37 Scm^2mol^{-1}. Anal. Calcd. for $Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4.benz$, $C_{50}H_{56}O_6Nb$ (%): C, 71.01; H, 6.60; Cl, 0; Nb, 11.01. Found: C, 71.00; H, 6.63; Cl, 0; Nb, 11.00. Anal. Calcd. for $Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4.sal$, $C_{43}H_{50}O_6Nb$ (%): C, 68.30; H, 6.61; Cl, 0; Nb, 12.30. Found: C, 68.34; H, 6.62; Cl, 0; Nb, 12.32. Λ_m, ($PhNO_2$): 0.28 Scm^2mol^{-1}.

RESULTS AND DISCUSSION

The reactions of methanolic solution of $[NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4]$ with equimolar amounts of acetylacetone(acacH), benzoin(benzH) and salicylaldehyde(salH) (LH) in benzene yield complexes of composition $[Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4.L]$. The reactions may be represented as:

$$NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4 + LH \xrightarrow{\text{benzene/methanol}} Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4L + HCl$$

where LH = acacH, benzH and salH and L = anions of acac, sal and benz.

The elemental analysis of the isolated complexes agreed well with their stoichiometric composition (Table 1). The complexes are fine coloured powders and are fairly soluble in common in organic solvents, the molar conductance values of millimolar solutions of the complexes in nitrobenzene suggest that they behave as non-electrolytes.

IR Spectra

The infrared spectra scanned in 4000−200 cm^{-1} region for the complexes have provided further information on their formation. The absence of absorption band at ~ 3333 cm^{-1} due to simple hydrogen bonded –OH group of the enolic form of acetylacetone, in complex suggested the loss of hydrogen of the –OH group as HCl gas. The sharp bands observed in 1600−1580 $cm^{-1}$and 1370−1350 cm^{-1} regions ascribed to υ_{asym}(C=O) and υ_{sym}(C=O) modes in the enolic form of acetylacetone have been observed to move to lower wave numbers by 20−25 cm^{-1} upon complexation. These trends are suggestive of bonding through oxygen atoms to the niobium metal in agreement with the earlier reports on metal acetylacetonates [16]. The sharp bands observed in 1535−1520 cm^{-1} region have been assigned to υ(C = C) mode.

The IR spectra of niobium(V) complexes derived from benzoin have shown the absence of absorption band due to υ(OH) mode known to occur at ~ 3360 cm^{-1} in pure benzoin, confirming the hydrogen chloride gas evolution during the reactions of monochloroniobium(V)2-/4-isopropylphenoxides with the ligand. The bands due to υ(C–O) mode known to occur in 1110−1090 cm^{-1} region in free benzoin appeared at lower wave numbers in complex. These findings suggest that the hydroxyl oxygen is coordinated to the niobium atom. All significant absorption bands are presented in Table 2.

Table 1. Analytical data of reaction products of $NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4$ with chelating ligands.

Complex Molecular formula (Formula weight)	**Colour**	**Elemental Analysis Found (Calc.) %**					**Molar Cond. in $PhNO_2$ (Scm^2 $mole^{-1}$)**
		Nb	***Cl***	***C***	***H***	***N***	
$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4.acac$ $C_{41}H_{52}O_6Nb$ (733)	Brown	12.65 (12.69)	--- ---	67.10 (67.12)	7.05 (7.09)	---	0.37
$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4.benz$ $C_{50}H_{56}O_6Nb$ (845)	Dark Brown	11.01 (11.00)	--- ---	71.01 (71.00)	6.60 (6.63)	---	---
$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4.sal$ $C_{43}H_{50}O_6Nb$ (755)	Brown	12.30 (12.32)	--- ---	68.30 (68.34)	6.61 (6.62)	---	0.30

acac = ion of acetylacetone, benz = ion of benzoin, sal = ion of salicylaldehyde

Table 2. IR spectral data (cm^{-1}) of reaction products of $NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4$ with chelating ligands.

Complex	Bands (cm^{-1})
$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4$.acac	3864s, 3754s, 3654s, 3421, 2962, 2924, 2872, 2962s, 2924s, 2872s, 2062, 1634, 1587, 1525s, 1484s, 1457s, 1420, 1368s, 1328s, 1261s, 1195s, 1149s, 1108s, 1079, 1027, 917, 888, 859, 836, 796, 744, 709, 663, 616, 582, 564, 530, 472, 426.
$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4$.benz	3414, 2961, 2918, 2866, 1664s, 1629s, 1594s, 1513s, 1484s, 1448s, 1420, 1330s, 1282s, 1250s, 1206, 1166s, 1149s, 1113, 1079s, 940, 871s, 830, 796, 752, 719, 674, 642, 582, 426, 356.
$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4$.sal	3391, 2961, 2930, 2866, 1663, 1629, 1546s, 1490s, 1461, 1397, 1279s, 1218, 1150s, 1117, 1073, 1027, 883, 836, 760, 709, 663, 612, 489, 449, 350.

where acac = ion of acetylacetone, benz = ion of benzoin, sal = ion of salicylaldehyde,

The υ(C=O) mode of free benzoin known to occur at ~ 1690 cm^{-1} has been observed to move to lower wave numbers confirming the bonding through carbonyl oxygen to the metal. It has thus been inferred from these observations that a simultaneous coordination from carbonyl as well as hydroxyl oxygen has resulted into the formation of niobium(V) chelate complex, is in agreement with the earlier reports on spectra of similar complexes [12, 13].

The absence of band due to υ(OH) mode known to occur in infrared spectra of $[NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4$.sal] at 3180 cm^{-1} in pure salicylaldehyde suggested the formation of compounds through deprotonation of –OH group. The υ(C=O) mode of free salicylaldehyde occurring at 1660 cm^{-1} has been found to shift to lower regions by ~ 25–30 cm^{-1} on complexation, which may be attributed to the coordination of carbonyl oxygen to niobium.

This is supplemented by a shift in the band at 1200 cm^{-1} in salicylaldehyde assignable to υ(C–C) mode to higher regions on complexation. These shifts are in agreement with the earlier reports on the spectra of similar complexes of metal halides. The sharp bands observed in 580−570 cm^{-1} region have been assigned to terminal υ(Nb–O) mode [17–20].

No band that could be assigned to bridging  mode expected around 520 cm^{-1} were observed in IR spectra of complexes suggesting the absence of phenoxy bridging, characteristic of parent niobium(V) complex.

^{1}H NMR spectra

Further evidence on the formation of mixed ligand niobium(V) complexes derived from acetylacetone, benzoin and salicylaldehyde (LH) has been obtained from a comparison of their room temperature ^{1}H NMR spectra with that of free ligands and parent complex $[NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4]$.

The ^{1}H NMR spectra of free acetylacetone is known to display signals at δ 2.14 and δ 3.57 ppm due to methyl and methylene protons respectively while the enolic form of acetylacetone exhibits signals at δ 5.08, δ 1.97 and δ 5.50 ppm due to OH, methyl and methine protons respectively. The most stable tautomeric form of 1,3-diketones is the enol form; the carbon-carbon double bond of the enol form is in conjugation with the second carbonyl group (Figure 1).

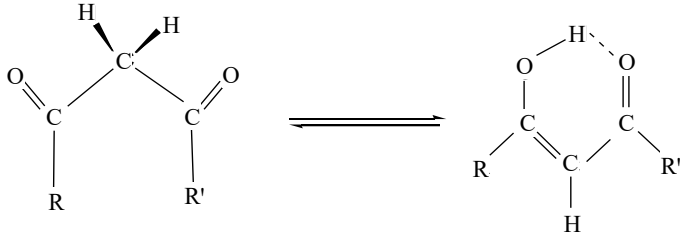


Figure 1. Proposed mechanism illustrating the keto–enol tautomerism of the β-dicarbonyl compound.

The hydroxylic absorption of enols occurs at very low field δ 4.0 to δ 5.0 ppm and is usually unaffected by dilution or solvent interaction. In spectra of acetic acid solutions of enols, both hydroxyl and carboxyl absorptions may be observed, which indicate negligible chemical exchange even in this solvent. The resonance due to hydroxyl group of the enol form of acetylacetone is reported to occur at δ 5.08 ppm (Figure 2).

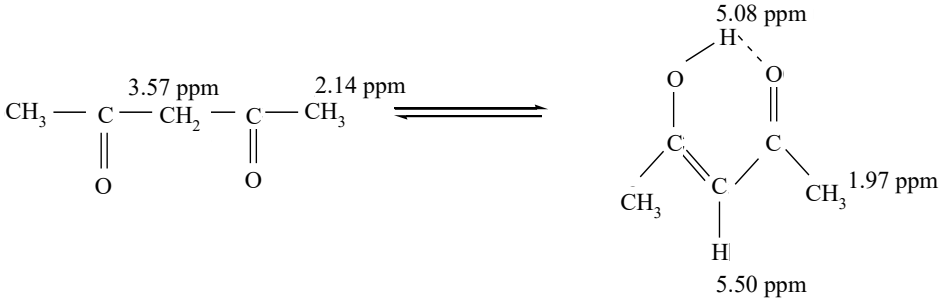


Figure 2. Keto–enol tautomerism of a β-diketone system with corresponding ^1H NMR chemical shifts.

The ^{1}H NMR spectra of enols show signals of both keto and and enols forms. Thus, interchange between these two forms on the NMR time scale is very slow. Integration of the spectrum of acetylacetone indicates the presence of 84% of the enol form and 16% of the keto form in the pure liquid at 40°. Acetylacetone exists in its enol tautomeric form to approximately 15% in aqueous solution, whereas in hexane it predominates at nearly 92%.

The greater enolization in hexane results from stabilization of the enol form by internal hydrogen bonding. In aqueous solutions the carbonyl groups are hydrated, or hydrogen bonded to water molecules, and there is less to gain by enolization.

Therefore, polar solvents which stabilize the keto carbonyls will decrease the percent of enolization depending on the degree of their stabilization. An increase in the concentration of a polar solvent in a solution will increase the degree of interaction with acetylacetone (Table 3). Experiments have established that the intramolecular hydrogen bond of acetylacetone stabilizes the enol tautomer by 5 to 10 kcal and the conjugated system further stabilizes this tautomer by another 2 to 3 kcal.

Table 3. ^{1}H-NMR data of reaction products of $NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4$ with chelating ligands (ppm).

Complex	**Substituent (Isopropyl protons)**		**Aromatic phenolic ring protons**	**(Ligands)**			
	$-(CH_3)_2$	-CH		$-CH_2$	$-CH_3$	-CH	$-C_6H_5$
$NbCl(OC_6H_4CH(CH_3)_2\text{-}2)_4$	1.20	3.26-3.37	6.77-7.14	---	---	---	---
$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4$.acac	1.15-1.17	3.20-3.25	6.69-7.08	3.44	2.01	---	---
$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4$.benz	1.15-1.19	3.20-3.25	6.69-7.08	---	---	---	7.58-7.96
$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4$.sal	1.18-1.25	3.34-3.52	6.72-7.09	---	---	10.15	7.46-7.68

where acac = ion of acetylacetone, benz = ion of benzoin and sal = ion of salicylaldehyde

The ^{1}H NMR spectra of [$Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4$.acac] displayed signals at δ 2.01 and δ 3.44 ppm due to methyl and methylene protons respectively. The aromatic protons of the phenolic ring were found to undergo slight upfield shifts by δ 0.05−0.08 ppm and appeared at δ 6.69−7.09 ppm in mixed-ligand complexes relative to that in the parent complex occurring at δ 6.77−7.14 ppm.

The ^{1}H NMR spectra of free benzoin is known to exhibit signals at δ 2.0, δ 6.01 and in 7.19−7.86 ppm range due to OH, CH and aromatic protons respectively. The free salicylaldehyde exhibits signals due to OH, CH and aromatic protons at δ 5.0, δ 10.24 and δ 6.92−7.64 ppm range respectively. The aromatic protons due to chelating ligands benzoin and salicylaldehyde appeared in δ 7.58−7.95 ppm and δ 7.46−7.68 ppm range respectively in their complexes (Figures 3–6).

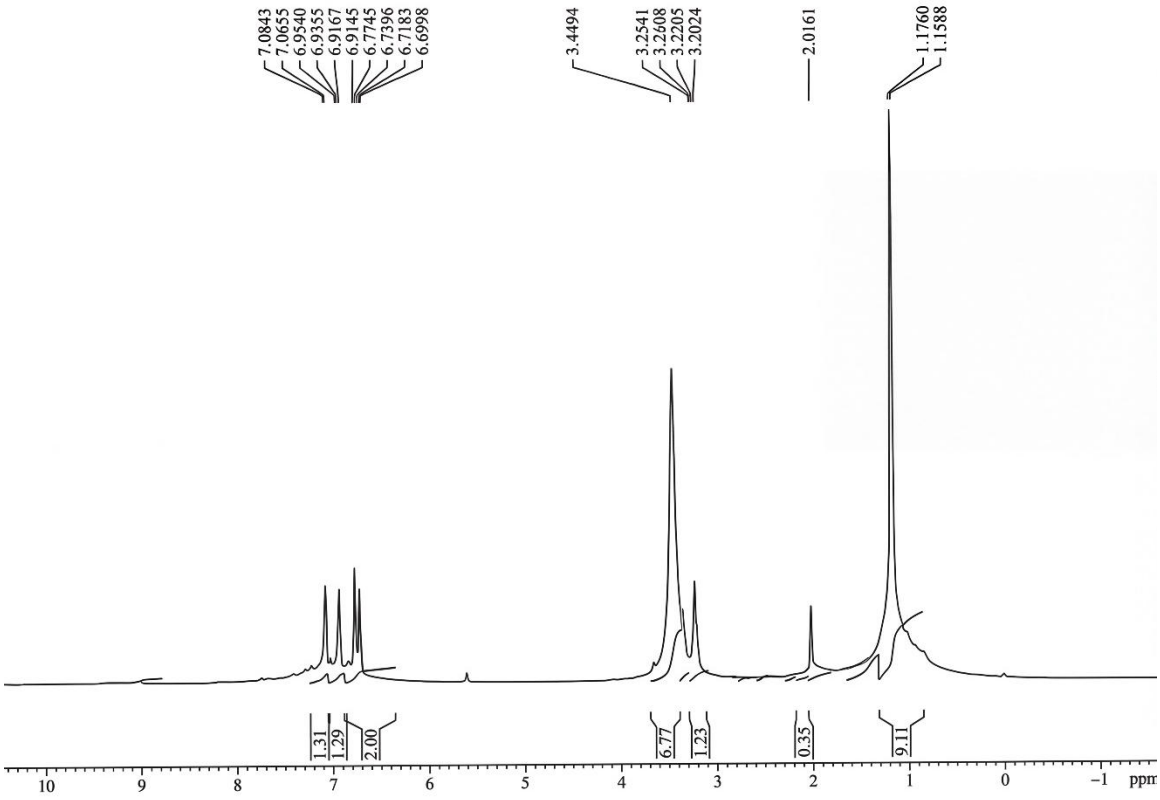


Figure 3. ^{1}H NMR spectrum of $Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4$.acac.

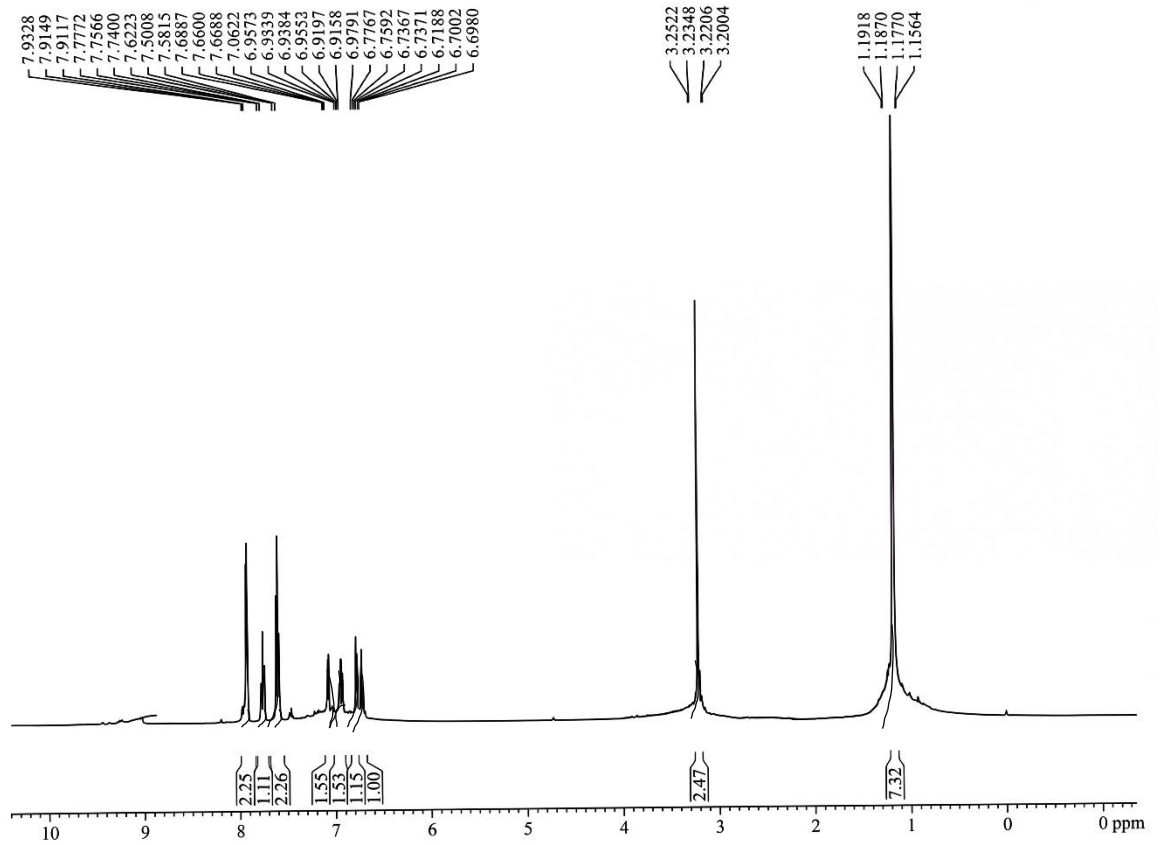


Figure 4. ^{1}H NMR spectrum of $Nb(OC_6H_4CH(CH_3)_2\text{-}2)_4$.benz.

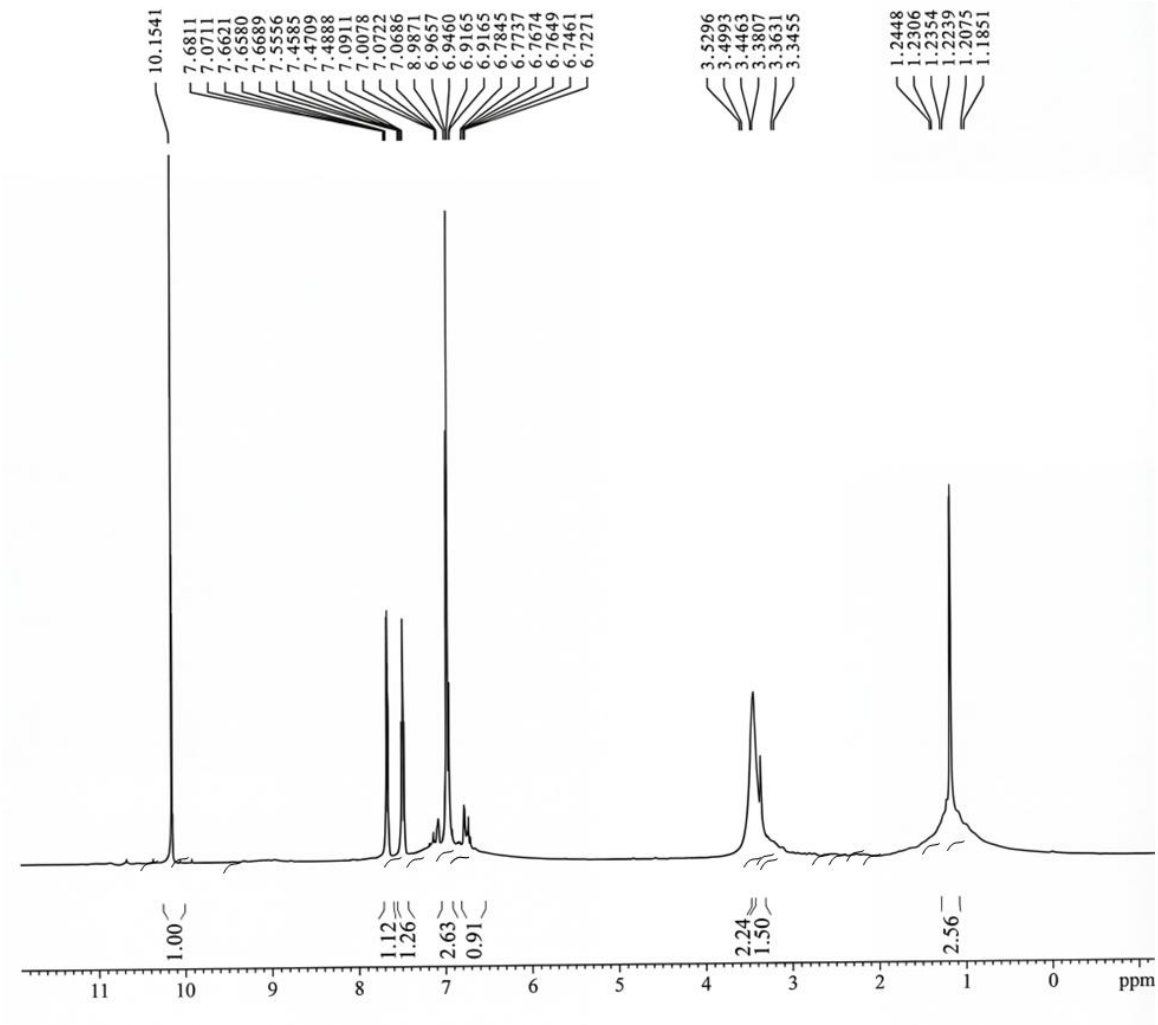
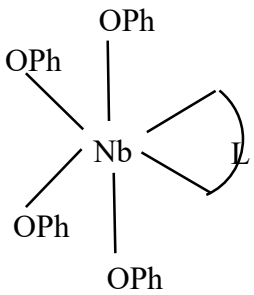


Figure 5. ^{1}H NMR spectrum of Nb($OC_6H_4CH(CH_3)_2$-2)$_4$.sal.

Thus, based upon IR and ^{1}H NMR spectral data coupled with physicochemical studies, a distorted octahedral structure for complexes [Nb($OC_6H_4CH(CH_3)_2$-2)$_4$.L] isolated from the reactions of parent monochloro 2-isopropylniobium(V) phenoxide with chelating ligands may tentatively be proposed as:



(where OPh = $OC_6H_4CH(CH_3)_2$-2 and L = anions of Acetylacetone (acacH), Benzoin (benzH) and Salicylaldehyde (salH))

Figure 6. Tentatively proposed structure.

CONCLUSION

The spectroscopic studies of newly synthesized niobium(V) complex suggested dimeric structure involving bridging through 2-isopropylphenoxo group. The reactions of NbCl($OC_6H_4CH(CH_3)_2$-2)$_4$

with chelating ligands such as β-Diketone, α-hydroxyketone and α-hydroxyaldehydes viz. Acetylacetone(acacH), Benzoin(benzH) and Salicylaldehyde(salH) (LH) yielded mononuclear 1 : 1 coordination compounds and based upon IR and ^{1}H NMR spectral data coupled with physicochemical analysis a distorted octahedral geometry around niobium in complexes may tentatively be proposed.

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