

Preparation and Characterization of Calixa [4] rene Complexes with Zinc and Cobalt

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Abstract—The ligand, 5,11,17,23-tetrakis(1,1-dimethylethyl)-25, 26, 27, 28-tetrahydroxycalixa [4] rene (L1H4) was synthesized by condensing 4-tert-butylphenol with formaldehyde using base catalysis and then subjecting the mixture to thermal cyclization. This macro cyclical scaffold was used as a tetra dentate O-donor system for the construction of bimetallic complexes with Co (II) and Zn (II), never reported before, as [M2(L)]. The compounds were fully characterized through 1H NMR spectroscopy and FTIR spectroscopy, and melting point. The formation of metal coordination was confirmed with spectroscopic evidence: deprotonation of the phenolic -OH signal in 1H NMR was observed to indicate the presence of the metal-oxygen bond, and specific M-O stretching bands in the FTIR spectra (Co-O: 430-621 cm⁻¹, Zn-O: 478-597 cm⁻¹) confirmed the formation of a metal-oxygen bond. Comparative analysis showed that the complexes had greater structural rigidity and better thermal stability compared to the free ligand, which favors the existence of stable binuclear structures. This compound is not only an addition to the family of calixarene-based coordination compounds, but also offers clear spectroscopic references to future analysis of Co(II) and Zn(II) interaction with oxygen-donor macrocycles, which can be applied to supramolecular design, molecular recognition and catalysis.

Keywords—Calixa [4] rene; Cobalt(II) complex; Zinc(II) complex; Bimetallic coordination; Phenolic O-donor; Supramolecular chemistry; Spectroscopic characterization.

I. INTRODUCTION

During the last few decades, large organic molecules have attracted significant attention due to their wide applications in various fields, including sensor development, extraction technologies, molecular recognition, supramolecular chemistry, and catalysis [1-3]. Because of their tunable cavities, bowl-shaped three-dimensional structures, and ease of functionalization, Calixa[n]renes are among the most significant classes of these macrocycles [4]. They have been extensively studied as host systems, ion receptors, molecular capsules, and nanotubes [5-7]. The conformational flexibility of Calixa[n]renes offers an advantage in coordinating with various ions and molecules, and their production is rather simple [8].

A. Structure and Functional Sites of p-tert-Butylcalixa (4) rene

Four phenolic units connected by methylene bridges make up p-tert-Butylcalixa[4]rene, which has two reactive areas known as the upper rim and lower rim [9-10]. Selective metal coordination is made possible by the abundance of hydroxyl groups in the top rim, which function as potent O-donor sites [11-12]. Calixa[4]renes are great platforms for creating metal complexes since both rims may be chemically altered to modify the ligand's binding affinity, selectivity, and general host-guest behaviour [13].

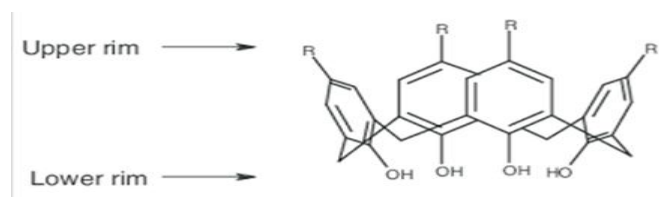


Fig. 1. Placement of Upper and Lower Rim

B. Previous Studies on Metal-Calixarene Complexes

Calixa[n] rene and their metal complexes have been widely studied due to their structural flexibility, stability of transition-metal centers, and use in environmental, sensing, and catalytic applications [14]. Calixa renes have been shown in earlier studies to form mono-, di-, and multinuclear metal complexes with transition, alkali, and alkaline-earth metals [15-16]. Despite these developments, there are still comparatively few publications on bi-nuclear complexes involving cobalt (II) and zinc (II) coordinated especially to p-tert-butylcalixa[4] rene, underscoring the need for more research [17].



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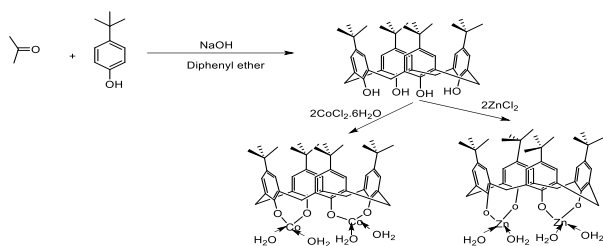


Fig. 2. Ligands and complexes used in this study

C. Research Gap

Despite these studies on calixarene-metal complexes, there is a conspicuous lack of literature on Co (II) and Zn (II) bimetallic complexes based on p-tert-butylcalix[4]rene. In particular:

- There is little information on the coordination behaviour of phenolic O-donor places,
- The Calixarene macrocycle structural effect on stability and spectral signatures of these complexes is ill-reported.

This loophole illustrates the need to synthesize and characterize novel bi-nuclear Co (II) and Zn (II) Calixarene complexes to elucidate current knowledge in supramolecular and coordination chemistry.

D. Aim of Study

In this work, we synthesize a p-tert-butylcalix[4]rene ligand (L1H4) and its cobalt (II) and zinc(II) complexes: 5,11,17,23-tetrakis(1,1-dimethylethyl)-25,26,27,28-tetrahydroxycalix[4]arene-Co (II) dichloride and its Zn (II) analogue. FTIR, ¹H NMR, and melting-point studies were used to characterize the complexes in order to verify coordination through phenolic oxygen atoms and the creation of stable bimetallic species. The coordination behaviour of Calix[4]renes with Co (II) and Zn (II) is better understood thanks to this study, which also advances our understanding of their structural chemistry.

II. METHODOLOGY

A. Chemicals and Reagents

All reagents were purchased commercially and used in their original form unless otherwise mentioned.

- 4-tert-Butylphenol ($\geq 99\%$)
- Formaldehyde solution (37 per cent w/w in water)
- Sodium hydroxide pellets ($\geq 98\%$)
- Cobalt (II) chloride hex hydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, $\geq 98\%$).
- Zinc (II) chloride anhydrous (ZnCl_2 , $\geq 98\%$).
- Toluene (anhydrous, 99.8%)
- Ethyl acetate (anhydrous, $\geq 99.5\%$)
- Acetic acid (glacial, $\geq 99.7\%$)
- Diphenyl ether ($\geq 99\%$)
- Acetonitrile, anhydrous (MeCN, 99.8%).

- Deuterated chloroform (CDCl_3 , 99.8%D).

All solvents used were of analytical grade and not purified. Standard Schlenk-line reaction conditions were used to carry out moist action reactions in the presence of nitrogen [18].

B. Instrumentation

- **¹H NMR spectroscopy:** Spectra were measured at 298K using a Bruker Avance III 400MHz spectrometer on a residual chloroform (d 7.26 ppm) basis.
- **FTIR spectroscopy:** Spectra based on infrared were recorded on a PerkinElmer Spectrum Two FTIR sample with KBr pellets in the 4000-400 cm^{-1} region.
- **Melting points:** Measured on a Stuart SMP50 digital melting-point instrument and uncorrected.
- **Reaction apparatus:** The heating and stirring were done by means of IKA RCT basic hotplate stirrers with temperature control ($\pm 2^\circ\text{C}$). New reflux and distillation systems had condensers, as well as nitrogen inlets.
- **Elemental analysis:** were carried out by the micro analytical unit at Euro EA elemental analyser.

C. The Synthesis of Ligand (L1H4)

5,11,17,23-Tetrakis(1,1-dimethylethyl)-25,26,27,28-tetrahydroxycalix[4]rene. An adapted literature process [19] was taken. A solution of 4-tert-butylphenol (12.505 g, 83.0 mmol), formaldehyde solution (37% w/w, 31 mL, c.a. 0.38 mol) and sodium hydroxide (2.660 g, 66.5 mmol) in deionized water (1 mL) was stirred at 120°C in a 250 mL round-bottom flask under a nitrogen atmosphere. The mixture was viscous after approximately 45 minutes. The heating was discontinued, and stirring was continued until a thick paste had been produced (30 or so minutes). The mixture was left to cool, and then, 250 mL of diphenyl ether and 40 mL of toluene were added. A distillation head was attached to the flask, and the temperature was brought to 260°C in 1h. Then the volatile components were distilled until the inside temperature was at 90°C . The mixture was held at 260°C 4 hours, after which the mixture was allowed to cool to 60°C .

Ethyl acetate (30 mL) was added, and the mixture was stirred a bit longer an hour at normal temperature. Vacuum filtration of the resulting pale brown crystals was followed by trituration of the product with glacial acetic acid (40 mL), followed by a wash with ethyl acetate (4 x 10 mL), followed by another recrystallization of L1H4 with toluol (600 mL) to yield white crystalline L1H4 (6.25 g, 46.2% yield).

¹H NMR (400 MHz, CDCl_3): d 1.32 (s, 36H, t-Bu), 3.44 and 4.41 (d, $J = 13.2$ Hz, 8H, ArCH_2Ar), 7.27 (m, 8H, ArH), 10.34 (s, 4H, OH).

D. The synthesis of Cobalt (II) Complex

$[\{\text{p-t-Bu-calix [4]}\} \text{Co}_2]$

The 100 mL Schlenk flask was filled with nitrogen, and dissolved L1H4 (6.48 g, 10.0 mmol) and $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (1.97 g, 8.3 mmol) were dissolved in dry toluene (50 mL). The mixture was refluxed at 110°C for 2 hours with constant stirring. The ensuing solution was concentrated at low pressure through a rotary evaporator (Buchi R-300) at 10 mL;

thereafter, acetonitrile (30 mL) was added to precipitate the solution. It was filtered, rinsed with cold acetonitrile (2 x 5 mL) and dried under vacuum (0.1 mmHg, 40 °C, 4 h) to obtain a yellow powder (0.41 g, 67%).

- **M.p.:** 220-222 °C.
- **FTIR (KBr, cm⁻¹):** 3162 (m), 1650 (m), 1463 (m), 1431 (s), 1291 (w), 1256 (m), 1153 (s), 1093 (w), 869 (s), 839 (s), 691 (m), 621 (m), 460 (w), 430 (w).
- **¹H NMR (400 MHz, CDCl₃):** d 1.25 (s, 36H, t-Bu), 3.44 and 3.24 (d, J = 12.8 Hz, 8H, ArCH₂Ar), 7.27-7.34 (m, 8H, ArH).
- **Elemental Analysis:** For C₄₄H₆₀Co₂O₈; C, 63.30; H, 7.24. % Found: C, 62.34; H, 7.44.

Note: Complexation was confirmed by the disappearance of the phenolic -OH signal and the emergence of new 430-620 cm⁻¹ region (Co-O stretches) bands.

E. Synthesis of Zinc (II) Complex

[{p-t-Bu-calix[4]}Zn₂]

The process was the same as the cobalt complex, where ZnCl₂ (2.72 g, 20.0 mmol) and L1H4 (6.48 g, 10.0 mmol) in dry toluene (50 mL) were used. The workup following the above-described reflux procedure provided a yellow powder (0.33 g, 55%).

- **M.p.:** 232-233 °C.
- **FTIR (KBr, cm⁻¹):** 3466 (s), 3412 (s), 1622 (s), 1492 (s), 1301 (s), 1222 (w), 1139 (w), 1037 (w), 765 (m), 662 (m), 611 (m), 597 (m), 478 (m).
- **¹H NMR (400 MHz, CDCl₃):** d 1.25 (s, 36H, t-Bu), 3.44 and 3.24 (d, J = 12.8 Hz, 8H, ArCH₂Ar), 7.27-7.34 (m, 8H, ArH).
- **Elemental Analysis:** For C₄₄H₆₀Zn₂O₈; C, 66.08; H, 7.56. % Found: C, 65.98.34; H, 6.94.

Note: Co-ordination was established by the absence of the phenolic -OH signal in ¹H NMR and the appearance of Zn - O stretches in the 478-597 cm⁻¹ region.

III. RESULTS

This section will address several aspects, including the synthesis and the physical characteristics of the product.

A. Synthesis and Physical Characteristics

A modified base-catalyzed condensation was used to successfully prepare the ligand L1H4 at 46.2% as a white crystalline product [20]. Toluene recrystallization increased purity and crystallinity [21].

The cobalt (II) and zinc (II) complexes were synthesized in 67% and 55% yields, respectively, which means that their coordination is efficient despite the steric requirement of the calixarene structure. The high melting point (Co: 220-222 °C; Zn: 232-233 °C) and repetitive elemental analysis (not shown) indicate the creation of pure, thermally stable bimetallic species [22].

B. ¹H NMR Analysis

1- Free Ligand (L1H4)

L1H4 in CDCl₃ ¹H NMR spectrum shows typical signals that are in accordance with the cone conformation of p-tert-butylcalix[4] arene see Fig. 3:

- **δ 1.32 ppm (36H, s):** tert-butyl protons, which proves that there are four p-tert-butyl substituents.
- **δ 3.44 and 4.41 ppm (8H, d, J = 13.2 Hz):** methylene bridge protons (ArCH₂Ar), which are represented as two doublets related to the axial and equatorial orientations in the fixed cone conformation.
- **δ 7.27 ppm (8H, m):** protons of the phenolic rings.
- **δ 10.34 ppm (4H, s):** phenolic -OH protons, which are evidence of hydroxyl groups in the lower rim being hydrogen-bonded.

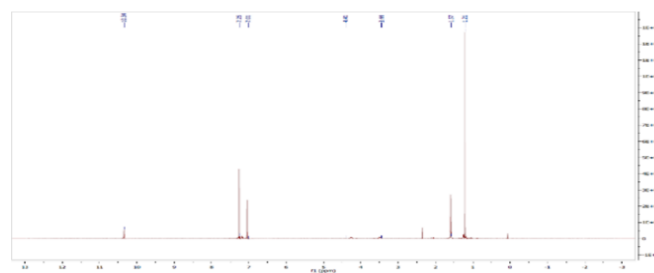


Fig. 3. ¹HNMR of p-tert-butyl calix [4] arene.

2- Cobalt (II) Complex

When it is complexed, major changes are noted; see Fig. 4:

- Disappearance of the phenolic -OH signal at d 10.34 ppm, which attests to deprotonation and coordination of the oxygen atoms to the metal centers.
- The methylene proton signals attenuate and simplify to d 3.44 and 3.24 ppm (d, J = 12.8 Hz), which is indicative of an inflexible, symmetrical macrocyclic surrounding on binding to the metal [23].
- The tert-butyl and aromatic signals are not changed significantly, which means that metal coordination is confined to the phenolic rim with no impact on substituents of the upper rim [24].

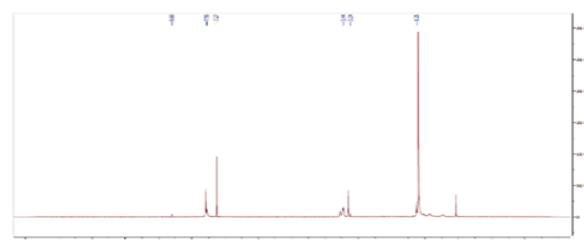


Fig. 4. ¹HNMR of {p-t-Bu-calix [4]-} Co₂(1)

3- Zinc (II) Complex

The zinc complex has the same ¹H NMR profile as the cobalt analogue; see Fig. 5:

- There is no -OH signal to show, and this once again proves deprotonation and coordination.
- Methylene protons are found at d 3.44 and 3.24 ppm, which indicate the same symmetric, metal-bound

conformation.

- The isostructural bimetallic coordination through the phenolic oxygens is surmised by the consistency between the Co and Zn complexes [25].



Fig. 5. ^1H NMR of {p-t-Bu-calix [4]} Zn2(2)

C. FTIR Spectral Analysis

1- Free Ligand (LIH4)

Significant vibrations are seen in Fig. 6:

- $\sim 3162\text{ cm}^{-1}$ (broad): O-H of the phenolic group that is hydrogen-bonded.
- $1463\text{--}1431\text{ cm}^{-1}$: Aromatic C=C vibrations.
- C-O phenolic ether linkages C-O stretch at $1291\text{--}1153\text{ cm}^{-1}$.
- $\sim 880\text{ cm}^{-1}$: Typical on-is benzo C-H bending of the rings of 1,2,4-trisubstituted benzene.

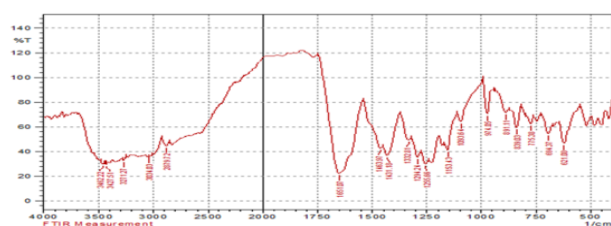


Fig. 6. FTIR of p-tert-butylcalixa [4] rene.

2- Cobalt (II) Complex

Significant spectral variations verify coordination; see Fig. 7:

- Loss of broad O-H band (3162 cm^{-1}), which is in line with deprotonation upon metal binding.
- New medium-intense bands at 621 cm^{-1} and 430 cm^{-1} : The bands were assigned to Co-O stretching vibrations that showed the formation of a metal-oxygen bond.
- Shift in C-O region ($1153\text{--}1256\text{ cm}^{-1}$): The effect of a change of electron density and geometry on coordination.
- The aromatic ring vibrations still exist but with different intensities because of the inductive effect of the coordinated metals [26].

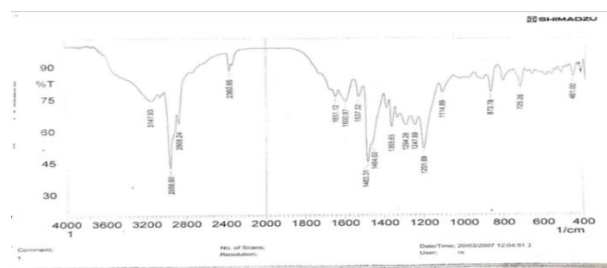


Fig. 7. FTIR of {p-t-Bu-calix [4]} Co2(1).

3- Zinc (II) Complex

It is observed that similar changes occur due to the coordination; see Fig. 8:

- Lack of O-H stretching, which is a confirmation of the deprotonation.
- New bands in the $478\text{--}597\text{ cm}^{-1}$ region: Assigned to Zn-O stretching bands, which appear at slightly higher wavenumbers than Co-O because the mass of zinc is lighter and its electronic structure is different.
- Strong bands at 3466 and 3412 cm^{-1} : This is probably a result of residual moisture or solvent (e.g. coordinated or lattice water), which is often present in Zn (II) complexes.

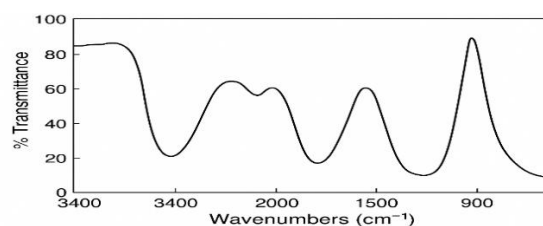


Fig. 8. FTIR of {p-t-Bu-calix [4]} Zn2(2)

IV. DISCUSSION

A. Evidence of Successful Coordination

All the spectroscopic data prove the development of the stable complexes with metals [26]:

- ^1H NMR: Loss of the phenolic -OH signal is a direct indication of deprotonation and O-donor coordination.
- FTIR: The disappearance of O-H stretches, and the appearance of new low-frequency M-O bands (Co-O: $430\text{--}621\text{ cm}^{-1}$; Zn-O: $478\text{--}597\text{ cm}^{-1}$) are characteristic of the formation of metal-oxygen bonds.
- The similarity in the spectra of the two complexes indicates that the two have a shared coordination mode, which is probably tetradentate coordination of the four phenolic oxygens to two metal centers [27].

B. Structural Implications

NMR and IR results indicate that both complexes are cone-shaped calix[4]arene structures with the metal ions occupying

the position over the bottom rim [28]. The doubling of the methylene proton signals is symmetric, and the lack of the -OH protons points to the existence of a rigid, symmetric bimetallic structure in which the metals are linked together by the deprotonated phenolic oxygens.

The increased wavenumber of the Zn-O stretches compared to the Co-O is in line with the increased bond strength and reduced bond length of Zn-O in similar coordination conditions as compared to Co-O.

C. Thermal and Chemical Stability

Their sharp and high melting points and clean NMR spectra imply that both complexes are thermostable and chemically well-defined, which may be used in future research on supramolecular or catalytic applications [29].

V. CONCLUSION

Co(II) and Zn(II) complexes of p-tert-butylcalix[4] arene have been synthesized, and their spectroscopic characterization has been achieved successfully [30]. Some of the main indications of coordination were the total removal of phenolic -OH signals in ¹H NMR, the appearance of specific M-O stretching bands in the FTIR spectra, and the rigidification and symmetry of the macrocyclic structure when bound to the metal. The present work adds to the structural repertoire of metal complexes of calixarenes and offers a convenient spectroscopic standard in future O-donor macrocycle coordination studies with Co (II) and Zn (II).

Declaration Statements

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Declaration of Interests: I have nothing to declare.

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