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SYNTHESIS AND CHARACTERIZATION OF [16]- MEMBERED DIOXA DIAZA NAPHTHALDEHYDE BASED MACROCYCLIC LIGAND AND ITS COMPLEXES OF Co(III), Ni(II) AND Cu(II) PERCHLORATE IONS

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Synthesis and characterization of dioxadiaz macrocyclic ligand and its transition metal complexes, dioxadiaz macrocycles, transition metal complexes, macrocyclic ligand.

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ABSTRACT: An hitherto new unreported [16]-membered dioxadiazanaphthaldehyde based macrocyclic ligand (L) 2,11-dioxa-23,30-diaza heptacyclo [30.4.0.0^{4,9}.0^{12,21}.0^{14,19}.0^{24,29}.0^{34,39}] -tetraconta-12,14,16, 18,20,22, 24,26,28,30,32,34,36,38,40-pentadecaene, has been synthesized by two different synthetic routes each involving two stages. 2-Hydroxy-1-Naphthaldehyde in the presence of potassium carbonate was treated with α,α' -dibromo-o-xylene to yield the dial derivative(I). The dial derivative(I) was further made to undergo Schiff base condensation with 1,2-diaminobenzene to yield the bright yellow macrocycle (L) in good yield. In the second method the Schiff base condensed product Napthaloph was synthesized and allowed to undergo Williamson's condensation with α,α' -dibromo-o-xylene to yield the ligand (L). The neutral sixteen membered tetradentate dioxadiaz ligand(L) readily complexes with Co(III), Ni(II) and Cu(II) perchlorate salts in 1:1 mole ratio to yield complexes of formulae [Co(L)X₂]ClO₄, [Ni(L)X₂], [Cu(L)X]ClO₄, (X = Cl⁻, Br⁻ and NO₃⁻). The complexes were also synthesized by the metal template method. The yield of the template procedure was found to be greater than the non-template method. The ligand and the complexes were characterized by elemental analysis, electronic spectroscopy, IR, Conductivity measurements, EPR, magnetic susceptibility, ¹HNMR, FAB-MS and Cyclic voltammetry studies. The [16]-membered tetradentate dioxadiaz macrocycle (L) was found to accommodate Co³⁺, Ni²⁺, and Cu²⁺ ions with ease due to the presence of flexible alkyl groups. Further studies with the inner-transition metal ions will be highly informative in understanding the coordinating capabilities of lanthanides and actinides.

INTRODUCTION: The design and synthesis of macrocyclic complexes^{1,2} initially has lured many a scientist owing to their significance in serving as biological mimics³⁻⁷.

The synthetic macrocyclic complexes mimic some natural occurring macrocyclic complexes⁸ resemblance with natural macrocyclic like metalloproteins, porphyrins and cobalamine^{9,10}. The transition metal macrocyclic complexes are active part of metalloenzyme proteins and coenzymes like haemoglobin, myoglobin¹¹⁻¹⁴.

Lanthanide macrocyclic complexes involving Gd and Eu are extensively used as NMR shift reagents¹⁵. Identification and realization of the applications of macrocyclic ligands and their transition and

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inner-transition metal complexes as base transfer catalystand in biological probe studies^{16, 17} have opened up new avenues of exploring their potency in various other fields recently the Napthaldehyde based Schiff bases are gaining significance due to their sequestrating properties towards transition metals^{18, 19}. The presence of azomethine group has been reported to possess remarkable antibacterial, antifungal, anticancer and antimalarial activities^{20, 21}. In order to understand the involvement of azomethine groups in the above said properties, we have reported the synthesis and spectral characterization of hitherto unreported new 16-membered naphthaldehyde based dioxadiaza tetradentate macrocyclic ligand (**L**), and its Co(III), Ni(II) and Cu(II) perchlorate complexes.

MATERIALS AND METHODS:

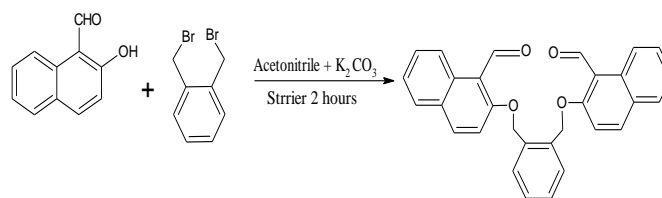
All the chemicals used in this study were of Analar grade. Metal salts were purchased from Merck and were used as such as received. Elemental analysis carried out on Elementar vario EL III-Germany. ¹H NMR spectrum was recorded used to Bruker model 4276. FT IR spectra recorded on Perkin Elmer on KBr pellets in the range of 4000 to 400cm⁻¹. The electronic spectrum recorded on Lambda 35 in the range of 200 to 800 nm using 10⁻³M concentration solution in DMF. NMR spectrum was recorded using Bruker model 4276. The new dialdehyde was synthesis from 2-hydroxyl-1-naphthaldehyde and α, α -dibromo-o-xylene in basic medium using potassium carbonate under reported procedure²².

Synthesis of dialdehyde:

To a solution of (1g, 5 mmol) of 2-hydroxy-1-naphthaldehyde in 15 mL of acetonitrile was added (0.345g, 2.5 mmol) of K₂CO₃ and the resulting solution was stirred for 30 minutes under thermostat condition at 60°C wherein a bright yellow colour solution was obtained. To this stirring solution was added a solution of (0.66g, 2.5 mmol) of α, α' -dibromo-o-xylene in 5 ml of acetonitrile in one lot. The resulting pale yellow solution was stirred until the solution decolorized and on addition of 20 mL of cold water it yielded a spongy white dialdehyde product. The spongy product was filtered using sintered crucible. Yield is 73%, m.pt 165°C. The synthesis of the ligand (**L**) is depicted in **Scheme.1** Anal.Cal. (C₃₀H₂₄O₄), C(33.37), H(5.05), N(5.40), O(56.08) Found:

C(33.31), H(5.02), N(5.37), O(56.04). IR (KBr Pellets) (C=O) 1664cm⁻¹, (C=C)_{aro} 1575cm⁻¹, (C-O) 1237cm⁻¹.

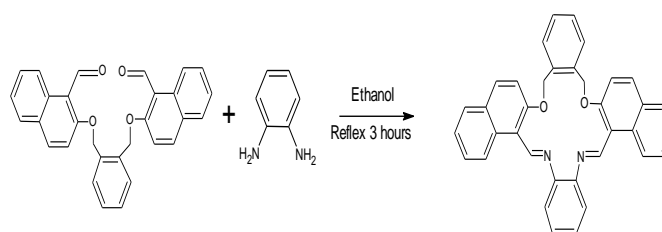
Scheme.1



Synthesis of macrocyclic ligand (**L**):

To a ethanolic solution of (1g, 0.4mmol) of the dialdehyde in 10ml of ethanol was added a solution 1,2-diamino benzene (0.8g, 0.4mmol) in 10 mL of ethanol solution under gradual addition in drops using a dropping funnel over 30 mins. The resulting pale yellow turbid solution was refluxed for 4 h wherein a clear bright yellow solution was obtained, on standing the solution to room temperature a bright yellow colored product separated out. Yield: 88% m.pt:177°C. Anal.Cal. (C₃₆H₂₆N₂O₂), C(83.37), H(5.05), N(5.40), O(6.17). IR (KBr Pellets) (C=N) 1611cm⁻¹, (C=C)_{aro} 1488cm⁻¹, (C-O) 1274cm⁻¹. The synthesis of the overall ligand is depicted in **Scheme 2**.

Scheme 2:



2,11-dioxa-23,30-diaza heptacyclo [30.4.0.0^{4,9}.0^{12,21}.0^{14,19}.0^{24,29}.0^{34,39}] - tetraconta -12,14,16,18, 20, 22, 24,26,28,30,32,34,36,38,40-pentadecaene.

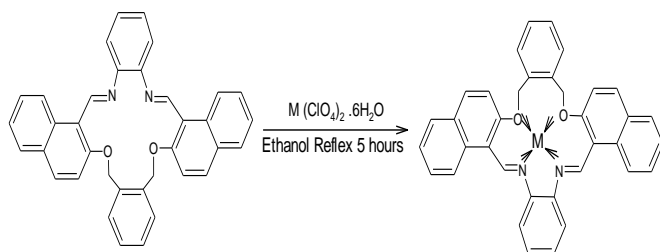
Synthesis of complexes:

General procedure for the synthesis of metal complexes of ligand (**L**):

To a refluxing solution of a ligand (10 mmol) in 20 mL of absolute alcohol was added an equivalent mole ratio of the corresponding perchlorate salts M(ClO₄) 6H₂O (10 mmol) where in (M= Co, Ni, Cu). To this refluxing metal perchlorate solution of the ligand (**L**) was added the corresponding axial monoanionic ligand X in appropriate mole ratio, (X= Br⁻, Cl⁻ and NO₃⁻) on continuing the reflux for

30 minutes yielded macrocyclic complexes in good yield. The complexes of varying colour and nature were isolated from the mother liquor owing to the sparingly soluble nature. The complexes were filtered and washed repeatedly with diethyl ether and dried over anhydrous CaCl_2 under vacuum condition. No further purification carried out as the micro crystalline colored complexes separated readily, in pure condition. The **Scheme 3** depicted the nature of bonding exhibited by the ligand.

Scheme 3:



RESULT AND DISCUSSION:

Synthesis and Characterization of the ligand(L):

The dioxadiazanaphthaldehyde based tetradentate macrocyclic was synthesized by two different methods. The first method involves the Schiff's base condensation of the naphthaldehyde and *o*-phenylenediamine in 2:1 mole ratio in acetonitrile, to yield the Schiff's base condensed product (Naphthaloph). The azomethine derivative was further reacted with α,α' -dibromo-*o*-xylene under Williamsons condensation in 1:1 mole ratio to yield the 16-membered N_2O_2 macrocyclic ligand(L).

The second method involves the Williamsons' condensation reaction of naphthaldehyde and α,α' -dibromo-*o*-xylene in 2:1 mole ratio to yield the dial derivative(I), further the dial derivative is made to undergo schiff's base condensation with 1,2-diamino benzene to yield the 16-membered macrocycle(L). The ligand isolated readily from the solution due to its sparingly soluble nature. The tetradentate dioxadiaz macrocyclic ligand(L), was characterized by elemental analysis, electronic spectroscopy, IR, ^1H , ^{13}C NMR, CIMS and Cyclic voltammetric studies. The elemental analysis was in good agreement with the experimental and theoretical value, which ensures the formation of the macrocyclic ligand. The presence of aldehyde

peak at 1664 cm^{-1} in the dial derivative(I) and the absence of the sharp $\nu(\text{OH})$ peak at 3404 cm^{-1} endures the formation of the dial intermediate (I)^{22, 23}. The infrared spectrum of the intermediate (I) and Ligand (L) is depicted in **Fig.1** and **Fig.2**.

The disappearance of the $\nu(\text{C}=\text{O})$ stretching frequency at 1664 cm^{-1} and the appearance of new peak at 1611 cm^{-1} has been assigned to $\nu(\text{C}=\text{N})$ stretching frequency²⁴. This confirms the formation of the macrocyclic ligand. The ^1H NMR spectrum of the dial derivative (I) exhibits a sharp singlet at 11 ppm assigned to the aldehydic protons, the multiplet centered around 7.2 to 8.1 ppm has been attributed to the aromatic protons from both naphthalene and benzene moieties. The four proton sharp singlet appearing at 5.4 ppm has been assigned to the methylene protons. The ^1H NMR spectrum of the intermediate (I) are depicted in **Fig.3**. The ^{13}C NMR of dial derivative (I) exhibits a sharp peak at 78 ppm with 70% intensity which has been assigned to the methylenic carbons, the sharp peak of weak intensity observed at 192 ppm has been assigned to aldehydic carbon and the medium intensity peaks centered around 112-138 ppm has been attributed to the aromatic carbon atoms pertaining to benzene and naphthalene moieties²⁵. The ^{13}C NMR spectrum of the intermediate (I) are depicted in **Fig.4**

The ^1H NMR spectrum of the ligand (L) exhibits a multiplet around 7.1 ppm to 8.1 ppm assignable to the aromatic protons of Naphthaldehyde and benzene groups, the singlet of medium intensity occurring at 9.4 ppm has been assigned to azomethine protons in the macrocycle. The unsymmetrical triplet occurring around 1.6 to 2.4 has been assigned to methylene protons. The singlet peak of medium intensity occurring at 15.1 ppm may be assigned to the hydrogen bond that can be formed due to a transfer of proton from the macrocycle moiety that can form hydrogen bond between the two oxa donors in the macrocycle. The ^1H NMR spectrum of the Ligand(L) is depicted in **Fig.5**. The mass spectrum of the dial derivative (I) exhibited the molecular base peak at 443 m/z of low intensity confirming the formation of the dioxadiaz tetra dentate macrocyclic ligand. The chemical ionization mass spectrum of the ligand exhibited a $[\text{M}+\text{H}]^+$ molecular ion peak at 519 m/e

value. The mass spectrum of the ligand (**L**) is depicted in **Fig.6**. The CHN analysis of the ligand is in good agreement of the observed value.

Characterization of the Co(III), Ni(II) and Cu(II) complexes of the ligand (**L**):

The perchlorate complexes of the macrocyclic ligand (**L**) were synthesized both by the reaction of the preformed ligand and metal perchlorate in 1:1 mole ratio in ethanol, and by the metal template method wherein the precursors and the corresponding metal perchlorates were made to react simultaneously in one pot synthesis method. The preformed ligand and metal perchlorate yielded better results than the template procedure. The varying coloured complexes obtained were characterized using elemental analysis, molar conductance, UV-vis spectroscopy, FAB-MS magnetic susceptibility and cyclic voltammetry.

The infrared spectral data of the ligand and the complexes are tabulated in **Table 1**. On complex formation the $\nu(\text{C}=\text{N})$ peak which appears at 1611cm^{-1} in the pure ligand has been shifted towards lower frequencies which is in good agreement with the coordination of the azomethine

group with the central metal ions. The occurrence of two types of $\nu(\text{C}-\text{O})$ stretching frequencies corresponding to $\nu(\text{C}-\text{O})$ aromatic and $\nu(\text{C}-\text{O})$ aliphatic nature ensures the coordination of the two oxa groups with the central metal ions. The ligand is found to act as neutral dioxadiaza tetradentate ligand. The stretching due to $\nu(\text{C}=\text{C})$ occurs within the range of $1450\text{-}1515\text{ cm}^{-1}$ in all the complexes. All the perchlorate complexes exhibit $\nu(\text{ClO}_4)$ stretching pertaining to free ionic perchlorate groups around $1059\text{-}1085\text{ cm}^{-1}$ in all the complexes²⁶⁻²⁸. The electronic spectral data reveal that all the metal ions exist in their usual oxidation states, the cobalt(III) complexes in which cobalt acquires d^6 state and is expected to exhibit a single electronic transition, and it is observed so indicating that the cobalt ion is in 3^+ oxidation state²⁹⁻³¹.

The electronic spectral data of the complexes are tabulated in **Table 2**. The molar conductance values of the complexes reveal that the complexes exist as both neutral entities and 1:1 type of electrolytes. The physical characteristics, molar conductance values and elemental analysis data of the complexes are tabulated in **Table 3** and **Table 4**

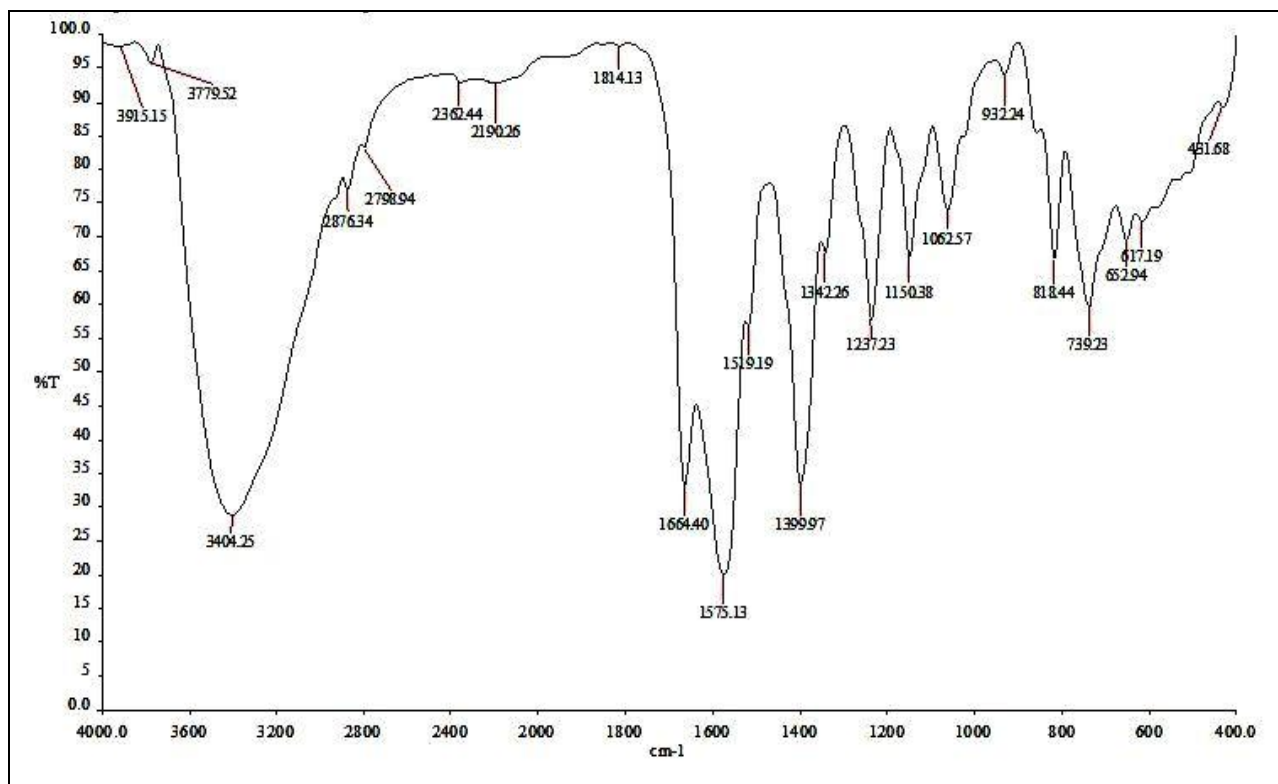


FIG.1: INFRARED SPECTRUM OF THE INTERMEDIATE (I)

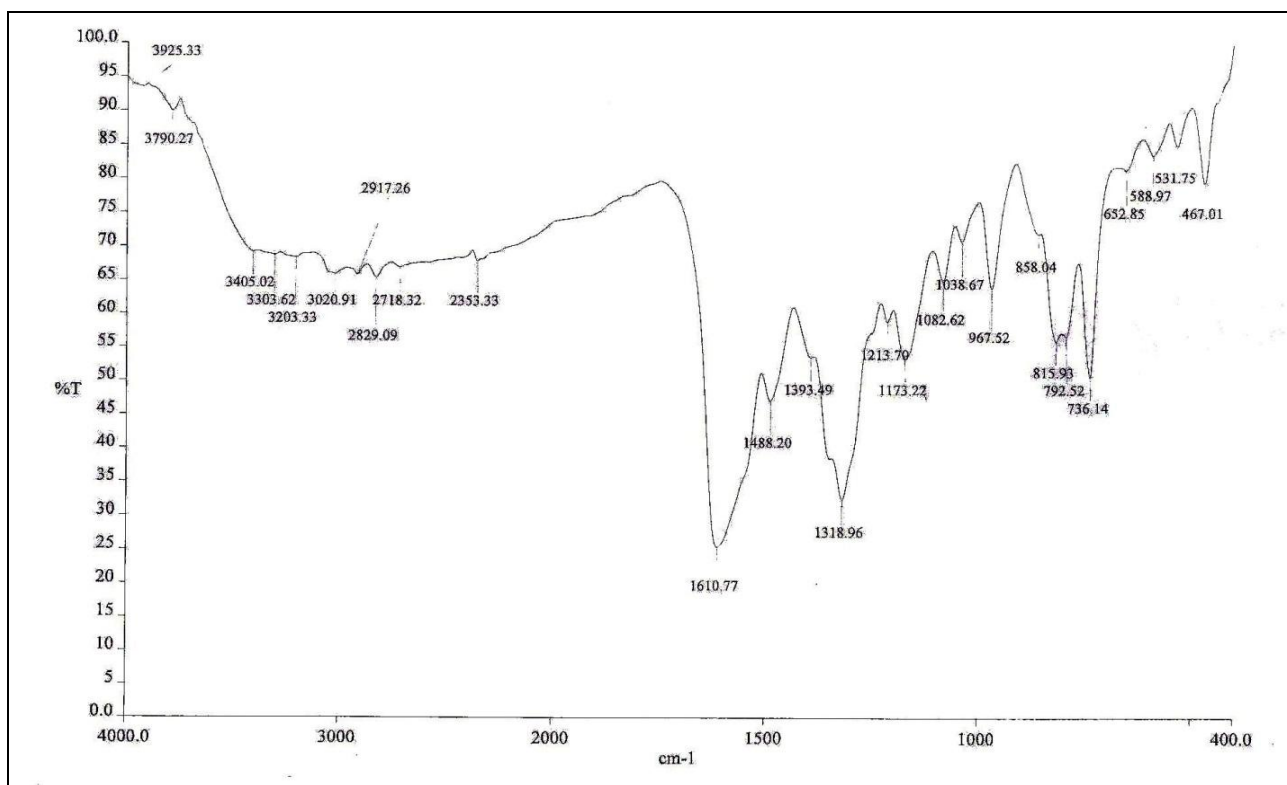


FIG.2: INFRARED SPECTRUM OF THE LIGAND(L)

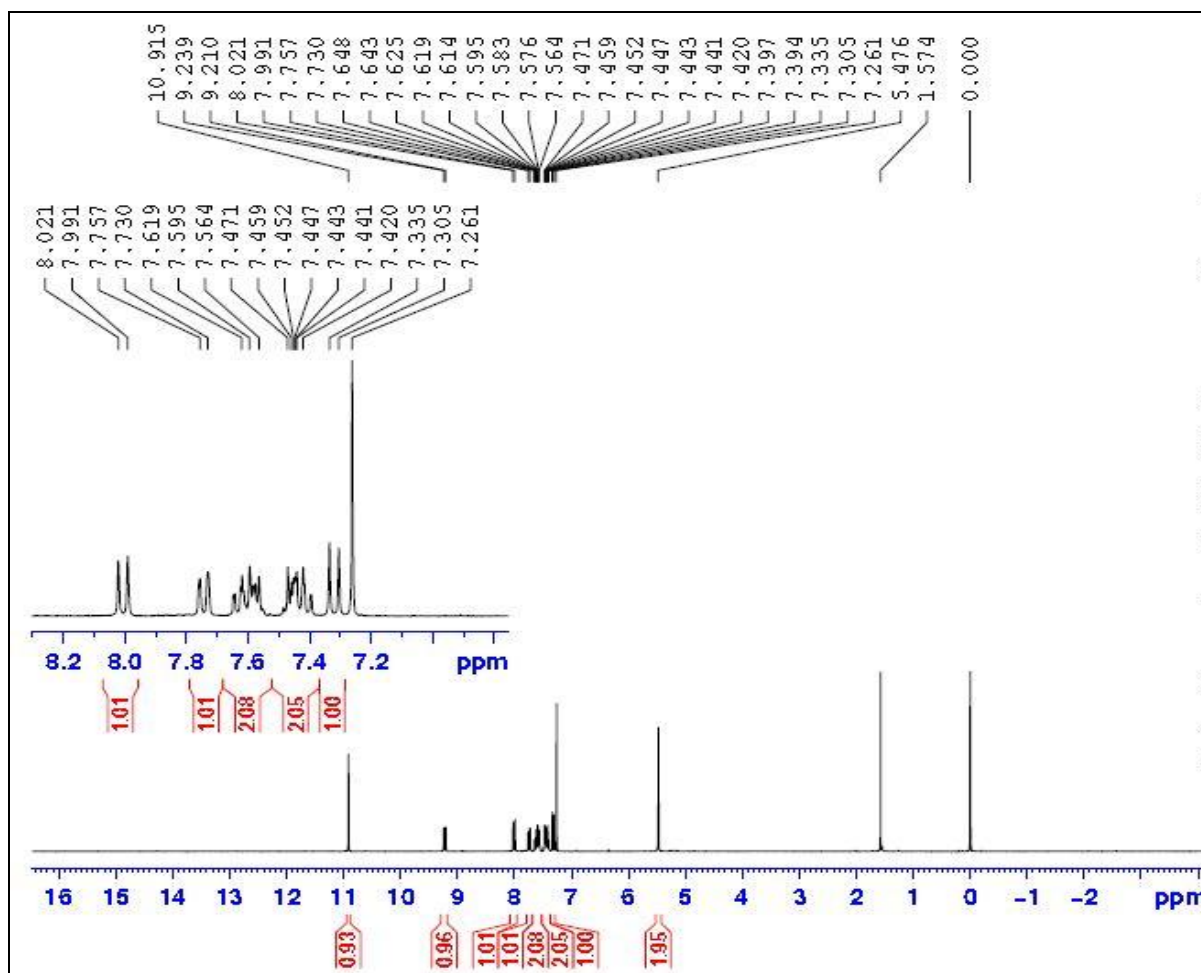


FIG. 3: ¹H NMR SPECTRUM OF THE INTERMEDIATE (I)

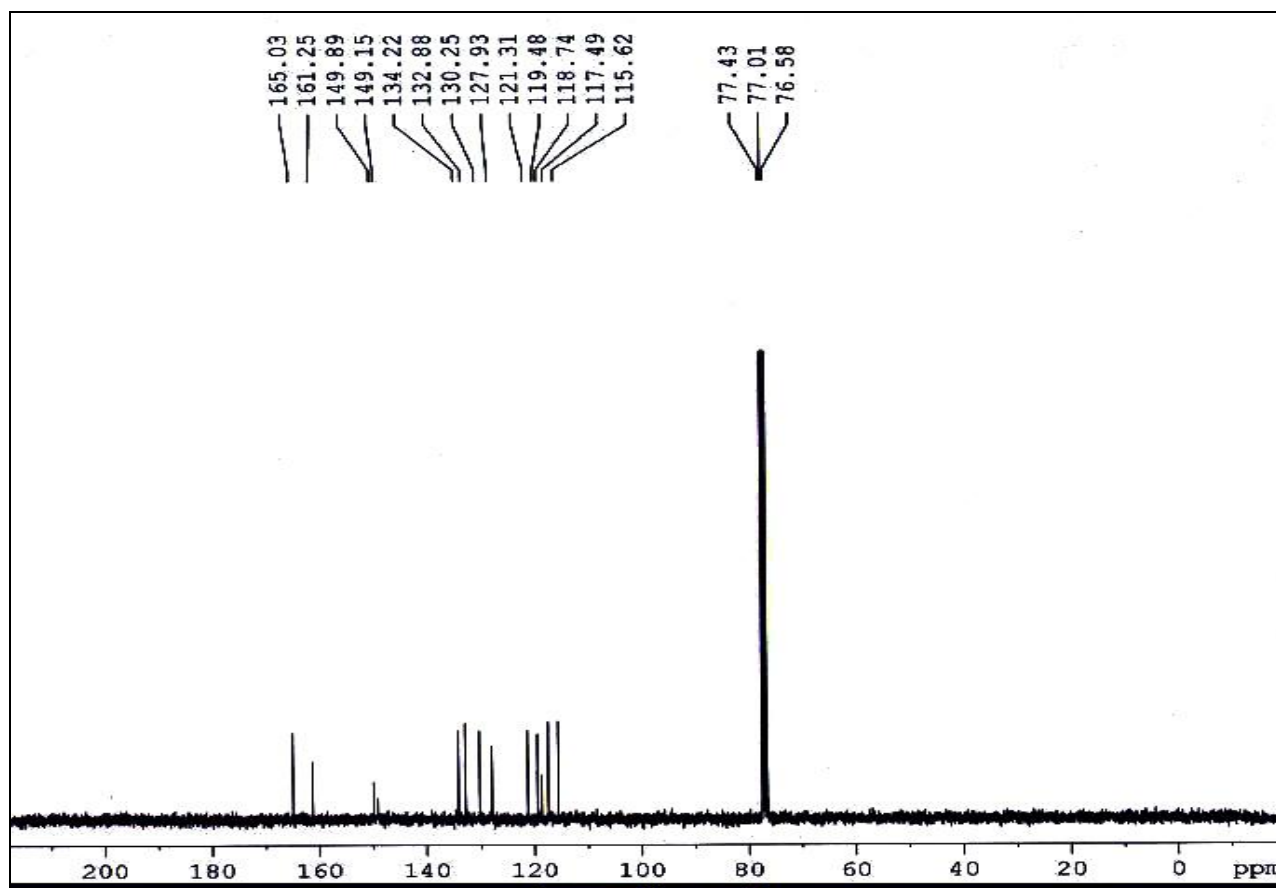


FIG. 4: ¹³C NMR SPECTRUM OF THE INTERMEDIATE (I)

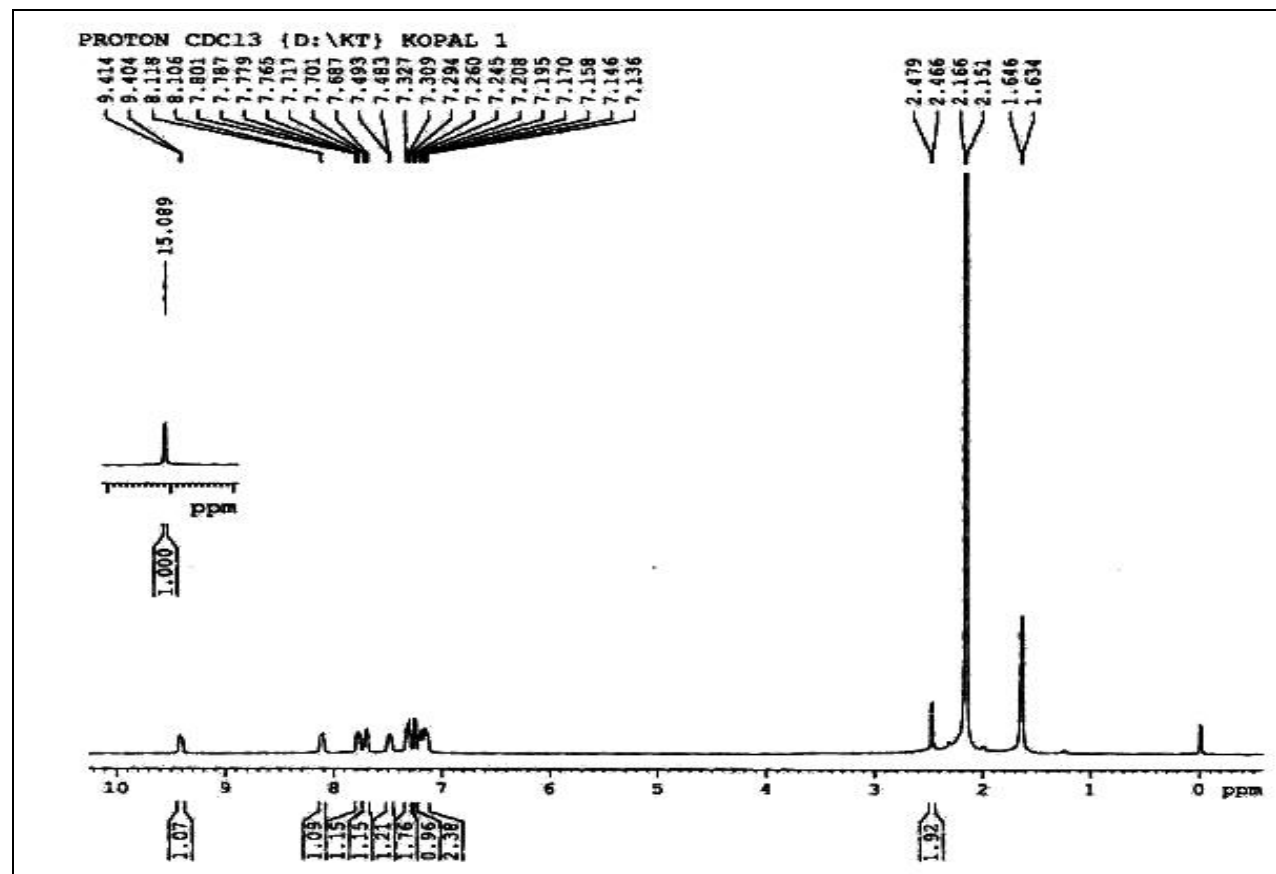


FIG.5: ¹H NMR SPECTRUM OF THE LIGAND (L)

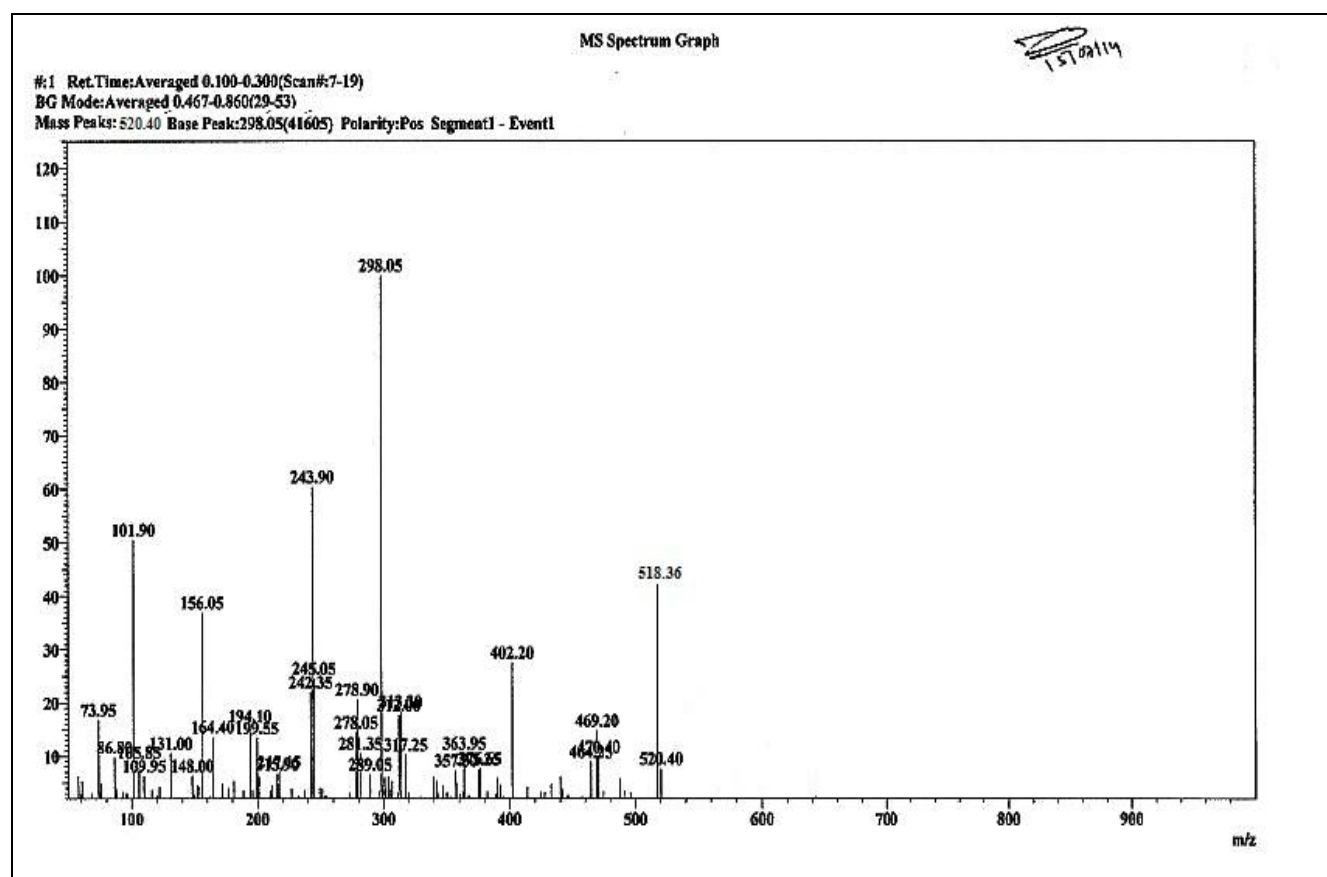


FIG.6: MASS SPECTRUM OF THE LIGAND (L)

TABLE 1: INFRARED SPECTRAL DATA OF THE LIGAND (L) AND ITS TRANSITION METAL COMPLEXES

Compound	ν (C=N) (cm^{-1})	ν (C=C) (cm^{-1})	ν (C-O) _{Aro} (cm^{-1})	ν (C-O) _{Ali} (cm^{-1})	ClO_4^-
Ligand	1611	1488	1274	1112	
[Co(L ₁)Cl ₂]	1589	1508	1271	1099	1090
[Co(L ₁)Br ₂]	1590	1508	1269	1088	1087
[Co(L ₁)(NO ₃) ₂]	1591	1507	1273	1105	1080
[Ni(L ₁)Cl ₂]	1595	1510	1270	1086	1075
[Ni(L ₁)Br ₂]	1597	1508	1268	1080	1077
[Ni(L ₁)(NO ₃) ₂]	1593	1507	1265	1083	1079
[Cu(L ₁)Cl]	1607	1529	1267	1090	1085
[Cu(L ₁)Br]	1606	1526	1272	1086	1087
[Cu(L ₁)NO ₃]	1601	1524	1268	1088	1080

TABLE 2: PHYSICAL PROPERTIES OF N – DIAL PERCHLORATE COMPLEXES

Name of the Complexes	Color	Molecular Weight	Yield %	Molar conductance ($\text{mho cm}^2 \text{mol}^{-1}$) mho	Nature of the electrolyte
[Co(L ₁)Cl ₂]	Pale Rose	725.63	73	68	1:1
[Co(L ₁)Br ₂]	Dirty White	814.53	75	72	1:1
[Co(L ₁)(NO ₃) ₂]	Pale Rose	776.73	74	74	1:1
[Ni(L ₁)Cl ₂]	Pale Rose	725.39	82	36	Neutral
[Ni(L ₁)Br ₂]	Black	746.19	84	35	Neutral
[Ni(L ₁)(NO ₃) ₂]	Black	846.29	80	37	Neutral
[Cu(L ₁)Cl]	Reddish Brown	679.75	76	70	1:1
[Cu(L ₁)Br]	Dark Brown	724.20	78	65	1:1
[Cu(L ₁)NO ₃]	Dark Brown	737.34	75	76	1:1

TABLE 3: ELECTRONIC SPECTRAL DATA OF THE LIGANDS AND ITS COMPLEXES:

Complexes	Frequency Range (cm ⁻¹)
[Co(L ₁)Cl ₂]	29850, 30769
[Co(L ₁)Br ₂]	29850
[Co(L ₁)(NO ₃) ₂]	29850, 30769
[Ni(L ₁)Cl ₂]	20661, 25906,30581
[Ni(L ₁)Br ₂]	20665, 25909,3058
[Ni(L ₁)(NO ₃) ₂]	20661, 25906,30581
[Cu(L ₁)Cl]	23529, 30211
[Cu(L ₁)Br]	23584, 30395
[Cu(L ₁)NO ₃]	23529, 30211

TABLE 4: CHN ANALYSIS:

Ligand	% of C		% of H		% of N	
	Cal value	Obs value	Cal value	Obs value	Cal value	Obs value
	80.5658	80.378	3.9675	3.934	15.2452	15.0243

CONCLUSION: The predesigned[16]-membered neutral dioxadiaza tetradentate macrocyclic ligand (L), has been achieved through, two step synthetic strategy. The conventional method and microwave assisted synthetic methods yield the ligand in good yields. The microwave assisted synthetic method, provided high yields of pure macrocycle. No further purification was required, to attain the sharp melting point. The sharp melting point and the good agreement of the elemental analysis along with the spectroscopic evidences confirms the formation the [16]-membered neutral dioxadiaza tetradentate macrocyclic ligand(L).

The dioxadiaza tetradentate macrocyclic ligand, readily complexes with the transition metal ions such as Cobalt(III), Nickel(II) and Copper (II) metal salts in ethanolic medium. The fine microcrystalline coloured complexes separated out of the reaction mixture due to its sparingly soluble nature. The complexes were characterized, without any further purification. The preformed ligand reacted with the corresponding metal salts in 1:1 mole ratio to yield complexes, the metal template method gave instant complexation but with relatively lower yields. The investigation of spectroscopic data of the metal complexes of the macrocycle revealed the formation of mononuclear complexes of molecular formula M(C₄₁H₄₃N₂O₂)X₂. The electronic spectral data along with the molar conductivity studies are in the favor of octahedral geometry of Co(III) and Ni(II) complexes and square pyramidal geometry of Cu(II) complexes. The macrocyclic cavity provided

by the [16]-membered tetradentate dioxadiaza macrocycle is tunable enough with the chosen metal ions to form stable complexes. The ligand has proven a good vehicle to study the physico chemical properties of the varying metal ions providing an identical frame work.

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