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Heterobimetallic [Ba(II)-Ti(IV)]-μ-oxoisopropoxide and its β-diketonates presursors; Synthesis, spectral and thermal studies

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ABSTRACT

Thermal condensation of Barium acetate and titanium(IV) isopropoxide resulting the synthesis of new heterobimetallic [Ba(II)-Ti(IV)]-μ-oxoisopropoxide. The characterization and structural elucidation of μ-oxo compound was made by liberated isopropanol estimation, elemental, spectral (IR, ^{1}H , ^{13}C, NMR and mass), thermal analysis, molecular weight data. Many isopropoxy substitution reactions of [Ba(II)-Ti(IV)]-μ-oxoisopropoxide compound with β-diketones in different molar ratios (1:1-1:4) yielded the mono to tetra derivatives [$\{BaO_2Ti_2(OPr^i)_{6-n}L_n\}$ (where n is 1-4 and L = acetylacetonate/benzoylacetonate anion)] respectively of the parent compound and have been characterized by elemental, liberated isopropanol and spectral analysis (IR, ^{1}H , ^{13}C NMR).The above studies revealed interesting facets in support of plausible structures of parent compound and its β-diketonates.

Key words: heterometallic-μ-oxoisopropoxide, Barium, Titanium, β-diketones, thermoanalysis

INTRODUCTION

Due to the perovskite structure and high dielectric constant of $BaTiO_3$ which is significantly higher than other ceramics like SiO_2 is used in fields requiring high piezoelectricity and high dielectric constants because of its polarization inversion and high dielectric constant.[1] However, the material for the insulating layer needs to be flexible and stretchable in order to grab the various shaped objects. In many studies of high dielectric constant composites, polyvinylidene fluoride with a relatively high dielectric constant has been used among polymer materials.[2,3] The investigation and the use of heterometallic alkoxides as single-source molecules precursors for synthesis of oxides have seen a rapid growth during the last more than one and half decade. The bimetallic oxo complexes, one among the best precursors play a significant role to prevent the phase segregation problem which leads to the formation of oxides of multiple components. The bimetallic oxo complexes provide homogeneity of the newly formed oxide phases at the molecular level. The above considered thing in the composition, stoichiometry, solubility and reactivity of ortho- and oxoalkoxides are widely used in the sol-gel synthesis of a series of very important composites. The control of particle size and the morphology of the oxide are of crucial importance nowadays both from the fundamental and industrial point of view. The mixed metal oxides prepared from heterometallic oxoalkoxides have been used for absorbing harmful chemicals[4-7] and gases such as SO_2, CCl_4, and decontaminating chemical warfare agents.[8] The alkaline earth metal titanates like barium titanate, calcium titanate, strontium titanate ($SrTiO_3$ and $Ba_xSr_{1-x}TiO_3$) due to their exceptional properties expose potential applications in multilayer ceramic capacitors, electro-optic, dielectric and piezoelectric devices.[9-12] The perovskite $BaTiO_3$ with its unique structure, higher stability and biocompatibility, finds potential application in the fields of communication, electronics and in biotechnology.[13-15] Recently, spherical $BaTiO_3$ have been

used in many studies for composites with high dielectric constants, and in the case of rod-like shape BT, many studies have investigated the aspect ratio which affects the dielectric constant.[24,25] Calcium titanate has also been used as a competent anticorrosion pigment for paints.[18] Apart from their role as precursors for mixed metal oxides, the bimetallic-μ-oxoalkoxides of transition metals have been found to rank among the excellent catalysts for the polymerization of heterocyclic monomers like lactones, oxiranes, thiiranes and epoxides.[19,20]

In the present investigation, heterobimetallic [Ba(II)-Ti(IV)]-μ-oxoisopropoxide is prepared from the condensation of one mole of barium acetate and two moles of titanium isopropoxide, and the reaction proceeds with stepwise formation of pure bimetallic [Ba(II)-Ti(IV)]-μ-oxoisopropoxide, which is a molecular species that can be purified by distillation, allowing the isolation of pure molecular precursors and to gain an insight in to its structure and prevent the phase secretion problem due to its strong tendency to hydrolysis, its β-diketonates are synthesized.

RESULTS AND DISCUSSION

The preparation of the heterometallic-μ-oxoisopropoxide $[BaO_2Ti_2(OPr^i)_6]$ proceeds the following reaction ***scheme 1***:

$$Ba(OAc)_2 + Ti(OPr^i)_4 \xrightarrow[120\ ^oC]{\text{decalin, xylene}} [(OAc)BaOTi(OPr^i)_3] + AcOPr^i$$

$$[(OAc)BaOTi(OPr^i)_3] + Ti(OPr^i)_4 \xrightarrow[190\ ^oC]{\text{decalin, xylene}} [BaO_2Ti_2(OPr^i)_6] + AcOPr^i$$

Scheme 1

To further gain an insight into the structure, effect on the solubility and stability, many reactions of $[BaO_2Ti_2(OPr^i)_6]$ with β-diketones (HL) were carried out in different molar ratios in refluxing benzene. The reactions yielded compounds of the type $[BaO_2Ti_2(OPr^i)_5L]$, $[BaO_2Ti_2(OPr^i)_4L_2]$, $[BaO_2Ti_2(OPr^i)_3L_3]$ and $[BaO_2Ti_2(OPr^i)_2L_4]$ according to the following reaction ***scheme 2***:

$[BaO_2Ti_2(OPr^i)_6]$ + nHL $\xrightarrow{\text{refluxing benzene}}$ $[BaO_2Ti_2(OPr^i)_{6-n}L_n]$ + nPr^iOH

(n= 1-4, HL = acetylacetone/ benzoylacetone)

Scheme 2

The $[BaO_2Ti_2(OPr^i)_6]$ and its β-diketonates are susceptible to hydrolysis and soluble in common organic solvents such as benzene, chloroform and carbon tetrachloride etc. The isopropanol liberated during the course of the reaction was collected azeotropically (isopropanol-benzene) and estimated oxidimetrically to check the progress of the reaction. It was observed that only four out of the six isopropoxy groups of [Ba(II)-Ti(IV)]-μ-oxoisopropoxide could be replaced by β-diketones. Futher replacement of isopropoxy groups could not be achieved even with an excess of ligand (β-diketones) and prolonged refluxing time (22 h). This indicates the non-replacement of bridging isopropoxy groups and that only terminal isopropoxy groups are substituted by β-diketones.

Spectral Analysis of $[BaO_2Ti_2(OPr^i)_6]$ and its β-diketonates

IR spectra

The strong bands observed at 1615 cm^{-1} and 1445 cm^{-1} due to asym $C{\cdots}O$ and sym $C{\cdots}O$ stretch respectively in the IR spectrum of calcium acetate are found absent in the spectrum of [Ba(II)-Ti(IV)]-μ-oxoisopropoxide indicating the complete removal of acetate groups. The spectrum of the μ-oxoisopropoxide shows absorption bands at ~1365 cm^{-1} and ~1175 cm^{-1} are the characterstics of *gem*-dimethyl portion and combination band ν(C-O+OPr^i) of the terminal and bridging isopropoxy group respectively. A band appearing at approximately 970 cm^{-1} is assigned to ν(C-O) stretching of bridging isopropoxy group.[21] The IR spectra of 1:1 to 1:3 β-diketonates show absorption bands in the region 1385–1330 cm^{-1} and 1180–1140 cm^{-1} which are characterstics of *gem*-dimethyl group and combination band ν(C-O+OPr^i) of the terminal and bridging isopropoxy groups respectively. Absence of band at 1385–1330 cm^{-1} in the spectrum of 1:4 β-diketonates indicates the absence of terminal isopropoxy group. A band is appearing at ~970 cm^{-1} due to

ν(C-O) stretching of bridging isopropoxy group. The IR spectrum of β-diketones display strong bands at ~1605–1585 cm^{-1} and ~1515–1505 cm^{-1} due to ν_{sym}(C=O) and ν_{asym}(C=C) respectively along with a broad band at ~ 3110–2705 cm^{-1} due to enolic ν(O-H). The non shifting of ν(C=O) frequency and the disappearence of broad band appearing in the region 3205–2710 cm^{-1} in β-diketones suggests that bonding takes place through both the oxygens of CO groups in the derivatives A number of vibrations are observed in the region 700-400 cm^{-1} due to M-O stretching vibrations in μ-oxo compound and its β-diketonates.[22]

NMR spectra

A sharp singlet observed at δ 2.3 ppm in the ^{1}H NMR spectrum of barium acetate is absent in the spectrum of $[BaO_2Ti_2(OPr^i)_6]$, which confirms the complete removal of acetate groups. ^{1}H NMR spectrum of [Ba(II)-Ti(IV)]-μ-oxoisopropoxide shows a number of peaks centered between δ 1.1 - 1.5 ppm due to the intermixing of methyl protons of terminal and bridging isopropoxy groups.[23] A multiplet centered at δ 4.6-4.8 ppm is observed due to the methine proton of isopropoxy group in the μ-oxo compound. The ^{1}H NMR spectrum of $[BaO_2Ti_2(OPr^i)_6]$ is presented in Figure 1. In the ^{1}H NMR spectra of the β-diketonates, the enolic peak at δ 12.5 ppm in β-diketones is found absent and a broad overlapping multiplet centered between δ 0.8–1.6 ppm in all derivatives at room temperature is due to intermixing of the methyl protons of the bridging and isobranching vibrations of isopropoxy groups. A broad multiplet centered at δ 4.0-4.6 ppm is due to the methine proton of isopropoxy groups in all spectra. All β-diketonates of μ-oxoisopropoxide compound show singlet at δ 2.1-2.3 ppm and δ 5.6-5.9 ppm due to methyl and methine proton of the ligand moiety respectively. Further, the peaks observed in the region δ 7.0–7.7 ppm in benzoyl acetone derivative of [Ba(II)-Ti(IV)]-μ-oxoisopropoxide are due to the phenyl ring protons.

The ^{13}C NMR spectrum of [Ba(II)-Ti(IV)]-μ-oxoisopropoxide shows two prominent peaks at ~ δ 26.6 and ~ δ 27.1 ppm assignable to the methyl carbon of terminal and bridging isopropoxy groups and the other two peaks observed at δ 61.7 ppm and δ 63.2 ppm in the ^{13}C NMR spectrum are due to terminal and

bridging methine carbon of the isopropoxy groups.[24] The ^{13}C NMR spectrum of [Ba(II)-Ti(IV)]-μ-oxoisopropoxide is shown in Figure 2. The ^{13}C NMR spectra of 1:1 to 1:3 β-diketone derivatives of μ-oxoisopropoxide compound show two prominent peaks between δ 26.8–27.8 ppm and δ 28.1–29.2 ppm assignable to the methyl carbon of terminal and bridging isopropoxy groups. The two peaks observed at δ 61.8–62.9 ppm and δ 62.8–63.7 ppm are assignable to the methine carbons of terminal and bridging isopropoxy groups in the derivatives. Single peak is observed at δ28.3-29.2 and 63.1-63.9 ppm in 1:4 derivatives. Two peaks observed in the range δ 182.6–193.1 ppm and δ 100.4–93.4 ppm are due to carbonyl carbon and methine carbon of ligand moiety in all β-diketonates. The peaks observed at δ 127.7, 126.5, 124.3 and 135.8 ppm are due to *ortho, meta, para* and substituted carbon of the phenyl ring respectively in the spectra of benzoylacetone derivative of parent compound.

Mass spectra

The positive ion mass spectrum of $BaO_2Ti_2(OPr^i)_6$ was carried in dry toluene containing 17% isopropanol by volume. The significant mass peaks observed at (m/z) 615.2, 558.8, 498.3, 391.8, 380.3, 285.1, 223.7, 199.2, 136.8 and 47.5 in the spectrum can be assigned to the fragments $BaO_2Ti_2(OPr^i)_6^+$, $BaO_2Ti_2(OPr^i)_5^+$, $BaO_2Ti_2(OPr^i)_4^+$, $BaO_2Ti(OPr^i)_3^+$, $BaO_2Ti_2(OPr^i)_2^+$, $BaO_2(OPr^i)_2^+$, $Ti(OPr^i)_3^+$, $BaOTi^+$, Ba^+ and Ti^+ respectively.[25] The mass spectrum of $BaO_2Ti_2(OPr^i)_6$ is shown in Figure 3.

Thermal Analysis of [$BaO_2Ti_2(OPr^i)_6$]

TGA of [$BaO_2Ti_2(OPr^i)_6$] has been performed up to 700 ^oC at 10^oC/min. The total weight loss of 86.45% was observed from 56.9 to 458.3^oC. A small weight loss between 58.9 to 180^oC is probably due to the presence of moisture and traces of solvent in compound. A rapid weight loss was observed between ~ 180 to 458.3^oC indicates the volatile nature of μ-oxo compound. Further, the remaining weight 11.35 % observed is due to the decomposition of partially hydrolysed μ-oxo compound in to mixed metal oxide.

The TGA of hydrolyzed product of $[BaO_2Ti_2(OPr^i)_6]$ have been performed up to 800°C at 10°C/min. The weight loss of about 1.5% in hydrolyzed product of parent compound is due to the traces of water present. The weight loss of 8.59% observed from 212°C to 345°C is probably due to the elimination of hydroxy groups and organic moieties[26] present in the hydrolyzed product $[BaO_2Ti_2(OH)_6]$ resulting from the hydrolysis of $[BaO_2Ti_2(OPr^i)_6]$. The final product remaining is probably the mixed metal oxide. The TG analysis is consistent with the formulation of the compound as $[BaO_2Ti_2(OPr^i)_6]$.

The molecular weight measurement carried out in dry benzene using cryoscopic method suggests monomeric nature of the compound.

The molecular weight measurement carried out in dry benzene using cryoscopic method suggests monomeric nature of β-diketonates.

EXPERIMENTAL

All the operations were carried out in dry nitrogen atmosphere using a vacuum line to exclude the moisture throughout the present work. Hydrocarbon solvents and reagents used were purified and dried by standard methods. The general technique were employed for obtain the physical measurement data as described elsewhere.[27-30] Barium acetate (Aldrich) was made anhydrous with acetic anhydride and titanium isopropoxide $[Ti(OPr^i)_4]$ (Aldrich) used without further purification. Acetyl acetone was dried prior to use and benzoyl acetone (Hi-media) was used as received. The isopropoxy groups in the μ-oxoisopropoxide and liberated isopropanol formed in preparation of β-diketonates were estimated oxidimetrically. Barium was determined complexometrically and gravimetric estimation has been done for titanium.[31] Titanium was estimated as TiO_2 via the formation of titanium-phenazone complex.[28] Proton NMR, ^{13}C NMR spectra were recorded in $CDCl_3$ on Bruker Avance II 400 NMR spectrometer. The Infrared spectra recorded using Perkin-Elmer 1710 FTIR spectrometer over the range 4000-400 cm^{-1}. TG studies have been made by rising the

temperature with a linear rate using Diamond TG/DTA Perkin-Elmer instrument. Elemental analyses were carried out by PerkinElmer 2400 II series CHNS/O Analyzer.

The mass spectrum recorded on a Waters QTOF2 mass spectrometer equipped with Quadrupole and time of flight (TOF) analyzers.

Synthesis of $[BaO_2Ti_2(OPr^i)_6]$

The [Ba(II)-Ti(IV)]-μ-oxoisopropoxide was synthesized by thermal condensation between $Ba(OAc)_2$ (2.719 g, 10.664 mmol) and $Ti(OPr^i)_4$ (6.036 g, 21.329 mmol) in mixture of xylene and decalin. The contents refluxed for about 18 h on a fractionating column and the isopropyl acetate formed in the reaction was removed continuously from 80°C until the temperature rose to the boiling point of decaline (190°C). The reaction was then continued at this temperature for 2 h to ensure its completion. The excess of decalin was distilled at 40-50°C/l mm, leaving behind a yellow semi-solid product which was dissolved in benzene. Slow evaporation of benzene gave a pale yellow glassy solid which get decomposed on heating strongly (dec. > 435 0°C). [Yield: 83%; For $[BaO_2Ti_2(OPr^i)_6]$ Anal.: Found(%): OPr^i, 54.9; Ba, 21.8; Ti, 14.6 calcd(%): OPr^i, 57.5; Ba, 22.3 Ti, 15.3].

Reaction of $[BaO_2Ti_2(OPr^i)_6]$ with acetylacetone (Hacac) in 1:1 molar ratio:

The $[BaO_2Ti_2(OPr^i)_6]$ (0.832 g, 1.352 mmol) and acetylacetone (0.135 g, 1.352 mmol) were refluxed in ~ 50 ml benzene in a flask connected to a short distillation column on an oil bath for about 8 h. The isopropanol liberated at 72-78^{o}C was fractionated as the binary azeotrope of isoproponol-benzene. The azeotrope was collected and checked for completion of the reaction. The excess of the solvent was then removed under reduced pressure yielding a yellowish semi-solid product. The syntheses of other β-diketonates were carried out by similar procedure and the analytical results have been summarized in Table 1 and Table 2.

CONCLUSION

The aforementioned studies reveals the suggestive structures of the $[BaO_2Ti_2(OPr^i)_6]$ and its β-diketonate of the type $[BaO_2Ti_2(OPr^i)_5(L)]$, $[BaO_2Ti_2(OPr^i)_4(L)_2]$, $[BaO_2Ti_2(OPr^i)_3(L)_3]$ and $[BaO_2Ti_2(OPr^i)_2(L)_4]$. TGA study reveals the volatile nature of parent compound and its hydrolysed product may fabricate the mixed metal oxides. It is observed the β-diketonate are found more stable and less prone to hydrolysis as compared to parent compound. The proposed structures of parent compound and its terta derivatives are shown in Figure 4 and Figure 5 respectively.

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TABLE 1 – Analytical and physical data of the studied compounds

S.No.	Comp. g (mmol)	Ligand g(mmol)	Molar ratio	Refluxing time (hrs)	Product (%)	Anal. Found (Calc.) HOPr^i g(%)	Ba (%)	Ti (%)
1.	$[BaO_2Ti_2(OPr^i)_6]$ 0.832(1.35)	Hacac 0.135 (1.35)	1:1	8	$[BaO_2Ti_2(OPr^i)_5(acac)]$ (76.8)	43.29 (45.03)	19.72 (20.91)	14.32 (14.35)
2.	$[BaO_2Ti_2(OPr^i)_6]$ 0.308 (0.5)	Hacac 0.1 (1.0)	1:2	9½	$[BaO_2Ti_2(OPr^i)_4(acac)_2]$ (78.3)	32.31 (33.95)	18.66 (19.71)	12.36 (13.52)
3.	$[BaO_2Ti_2(OPr^i)_6]$ 0.297 (0.48)	Hacac 0.144 (1.44)	1:3	10½	$[BaO_2Ti_2(OPr^i)_3(acac)_3]$ (80.2)	23.52 (24.08)	17.52 (18.63)	13.25 (12.78)
4.	$[BaO_2Ti_2(OPr^i)_6]$ 0.287 (0.46)	Hacac 0.186 (1.86)	1:4	11	$[BaO_2Ti_2(OPr^i)_2(acac)_4]$ (79.2)	14.23 (15.22)	15.89 (17.67)	12.01 (12.12)
5.	$[BaO_2Ti_2(OPr^i)_6]$ 0.389 (0.63)	Hbzac 0.102 (0.63)	1:1	7	$[BaO_2Ti_2(OPr^i)_5(bzac)]$ (75.8)	40.15 (41.14)	18.56 (19.10)	12.35 (13.11)
6.	$[BaO_2Ti_2(OPr^i)_6]$ 0.343 (0.55)	Hbzac 0.179 (1.11)	1:2	8	$[BaO_2Ti_2(OPr^i)_4(bzac)_2]$ (73.8)	27.23 (28.81)	15.32 (16.72)	10.87 (11.47)

7.	$[BaO_2Ti_2(OPr^i)_6]$	Hbzac	1:3	9½	$[BaO_2Ti_2(OPr^i)_3(bzac)_3]$	18.96	14.21	09.85
	0.286 (0.465)	0.226 (1.39)			(73.4)	(19.21)	(14.87)	(10.20)
8.	$[BaO_2Ti_2(OPr^i)_6]$	Hbzac	1:4	11½	$[BaO_2Ti_2(OPr^i)_2(bzac)_4]$	11.50	12.80	08.90
	0.265 (0.43)	0.279 (1.72)			(74.5)	(11.53)	(13.39)	(09.18)

TABLE 2 – Analytical and physical data of the studied compounds

S.No.	Compound	Empirical formula	Formula weight	Anal. Found (calcd) %		
				C	H	O
1.	$[BaO_2Ti_2(OPr^i)_6]$	$C_{18}H_{42}BaTi_2O_8$	615	33.95	6.58	19.85
				(35.12)	(6.83)	(20.81)
2.	$[BaO_2Ti_2(OPr^i)_5(acac)]$	$C_{20}H_{42}BaTi_2O_9$	655	34.99	6.11	20.12
				(36.64)	(6.41)	(21.98)
3.	$[BaO_2Ti_2(OPr^i)_4(acac)_2]$	$C_{22}H_{42}BaTi_2O_{10}$	695	37.05	6.14	22.21
				(37.98)	(6.04)	(23.02)
4.	$[BaO_2Ti_2(OPr^i)_3(acac)_3]$	$C_{24}H_{42}BaTi_2O_{11}$	735	38.12	5.32	23.25
				(39.18)	(5.71)	(23.94)
5.	$[BaO_2Ti_2(OPr^i)_2(acac)_4]$	$C_{26}H_{42}BaTi_2O_{12}$	775	38.91	5.23	23.54
				(40.25)	(5.41)	(24.77)
6.	$[BaO_2Ti_2(OPr^i)_5(bzac)]$	$C_{25}H_{44}BaTi_2O_9$	717	40.75	5.83	19.28
				(41.84)	(6.14)	(20.08)
7.	$[BaO_2Ti_2(OPr^i)_4(bzac)_2]$	$C_{32}H_{46}BaTi_2O_{10}$	819	45.32	5.58	19.22
				(46.88)	(5.61)	(19.53)
8.	$[BaO_2Ti_2(OPr^i)_3(bzac_{)3}]$	$C_{39}H_{48}BaTi_2O_{11}$	921	51.32	5.43	19.47
				(50.81)	(5.21)	(19.1)
9.	$[BaO_2Ti_2(OPr^i)_2(bzac)_4]$	$C_{46}H_{50}BaTi_2O_{12}$	1023	52.45	4.44	18.56
				(53.95)	(4.88)	(18.76)

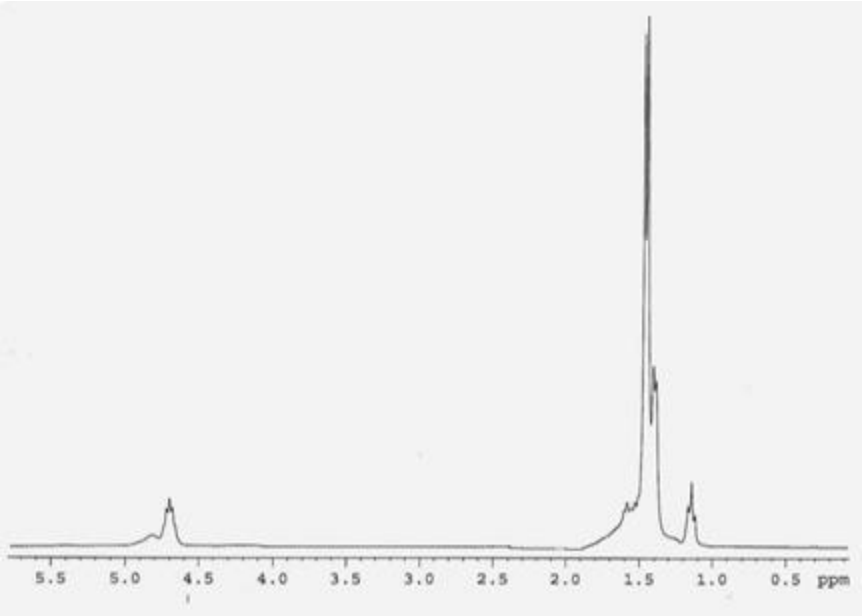

Figure 1. ^{1}H NMR spectrum of $[BaO_2Ti_2(OPr^i)_6]$

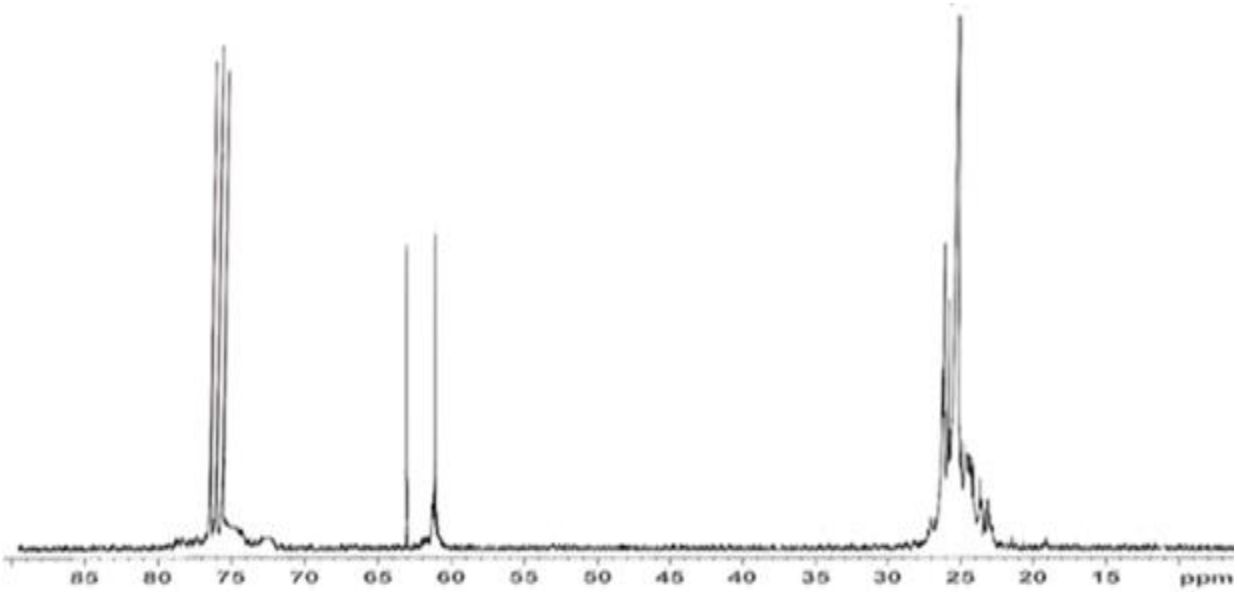

Figure 2. ^{13}C NMR spectrum of $[BaO_2Ti_2(OPr^i)_6]$

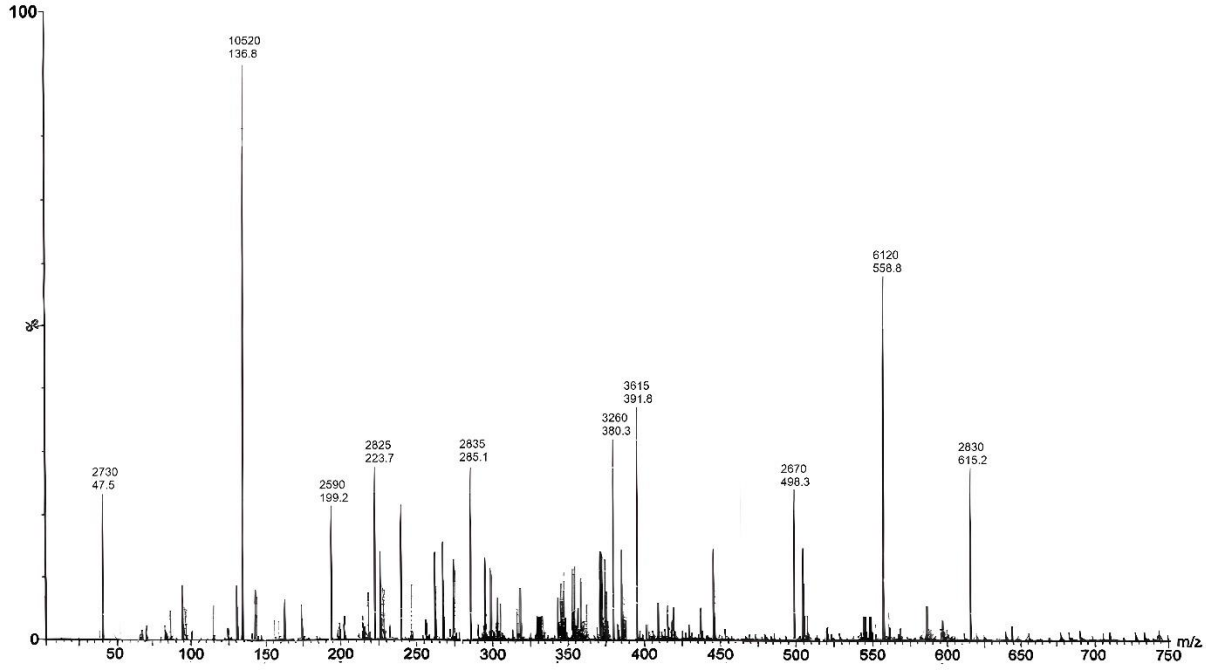

Figure 3. Mass spectrum of $[BaO_2Ti_2(OPr^i)_6]$

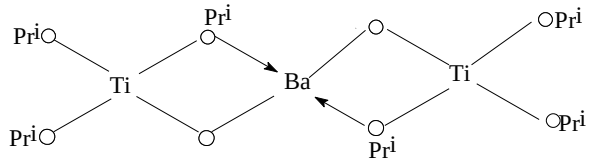

Figure 4: Suggested structure of $[BaO_2Ti_2(OPr^i)_6]$

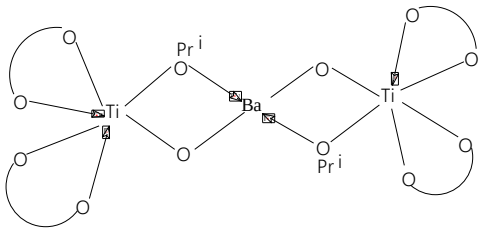

Figure 4: Suggested structure of $[BaO_2Ti_2(OPr^i)_2L_4]$