# SYNTHESIS OF SELECTED MACROCYCLIC COMPLEXES AND INVESTIGATION OF THEIR ANTIBACTERIAL AND ANTIFUNGAL ACTIVITIES

*A Dissertation*
*Submitted to the Partial Fulfilment of the Requirement for the Degree of Doctor of Philosophy in Chemistry.*

**SUBMITTED BY**

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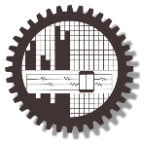

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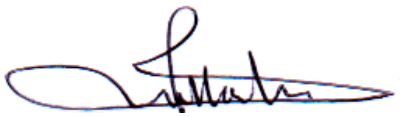

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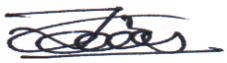

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The thesis titled "**SYNTHESIS OF SELECTED MACROCYCLIC COMPLEXES AND INVESTIGATION OF THEIR ANTIBACTERIAL AND ANTIFUNGAL ACTIVITIES**" submitted by MOHAMMAD JAKIR HOSSAIN, Roll No.0412034202F, and Session: April-2012 has been accepted as satisfactory in partial fulfillment of the requirement for the degree of Doctor of philosophy (Ph.D) in Chemistry.

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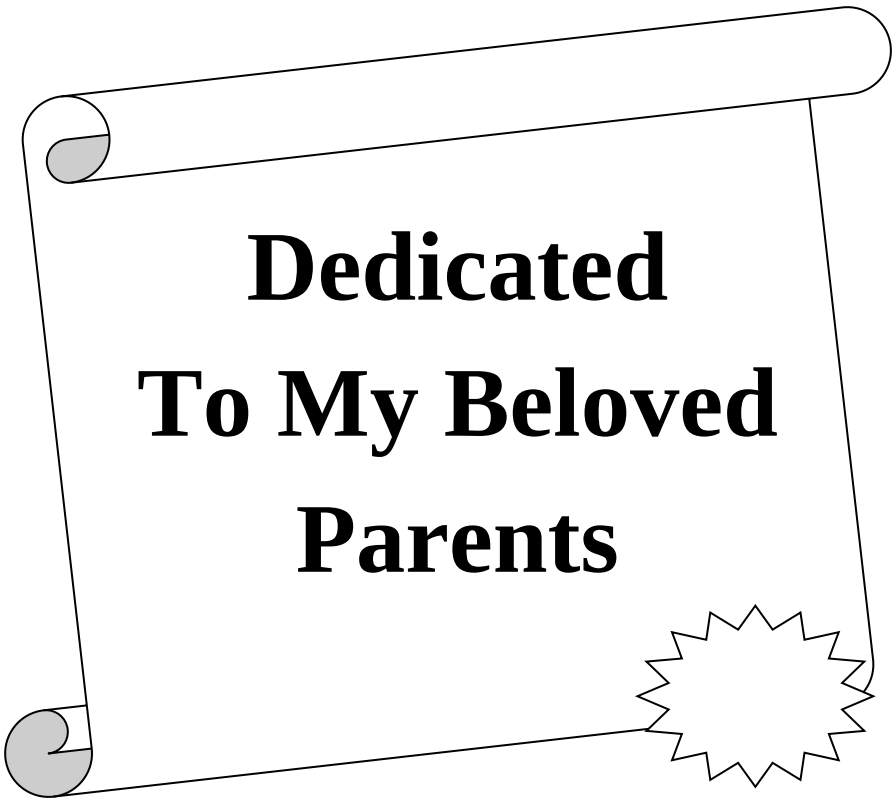
Dedicated
To My Beloved
Parents

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Author

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## BIOLOGICAL ACTIVITY



### MINIMUM INHIBITORY CONCENTRATIONS (MIC) OF COMPLEXES

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**Complex compound of Ag(II)**



**A New macrocyclic complexes of metal (II)ion**



## PHOTOGRAPHIC

## LIST OF ABBREVIATION

| | | |
|---|---|---|
| 1. | g | Gram |
| 2. | cm | Centimeter |
| 3. | $cm^{-1}$ | Per centimeter |
| 4. | Fig | Figure |
| 5. | i.e. | That is |
| 6. | K | Degree kelvin |
| 7. | m.p. | Melting point |
| 8. | mL | Milliliter |
| 9. | mmol | Millimol |
| 10. | No | Number |
| 11. | % | Percent |
| 12. | ∧ | Conductance |
| 13. | $\chi_g$ | Mass susceptibility |
| 14. | $\chi_m$ | Molar mass susceptibility |
| 15. | $v$ | Absorption maximum |
| 16. | µg | Microgram |
| 17. | IR | Infrared |
| 18. | $E_{qu}$ | Equation |
| 19. | B.M. | Bohr magnetron |
| 20. | M | Molarity |
| 21. | DMSO | Dimethylsulfoxide |
| 22. | DMF | Dimethyl formamide |

## SUMMARY OF THE PRESENT WORK

The very important large field in chemistry especially in bioinorganic chemistry is occupied by macrocyclic complex compounds. Macrocyclic complex compound is such type of a substance which contains macrocyclic ligand. Hemoglobin, myoglobin, vitamin $B_{12}$, chlorophyll etc. are naturally occurring macrocyclic complexes which play a vital role in all living beings. The present work is divided into two parts. Firstly preparation and characterization of some complexes and secondly study of their antibacterial and antifungal activity.

**Part-I**

In the present work some macrocyclic complex compounds of Pb(II), Zn(II), La(II), Cd(II) and Ag(II) containing a ligand having tetraoxo tetrahydrazin moiety were prepared by template condensation of malonodihydrazide ($C_3H_8N_4O_2$) with different aldehyde.

The complexes $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$, $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$, $[Pb(C_{14}H_{24}N_8O_4)(ClO_4)_2]$, $[Pb(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ $[Pb(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ (1-6) were prepared by the reaction of malonodihydrazide with Pb(II) perchlorate hexahydrate in presence of Formaldehyde, Acetaldehyde, Butanaldehyde, Crotonaldehyde, Banzaldehyde and Cinnamaldehyde respectively in 2:1:2 molar ratio.

Similarly the complexes $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ , $[Zn(C_{10}H_{20}N_8O_4)(ClO_4)_2]$, $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ , $[Zn(C_{24}H_{24}N_8O_4)(ClO_4)_2]$, $[Zn(C_{20}H_{28}N_8O_4)(ClO_4)_2]$, $[Zn(C_{14}H_{24}N_8O_4)(ClO_4)_2]$, $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ , $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$,

[La ($C_{14}H_{28}N_8O_4$)($ClO_4$)$_2$] , [La( $C_{14}H_{24}N_8O_4$)($ClO_4$)$_2$], [La ($C_{24}H_{28}N_8O_4$)($ClO_4$)$_2$] , [La ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$],[Cd($C_8H_{16}N_8O_4$)($ClO_4$)$_2$], [Cd ($C_{10}H_{20}N_8O_4$)($ClO_4$)$_2$], [Cd ($C_{14}H_{28}N_8O_4$)($ClO_4$)$_2$], [Cd ( $C_{14}H_{24}N_8O_4$)($ClO_4$)$_2$], [Cd ($C_{24}H_{28}N_8O_4$)($ClO_4$)$_2$], [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$], [Ag($C_8H_{16}N_8O_4$)($ClO_4$)$_2$], [Ag($C_{10}H_{20}N_8O_4$)($ClO_4$)$_2$], [Ag ($C_{14}H_{28}N_8O_4$)($ClO_4$)$_2$], [Ag( $C_{14}H_{24}N_8O_4$)($ClO_4$)$_2$], [Ag ($C_{24}H_{28}N_8O_4$)($ClO_4$)$_2$], [Ag ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] (7-30) were prepared by the reaction of malonodihydrazide with Zn(II), La(II), Cd(II), Ag(II) perchlorate hexahydrate in presence of Formaldehyde, Acetaldehyde, Butanaldehyde, Crotonaldehyde, Banzaldehyde and Cinnamaldehyde respectively in 2:1:2 molar ratio.

The New macrocyclic complexes [Pb($C_{10}H_{12}N_4O_4$)($ClO_4$)$_2$] were prepared by the reaction of ethylinediamine with Lead(II) perchlorate hexahydrate in presence of malonic acid in 2:1:2 molar ratio.

The complexes **1-31** have characterized on the basis of elemental analysis, UV-visible and IR spectral analysis, magnetic moment, thermal analysis, XRD analysis and conductance measurement, and some other physical properties.

**Result:** The U.V- visible analysis of the all complexes are shown in octahedral geometry. The molar conductance data are shown non-electrolytic in nature. All the complexes for the IR band are indicated $\nu$ (N-H), $\nu$ (M-H), $\nu$ ($ClO_4$) band are present. The magnetic moment data indicate $d^{10}$ system and thermal properties indicate the stability of the complexes. Among the all complexes only La containing complexes are crystalline in nature.

**Part-II**

The antibacterial activity of the complexes **(1-31)** and the ligands have been studied against fourteen pathogenic bacteria (viz. *Salmonella-17, Klebsilla, Shigella dysenteriae, Shigella shiga, Shigella boydii, Shigella sonnei, Shigella flexneri, Escherichia coli, Pseudomonas aeruginosa, Salmonella, Bacillus megaterium, Sarcina lutea, Staphylococcus aureus, Bacillus cereus*) and the minimum inhibitory concentrations of the complexes in Pb 1, 3, 4 Zn 3, 4, 5 La 1, 3, 4 Cd 1, 2, 6 Ag 1,2, 3 were found. Among the 31 complexes the complexes in Pb 3, Zn 5, La 4, Cd 6 Ag 1 are showed excellent activity against the above fourteen pathogenic bacteria. The results were compared with the standard compound, kanamycine.

The antifungal activity of the complexes **(1-31)** and the ligands have been studied against four fungal (viz. Aspergillus Niger, Socehoramyces cerevisae, Pericillium notatum, mueor sp) but could not found any activity of the complexes in four fungal.

# GENERAL INTRODUCTION

## 1.1 INTRODUCTION

Macrocyclic complex compounds are an immense filed in chemistry, especially in coordination chemistry and in bio-inorganic chemistry. The chemistry of macrocyclic complexes has received much attention and such compounds have been extensively studied in recent years [1]. Macrocyclic metal complexes are of great importance due to their resemblance of many natural systems such as porphyrine ,hemoglobine, mioglobine, vitamine $v_{12}$ etc. The field of macrocyclic chemistry of metals is developing very rapidly because of its importance in the area of coordination chemistry, medicinal chemistry, toxicology, ligand selection in metalloproteins and enzymes[2,3]. Synthetic macrocyclic ligangs are of significance because porphyrin play vital role in biological systems, such chelating molecules are important since they are capable of furnishing an environment of controlled geometry and ligand field strength[4]. These compounds have also been screened for toxicological effect [5,6]. The coordination in organometallic compounds having metal nitrogen bonding occupy an important position amongst the recent developments related to bioinorganic systems [7]. Metal ion recognition is of fundamental importance to broad areas of both chemistry and biochemistry. The importance of metal ion in biological systems as macrocyclic compounds is well established because of their catalytic behavior in a number of redox reactions of biological significance [8]. In the recent years there is a growing interest in the chemistry of hydrazides, hydrazones and arylhydrazones owing to their pronounced biological activity and analytical applications[9]. Many macrocyclic complexes were prepared by the aid of metal ions as templates to direct the condensation

reaction towards ring closure [10]. Template synthesis of 14,15,16,18-membered macrocyclic complexes of Metal (II) by the reactions of amine/hydrazide with aldehydes/ketones are well known[11] and some of them possess good activity in the biological system [12-15].

The chemistry of macrocyclic complexes is also important due to their use as dyes and pigments as well as NMR shift reagents. Furthermore, some macrocyclic complexes have been found to exhibit potential antibacterial activities.[16]

Prompted by these facts, in the present paper, a series of macrocyclic complexes of Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) obtained by template condensation reaction of malonyldihydrazide and benzil are reported. The complexes were characterized with the help of various physic-chemical techniques, such as elemental analyses, IR, NMR and electronic spectral studies and magnetic susceptibility and molar conductance measurements. These macrocyclic complexes were also screened for their in vitro antibacterial and antifugal activity.[17]

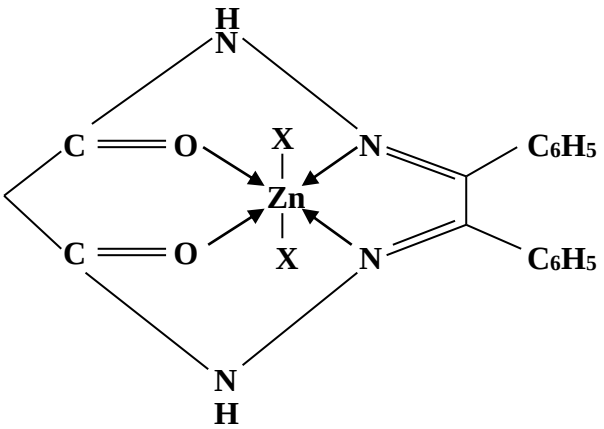



**Fig. 1 ( Macrocyclic complex of $Zn^{2+}$ )**

The transition and lanthanide metal ions have been used as templates in the synthesis of 10, 12, 13, 14 and 18-membered polyaza macrocyclic complexes[121]. The potential of macrocyclic ligand systems with their central cavity for use as metal ion selective reagent has been widely recognized now a day[122]. Apart from more obvious parameters such as donor atom, radii and type of hybridization, a range of other structural factors, including chelate ring size, extent of ligand rigidity and the presence or absence of ring substituent's can all influences the geometry of the binding cavity often in subtle ways. For a number of systems in which the metal ion fully occupies the macrocyclic cavity.[18-25]

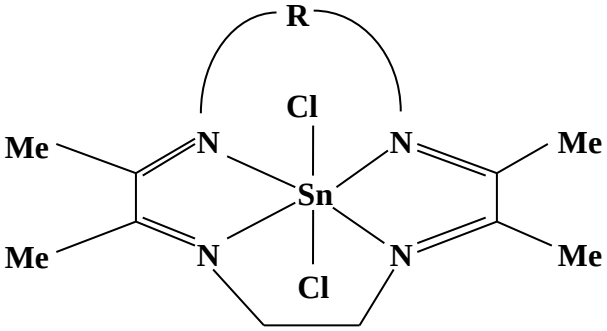



**Fig. 2 ( Macrocyclic complex of $Sn^{2+}$ )**

The main purpose of each method is to prevent the crystallite agglomeration, control the particle shape, size, and crystal phase of ZnO nanoparticles. We know that ZnO manoparticles with certain morphology and size could be obtained using suitable surfactant and solvent. Moreover, it requires suitable conditions to stabilize the nanoparticles. However, most of the methods have been found to be expensive, polluting, and time consuming. Therefore, it

still remains and extremely difficult challenge to find a simple and mild synthetic route to synthesize well-controlled ZnO nanoparticles[26].

It has been reported that thermal decomposition method is an efficient, simple, one-step, solvent free approach to generate ZnO nanoparticles. As compared to others method, it is much faster, pollution free, and economical. However, the use of macrocylic complexes as precursors for the preparation of metal oxide nanomaterial's such as ZnO using thermal decomposition has not yet been investigated[27-30]. Hence we made an attempt to synthesize ZnO nanopartcles using Zn(II) complex of tetraiminediphenol macrocycle as precursor for the first time.

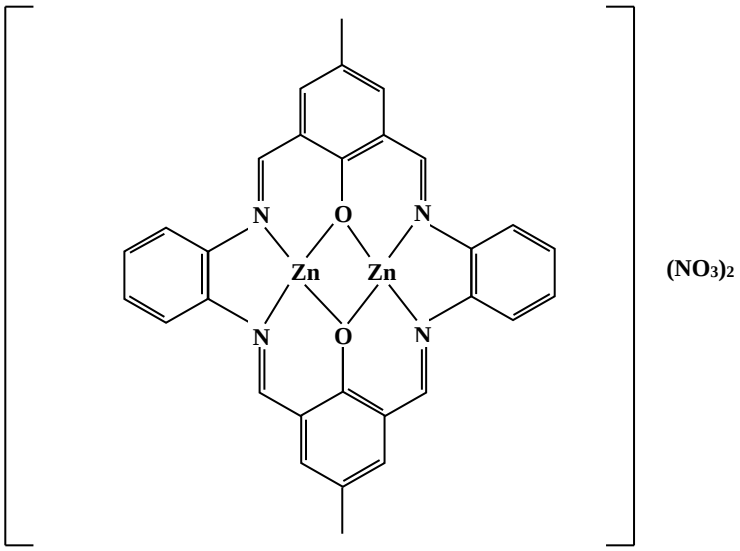



**Fig.3** Structure of the complex $[Zn_2L](NO_2)$

Naturally occurring macrocylic-ligand transition metal complexes such as complexes of the porphyrin or corrin ring systems and the industrially important metal phthalocyanine complexes have been studied for many years. More recently, a large number of other macrocyclic ligands have been

synthesized and their metal complexes have been extensively studied. The present review gives an outline of the transition-metal chemistry of this latter group of cyclic ligands with an emphasis being placed on the more recent work.

The chemistry of synthetic macrocyclic ligands can be divided into two broad divisions. Firstly there are the cyclic polyether's of the 'crown' type of which (Fig-4) is a typical example[31]. Ligands of this general category have received much recent attention because of their unusual behavior towards a range of non-transition metal ions[32]. Few studies involving transition metal ions have been reported[33] and it is evident that the majority of such polyether ligands show a limited tendency to form stable complexes with these ions[34].

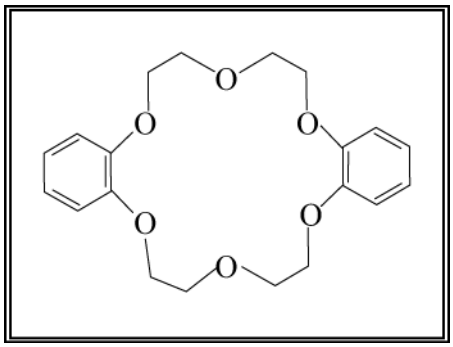



**Fig. 4** (Crown)

The modern study of coordination compounds begins with two famous men, Alfred Werner and Sophus Mads Joargensen. Although S.M. Jorgensen (1837-1914) started the extensive studies on the thesis of complex compounds, it was not until 1906 when the recognition of the true nature of complexes began with Alfred Werner in 1893, proposed the coordination theory[35]. For this pioneering work Alfred Werner received the Nobel price in 1913. In fact he was the founder of modern coordination chemistry who postulated the first successful theory, known as "Werner's coordination

Theory", to explain, the formation, properties and stereochemistry of coordination compounds. The independent approaches of Sidgwick[36] and Lowry[37], who proposed that a chemical bond required the sharing electron Pair led to the idea that a neutral molecule with an electron pair (Lewis-base) can donate these electrons to a metal ion or other electron acceptor (Lewis-acid). Although the electron pair donor concept of Lewis base is still useful for many Lewis acid base interactions for complex formation[38], it is apparent that the natures of bonding in metal complexes require more detailed consideration to understand. The detailed and more modern concepts to explain formation of bonds, the associated bond properties, structures, stabilities and the molecular properties as a whole are more conveniently and successfully considered in terms of modern bonding theories the valence bond theory[39], the crystal filed theory and the ligand field theory[40] and the molecular orbital theory[41].

The coordination chemistry of hydrazones is an intensive area of study and numerous transition metal complexes of these ligands have been investigated[42]. The development of the field of bioinorganic chemistry has increased the interest in Schiff base complexes, since it has been recognized that many of these complexes may serve as models for biologically important species[43-46]. Coordination compounds derived from aroylhydrazones have been reported because of their anti-tuberculosis, antimicrobial and corrosion inhibitors[47-50].

## 1.2 CLASSIFICATION OF LIGANDS

A coordination compound contains a central metal atom or ion surrounded by a number of oppositely charged ions or neutral molecules known as ligands likes $NH_3$, $CN^-$, $Cl^-$ etc. There are many kinds of ligands[51].

### 1.2.1 Unidentate Ligands

Any individual atom, ion or molecule that is attached coordinately to one central metal atom is called unidentate ligand. The unidentate ligands are $F^-$, $Cl^-$, $Br^-$, $NH_3$, $H_2O$, $OH^-$.

### 1.2.2 Bidentated Ligands

The bidentated ligands are the molecules which can form simultaneously two coordinate bonds with the same metal ion. The bidentated ligands are ethylenediamine, Bis (dipheylpbosphio) ethane (diphos or dppe), 2 2'-Bipyridine (bpy), 1.10-Phenanthroline (phen) etc. Below the Fig 1.1 and 1.2 as follows:

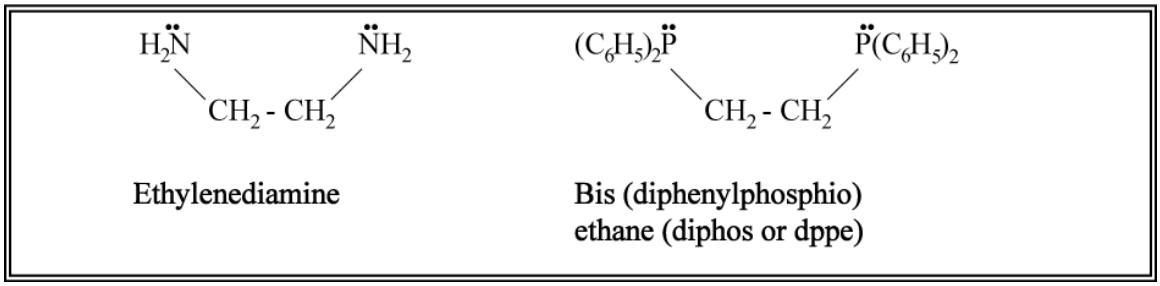



**Fig. 1.1**

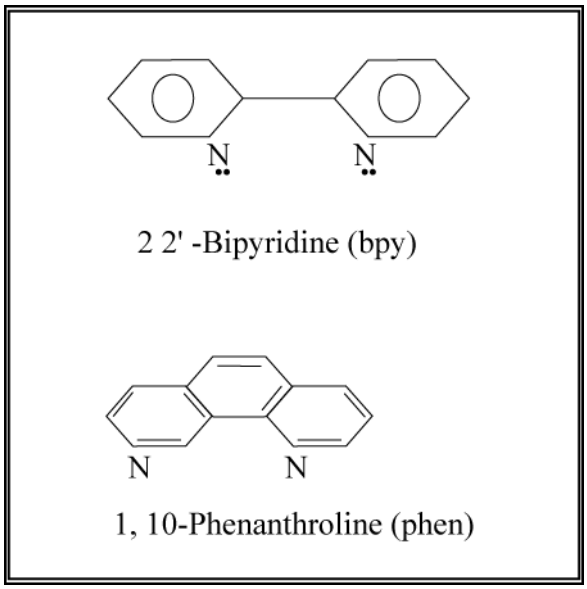



**Fig. 1.2**

### 1.2.3 Tridentate Ligands

The tridentate ligands are the molecules which can form three coordinate bonds simultaneously with the same metal ion. Some of the most important tridentated ligands are follows. (Fig 1.3).

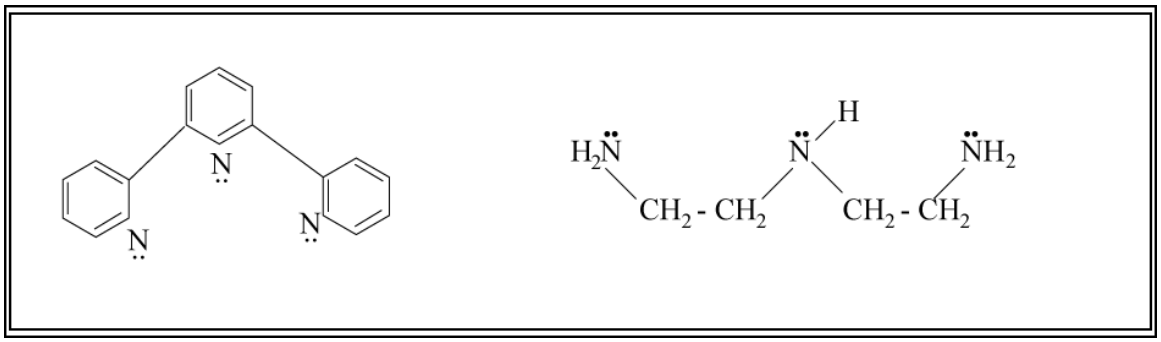


**Fig. 1.3** (Diethylene triamine and tridentate aromatic ligand)

### 1.2.4 Tetradentate ligands

A tetradentate ligand can be defined as any molecule or ion that has four pair of electrons which can be donated to the central metal ion. Then the molecule or ion is called tetradentate ligand[53]. (as in Fig-1.4)

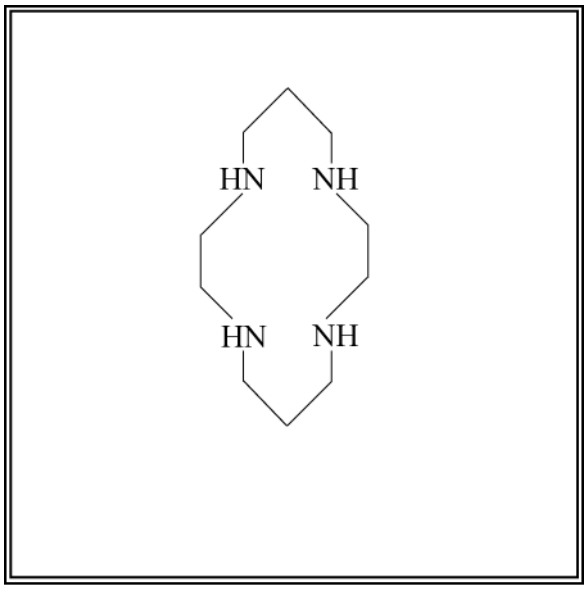



**Fig. 1.4**

( Cyclic tetradentate ligand )

### 1.2.5 Bridging Ligand

A bridging ligand links two or more metal center. Virtually all inorganic solids with simple formulas are coordination polymers consisting of metal centres linked by bridging ligands. This group of materials includes all anhydrous binary metal halides and pseudohalides. Bridging ligands also persist in solution. Polyatomic ligands such as carbonate are ambidentate and thus are found to often bind to two or three metals simultaneously.

### 1.2.6 Non-innocent Ligand

Non-innocent ligands form bond with metals in such a manner that the distribution of electron density between the metal center and ligand is unclear. Describing the bonding of non-innocent ligand often involves writing multiple resonance forms which have partial contribution to the overall state.

### 1.2.7 Bulky Ligand

Bulky ligands are used to control the steric properties of a metal center. They are used for many reasons, both practical and academic. On the practical side, they influence the selectivity of metal catalyst, e.g in hydroformylation. Of academic interest, bulky ligands stabilize unusual coordination sites, e.g reactive coligands or low coordination numbers. Often bulky ligands are employed to simulate the steric protection afforded by proteins to metal containing active sites. Of course excessive steric bulk can prevent the coordination of certain ligands.

### 1.2.8 Strong Field and Weak Field Ligands

In general, ligands are viewed as electron donors and the metals as electron acceptors. Bonding is often described using the formalisms of molecular orbital theory. The highest Occupied Molecular Orbital (HOMO) can be mainly of ligands or metal character.

Ligands and metal ions can be ordered in many ways; one ranking system focuses on ligand ‘hardness’. Metal ions preferentially bind certain ligands. In general, ‘soft’ metal ions prefer weak field ligands, whereas ‘hard’ metal

ions prefer strong field ligands. According to the molecular orbital theory, the HOMO of the ligand should have an energy that overlaps with the Lowest Unoccupied Molecular Orbital (LUMO) of the metal preferential. Metal ions bound to strong field ligands follow the Aufbau principle, whereas complexes bound to weak field ligands follow Hund's rule. Binding of the metal with the ligands results in a set of molecular orbitals, where the metal can be identified with a new HOMO and LUMO (the orbitals defining the properties and reactivity of the resulting complex) and a certain ordering of the 5d-oritals.

### 1.2.9 Tetra Azamacrocyclic Ligands

The macrocycles cyclen and cyclam {[12-ane]$N_4$ and [14-ane]$N_4$} have been known for several decades, and their complexation chemistry with a large variety of metal ions has been studied thoroughly. Such macrocyclic ligands often lead to complexes with enhanced thermodynamic and kinetic stability with respect to metal ion dissociation, compared to their open-chain analogues. (as in Fig-1.5)

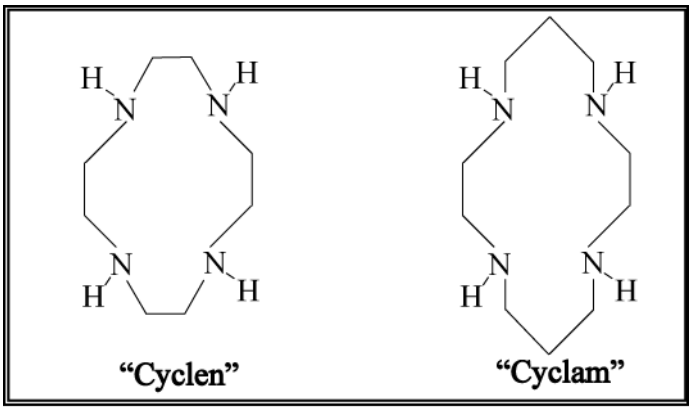



Fig. 1.5

Metal ions with a preference for coordination numbers > 4 will require further ligands to be bound (apart from the four nitrogen atoms of the macrocycle), and these may be provided by the functionalisation of the macrocycle with additional pendent coordinating groups. This leads to higher-dentate ligands whose properties and selectivity for certain metal ions over others may be quite different from those of the unsubstituted parent macrocycles.

During recent work, cyclam-based macrocycles incorporating two additional coordinating groups (eg. pyridyl groups) have been prepared via di-N-alkylation. Since cyclam has $C_2$ symmetry, there are three different isomers for a di-N-substituted system, the 1,4-, 1,8- and 1,11-functionalised ligands[54]. For example, 1,8-and 1,11-bis (pyridylmethyl) cyclam have been synthesised, and it turns out that the stuctures of the complexes they form with copper(II) are completely different! (As in Fig-1.6)

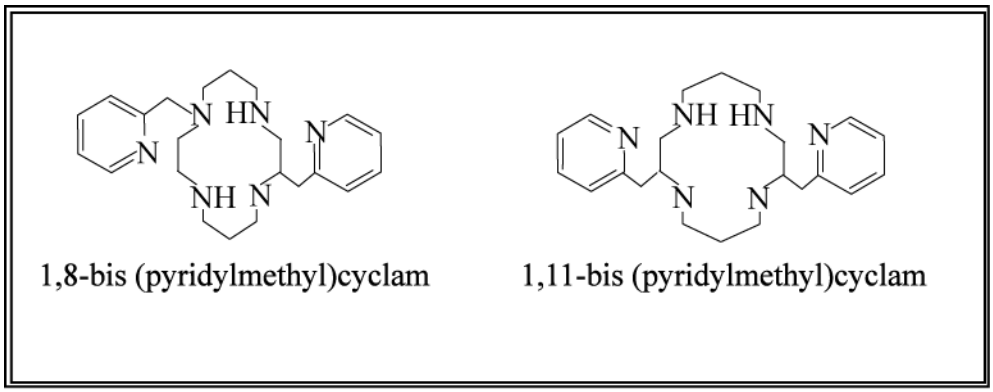



**Fig. 1.6**

The chemistry and complexation properties of macrocyclic dioxotetraamines were investigated[24]. These macrocycles contain two amino nitrogens and two amides. (as in Fig-1.7)

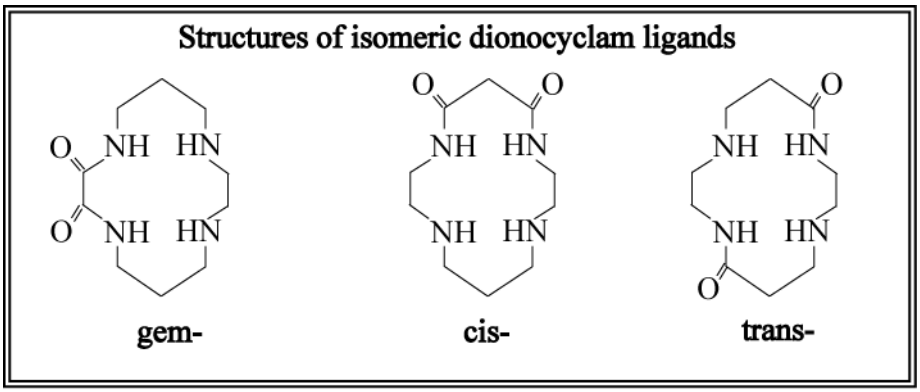



Fig. 1.7

As with cylam and cyclen, the amino nitrogens with additional coordinating groups form new hexadentate ligands. They are able to bind to metals like copper(II) and nickel(II) with simultaneous dissociation of the two amide protons, such that metal binding is highly pH-sensitive and reversible (-a very useful property for metal-sensing applications). The copper(II) complex of a functionalised trans system at neutral and basic pH, and found very different structures according to whether just one or both of the amides are deprotonated[55].

## 1.3 THE LIGANDS OF SCHIFF BASE

Schiff base complexes have remained an important and popular area of research due to their simple synthesis, versatility, and diverse range of applications[56]. The interest in the design, synthesis and characterization of the transition metal complexes of unsymmetrical Schiff base ligands has come from the realization that coordinated ligands around central metal ions in natural systems are unsymmetrical[57]. Recently, this class of compounds has also attracted much attention in the field of optoelectronic technologies for their large nonlinear responses[58]. In the area of the bioinorganic chemistry, interest in the Schiff base complexes with transition and inner-transition metals has centered on the role of such complexes in providing synthetic interesting models for the metal-containing sites in metallo-proteins and enzymes[59,60]. They appear to be of importance for a broad range of transition metal catalyzed reactions including lactide polymerization[61,62], epoxidation of olefins[63] and hydroxylation[64]. Several reviews have been published on metal Schiff bases especially on metal Salen Schiff base complexe6[5,66]. Unsymmetrical Schiff base ligands have clearly offered many advantages over their symmetrical counterparts in the elucidation of the composition and the geometry of the metal ion binding sites in the metallo-proteins and the enzymes, and the selectivity of the natural systems with synthetic materials[67].

The aminooxidase enzyme requires such a coenzyme besides copper(II) ions for catalytic activity. Urease, the first enzyme crystallized to be shown to possess nickel ions[68], is an important enzyme in both agriculture and medicine, which rapidly catalyzes the hydrolysis of urea to form ammonia

and carbamic acid[69]. Recently Cu(II) and Ni(II) Schiff base complexes have been investigated as inhibitor against urease and xanthine oxidase(XO)[70].

Some unsymmetrical tetradentate Schiff base ligands[71-73] and their Ni(II) and Cu(II) complexes were synthesized and characterized by elemental anaylsis, IR, ′H NMR, UV-Vis spectra, mass spectra and magnetic moments. The thermodynamic formation constants ($K_f$) of the complexes were determined spectrophotometrically and their free energies were calculated at $25^0$C.

Most of the binuclear complexes have been prepared from tridentate Schiff bases containing ONO or ONS donor atoms; they are derived from the condensation of salicylaldehyde or acetylacetone with o-aminophenols, aminoalcohols, o-aminoacids, o-aminothiophenols and aminothiols (as in Fig. 1.8).

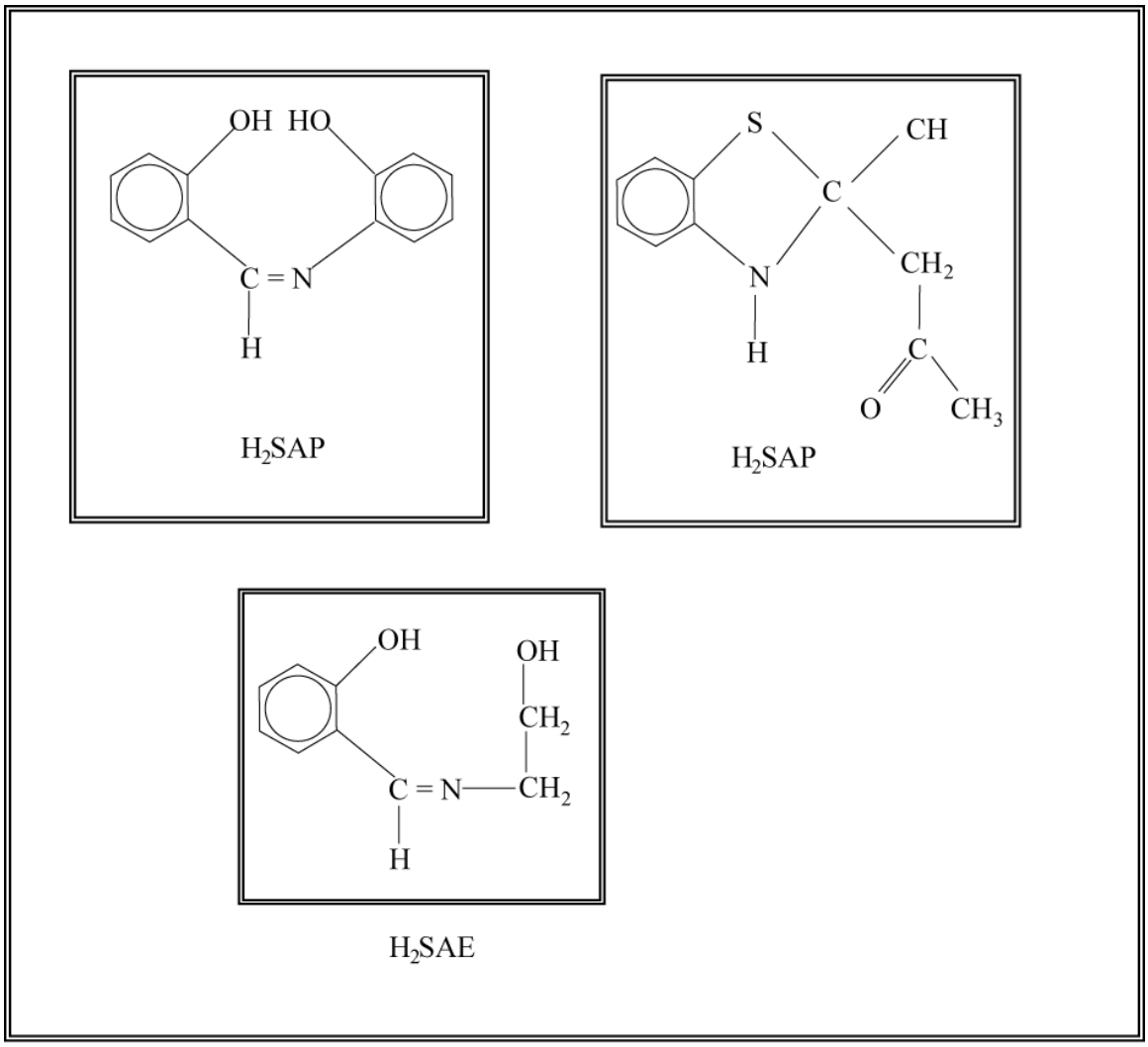



**Fig. 1.8**

When these ligands react with metal ions, their tridentate character leads to polymerization and polynuclear complexes with anomalous magnetic properties. It was presumed taht the copper(II) ions of these chelates would have and unusual coordination number of three[74]

**Some examples of open chain Schiff bases:** Are given below as in Fig. 1.9

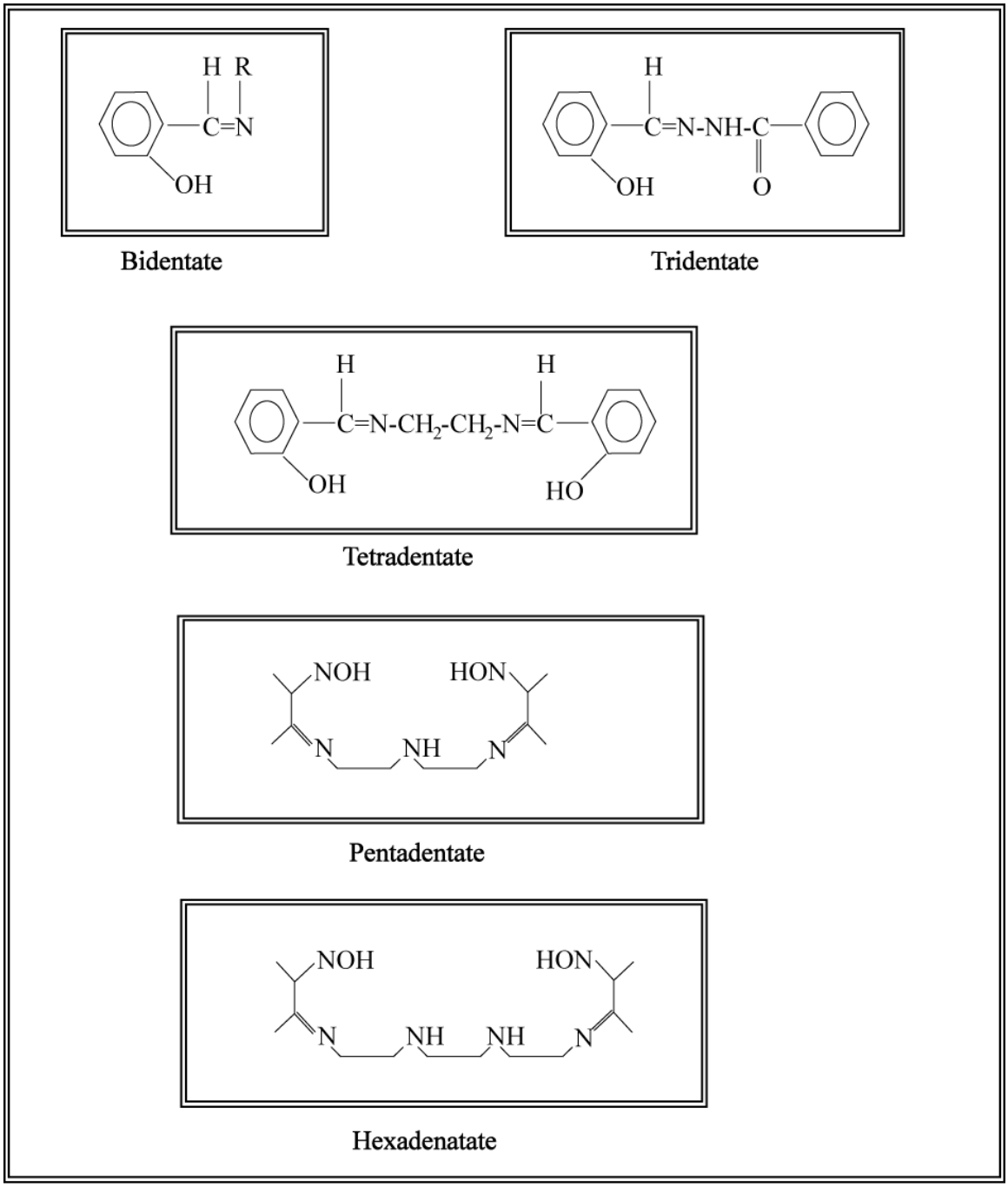



**Fig. 1.9**

Schiff bases containing polydentate group[75] have not only produced stable metal compounds but these ligand and their metal compounds play

significant role in stereochemistry, structure, isomerism, spectroscopy, kinetics and mechanism of reactions, model system of biochemical interest, stabilizes, polymers, photography, electro optical display devices and agriculture. The chelating properties of these ligands are of interest to many authors[76].

The commonest donor atoms in the Schiff base ligands are oxygen and nitrogen, less commonly halogen, sulphur, photosphorus, arsenic and carbon atmos act as donors. In some cases bond appears to act as donors. Some examples of such kinds of Schiff bases ligands are depicted below[77] (as in Fig. 1.10)

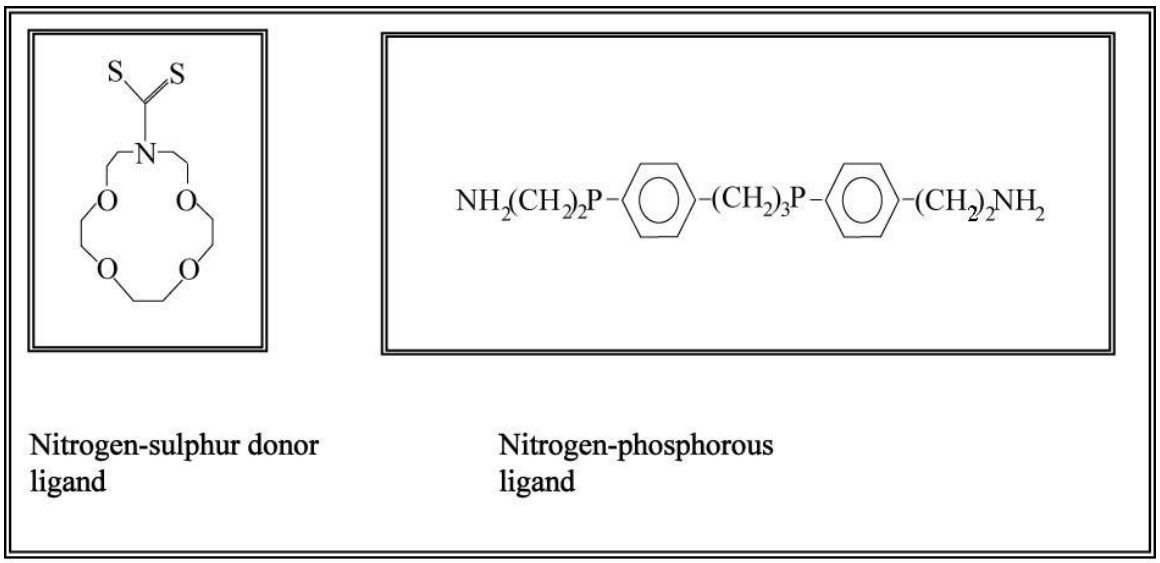



**Fig. 1.10**

## 1.4 PREPARATION AND MECHANISM OF SCHIFF BASE LIGANDS[78]

The Schiff base ligands are formed by the condensation of primary amine and aldehydes or ketones looses the acidic protons and behave as chelating agent (as in Fig. 1.11)

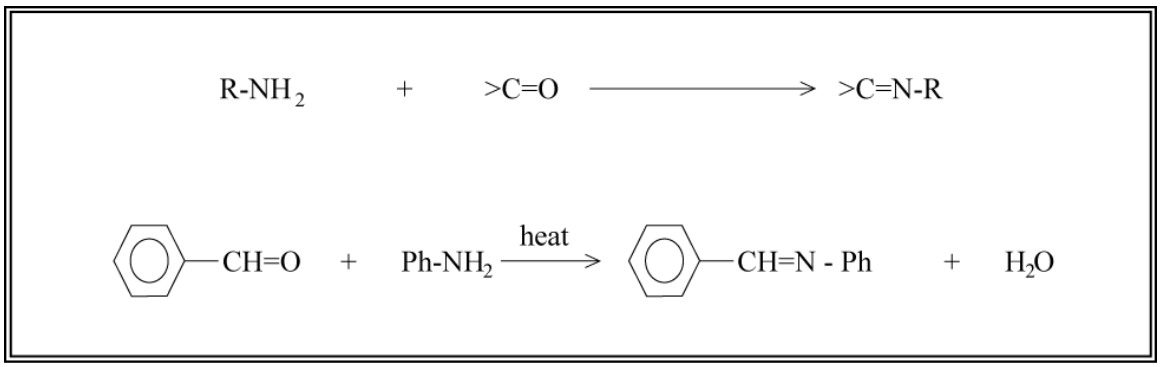
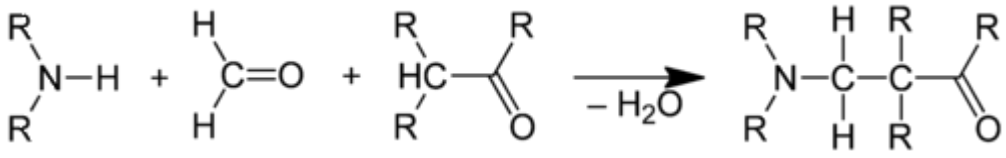



**Fig. 1.11**

The Mannich reaction is an organic reaction which consists of an amino alkylation of an acidic proton placed next to a carbonyl functional group with formaldehyde and ammonia or any primary or secondary amine. The final product is a β-amino-carbonyl compound also known as a Mannich base[79]. Reactions between aldimines and α-methylene carbonyls are also considered Mannich reactions because these imines form between amines and aldehydes. The reaction is named after chemist Carl Mannich[80,81].



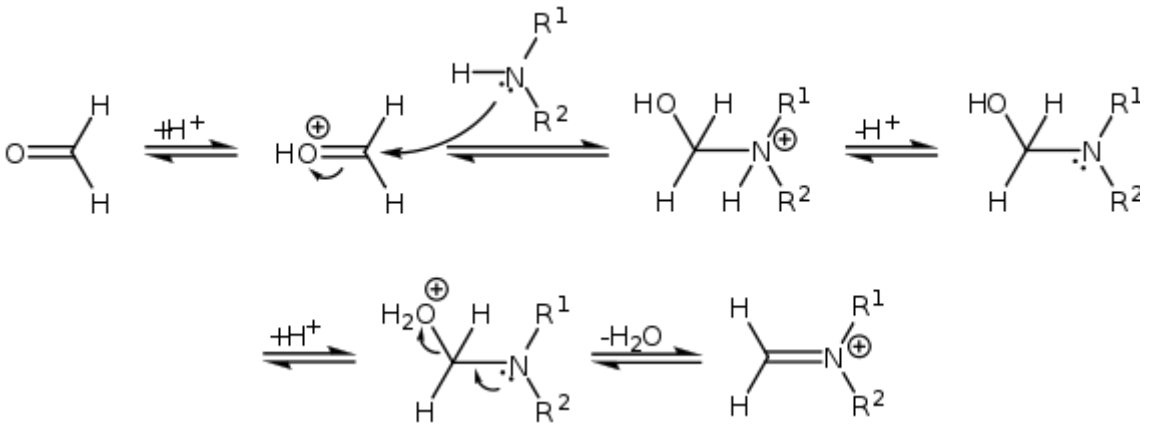

The Mannich reaction is an example of nucleophilic addition of an amine to a carbonyl group followed by dehydration to the Schiff base. The Schiff base

is an electrophile which reacts in the second step in a electrophilic addition with a compound containing an acidic proton (which is, or had become an enol). The Mannich reaction is also considered a condensation reaction.

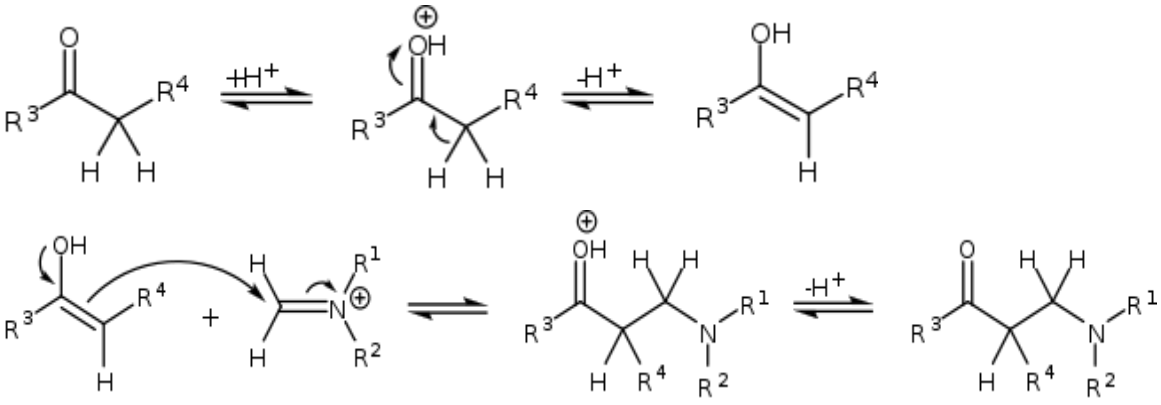
In the Mannich reaction, ammonia or primary or secondary amines are employed for the activation of formaldehyde. Tertiary amines lack an N-H proton to form the intermediate imine. α-CH-acidic compounds (nucleophiles) include carbonyl compounds, nitriles, acetylenes, aliphatic nitro compounds, α-alkyl-pyridines or imines. It is also possible to use activated phenyl groups and electron-rich heterocycles such as furan, pyrrole, and thiophene. Indole is a particularly active substrate; the reaction provides gramine derivatives.

**Reaction mechanism**

The mechanism of the Mannich reaction starts with the formation of an iminium ion from the amine and the formaldehyde.



Because the reaction takes place under acidic conditions, the compound with the carbonyl functional group (in this case a ketone) can tautomerize to the enol form, after which it can attack the iminium ion.



## 1.5 UNSYMMETRICAL SCHIFF BASE LIGAND

A unsymmetrical Schiff base ligand as in Fig. 1.12. that is very resistant to oxidation and consequently useful for complexing metals in high oxidation states[82].

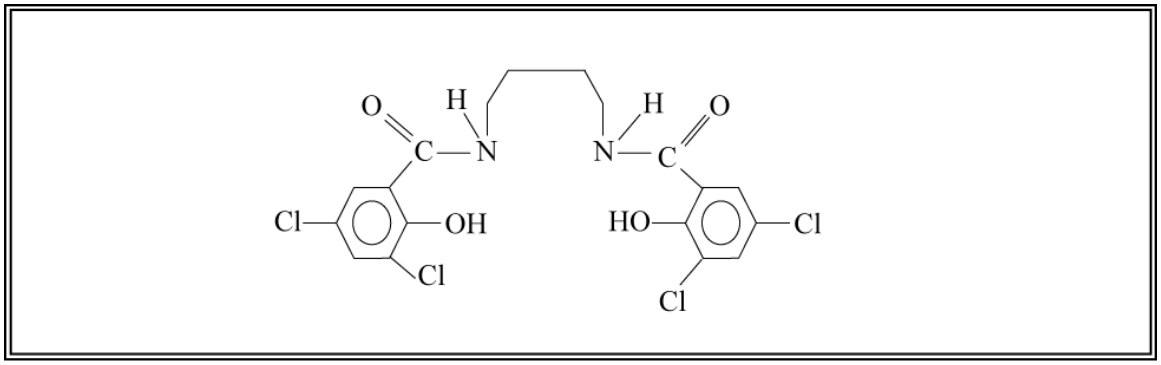


**Fig. 1.12**

(Unsymmetrical ligand)

## 1.6 THE MACROCYCLIC LIGANDS AND THEIR BEHAVIOUR

The large ring compounds whose structures are such that several donor atom's can bind to a metal most commonly nitrogen donors. However, mixed N,O; N,S; N,O,S; N,O,P; and so on, donors are known. Depending on the donor atoms these can be designated $N_4$, $N_2$, $O_2$, $O_4$ and so on. The

heterocyles can be broadly classed into these without and those with conjugated π system.

### 1.6.1 Broad classification of macrocyclic ligands:[83]

Planar with unsaturated rings as in porphyrin and its derivatives, the metal atom may be out of the plane of the N donor atom ligand (as in Fig. 1.13).

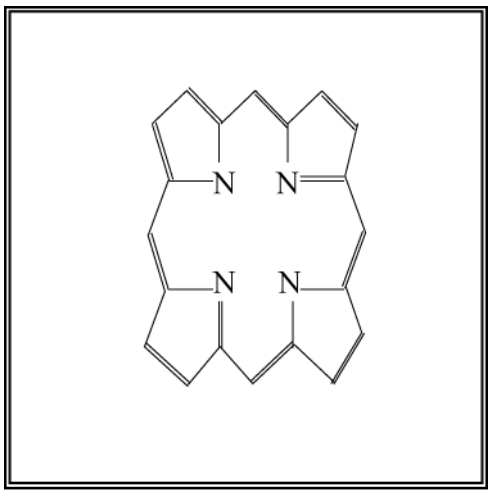



**Fig. 1.13**

(porphyrin N donor ligand)

### 1.6.2 Macrocyclic complexes have the following characteristics.[84]

i. A marked kinetic inertness both to the formation of the complexes from the ligand and metal ion.

ii. They can stabilize high oxidation state, that are not normally readily attainable, such as Cu(III) or Ni(III).

iii. They have high thermodynamic stability than the formation constants for non-macrocyclic $N_4$, ligand. Thus for Ni(II) the formation constant for the macrocyclic tetradentate Ni complex (Fig. 1.14) is about five orders of magnitude greater than that for the non-macrocyclic tetradentate Ni complex (Fig. 1.15).

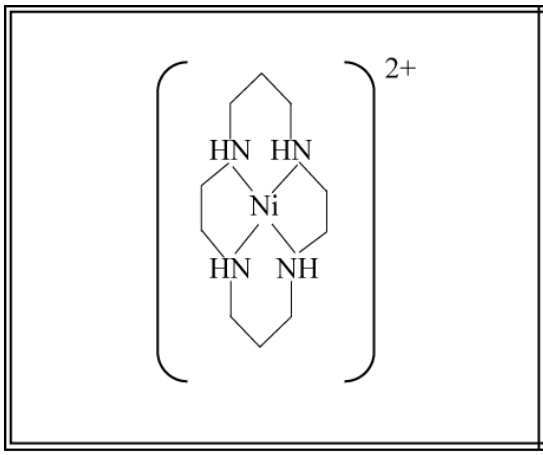



Fig. 1.14

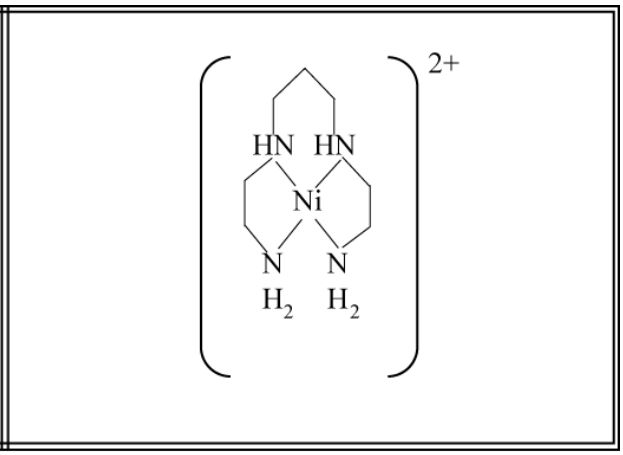



Fig. 1.15

(Highly thermodynamic stability of complex) (non-macrocyclic tetradentate complex)

## 1.7 TEMPLATE SYNTHESIS OF MACROCYCLIC COMPLEXES

The rich chemistry of the complexes of macrocyclic ligands continues to be a subject of growing importance, as reflected by a number of recent reviews and books[85]. Much of this work has been stimulated by the recognition of the high kinetic and thermodynamic stability of the complexes formed by macrocyclic ligands, the so-called "macrocyclic effect"[86], and also by the realization of the key importance of cyclic ligand systems in biology, for example, the protoporphyrin IX ligand of heme, or the corrin system found in the Vitamin $B_{12}$ coenzyme[87]. Because of this work, macrocyclic ligands are discussed routinely as an integral part of any course on transition metal coordination chemistry. However, comparatively few undergraduate experiments are available to illustrate the practical side of macrocyclic synthesis, although one example, of the synthesis of a binuclear macrocycle, was reported fairly recently[88].

Many of the synthetic routes to macrocyclic ligands involve the use of a metal ion template to orient the reacting groups of the ligand in the desired conformation for optimum ring closure. The favorable enthalpy for the formation of the metal-ligand bonds overcomes the unfavorable entropy from the ordering of the multidentate ligand around the metal ion and thereby promotes the cyclization reaction. This phenomenon has been described as the "coordination template effedct"[89]. (as in Fig. 1.16)

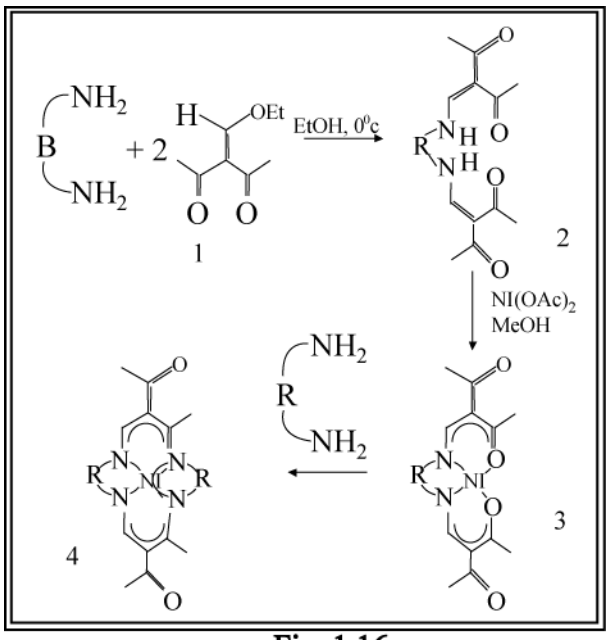
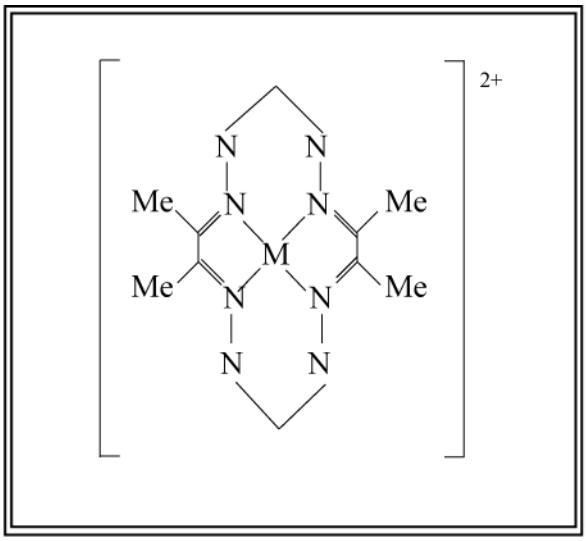



**Fig. 1.16**

(cyclization reaction of macrocyclic effect)

The diacetyl dihydrazone reacts with Ni(II) in the presence of formaldehyde in different ratio to give the octaazamacrocyclic (14-membered), tricyclic ligand complexes with two cyclic ethers fused to the macrocyclic ring, quadricyclic ligand complexes[90].(as in Fig. 1.17)



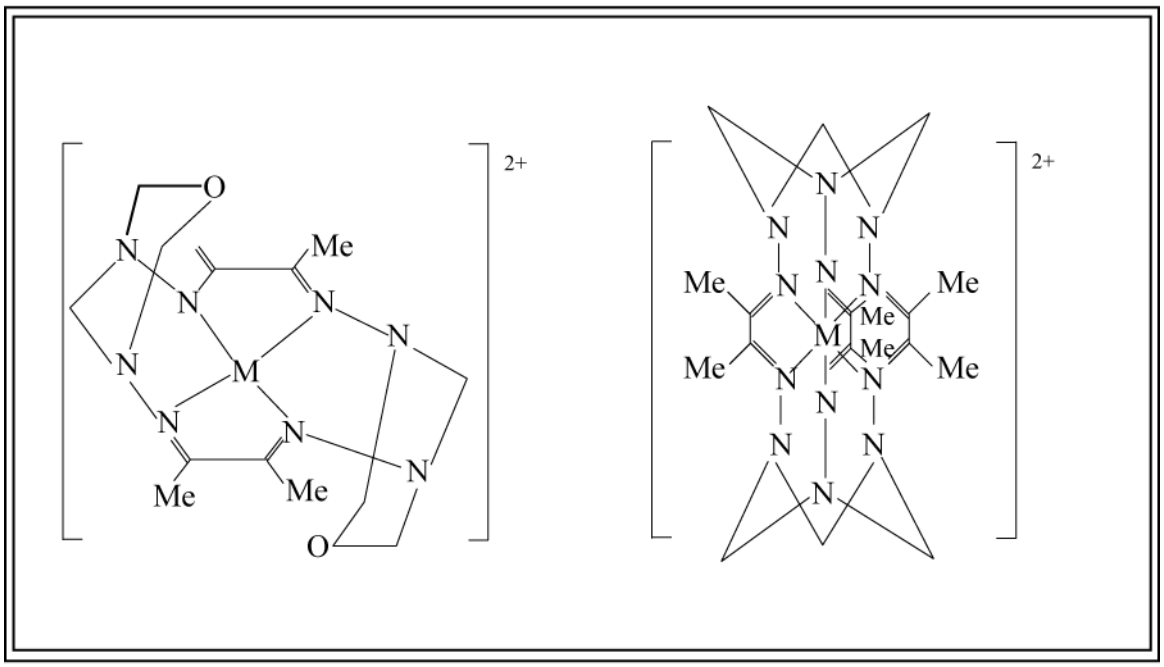




**Fig. 1.17:** Template complexes formed by diacetylmonoxime hydrazone and formaldehyde.

It is to be noted that there must be some control of the reaction by the size of the metal used. If the ion is too small or too large, no macrocyclic complex may be formed[91].

## 1.8 MACROCYCLIC COMPOUNDS CONTAINING "OXO" GROUPS:

The scarcity of highly oxidized middle and later transition metal complexes challenges chemists to develop ligand complements compatible with oxidizing metal centers[92].

Two chromium(V)-oxo complexes, $[(CH_3)_4N][Cr(O)(\eta^4\text{-}1)$ and $[(CH_3)_4N][Cr(O)(\eta^4\text{-}2)]$, have been synthesized and characterized by x-ray crystallography and IR and EPR spectroscopies. Because exchange of the oxo ligand with water is slow, the easily synthesized, stable, crystalline $^{18}O$-labeled diperoxide $(CH_3)_2(H^{18}O^{18}O)CCH_2CH_2C(^{18}O^{18}OH)(CH_3)_2$ was prepared and used to conveniently synthesized $^{18}O$-labeled oxo complexes in high yields. The bonding of the two unique oxidation-restistant macrocyclic tetraamides to chromium is compared. The discovery of these unusual structural parameters expands the class of nonplanar amides arising from ring constraint. (Fig. 1.18)

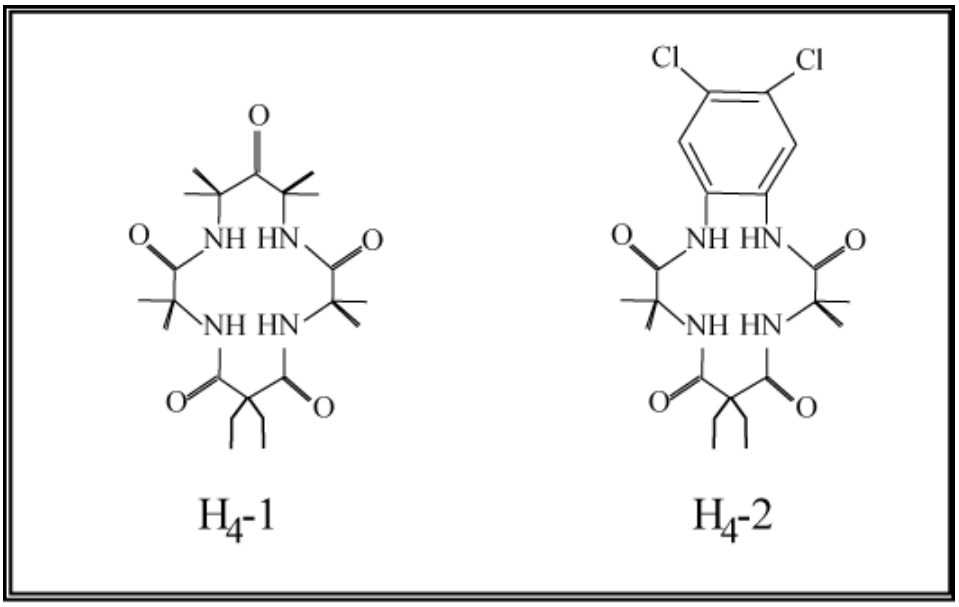



**Fig. 1.18**

(Complex[$(CH_3)_4N$][Cr(O)($\eta^4$-1) and [$(CH_3)_4N$][Cr(O)($\eta^4$-2)])

## 1.9 COORDINATION COMPLEX OF PERCHLORATE ION

The recent article describing the use of [Cu(tmen)(arac)]$ClO_4$ as a color indicator for solvent parameters[93] fails to identify the potential danger associated with the preparation and handling of this salt. Most of us are aware that "organic perchlorates are self-contained explosives[94] However, many overlook the fact that a perchlorate salt of a cation, such as a complex ion that contains an organic group or other oxidizable atoms, is also an explosive (although the conditions required to initiate an explosion vary from sample to sample). For example, one sample of $Co(H_2O)_3(ClO_4)_2$ detonated under a slight impact while attempts to repeat the detonation with other samples were not successful[95]. Such compounds must be handled with great care[96], if at all.

The coordination chemist's grapevine is replete with stories of perchlorate explosions, most recently the explosion of a preparation of about 3 g of a perchlorate salt of a rhodium polyamine complex that detonated in a rotary evaporator over a hot water bath. This violent explosion destroyed the evaporator, smashed a lab jack, cracked the bench top, and chipped walls over 15 ft away. Fortunately, this happened in an empty lab. Explosions of other perchlorate salts of complex ions have resulted in serious injuries[97-99].

Testing a perchlorate for sensitivity may not be reliable. A recent letter[100] described the detonation of a perchlorate adduct of polyacetylene that previous tests suggested was relatively stable. Impurities and changes in crystalline type, habit, or size can have profound effects on the sensitivity of an explosive[101].

One solution to the problem of dealing with these explosive salts lies in replacing the perchlorate ion with a nonoxidizing ion. The ready availability of tetrafluoroborate salts and tetrafluoroboric acid, ($HBF_4$), and the charge and size compatibility of $BF_4^-$ and $CIO_4^-$ suggest that tetrafluoroborate salts make excellent substitutes.

Dicrpper(II) complexes of 20 member $N_4$ bincleating macrocyclics complexes of $L^1$, $L^2$, $L^3$ have been derived by a cyclic (2+2) condensation of diformylthiophene or diformylfuran with the appropriate diamine have been complexes $[Cu_2(L^1)(OH_2)(ClO_4)_2]H_2O$ and $[Cu_2(L^3)(OH_2)_2(ClO_4)_2]$ where the coordinated perchlorate bond is at (1085-620) $cm^{-1}$ synthesis of the dicopper(II) complexes[102], the macrocyclic ligand denoted by $L^1$ and $L^2$ as follows.

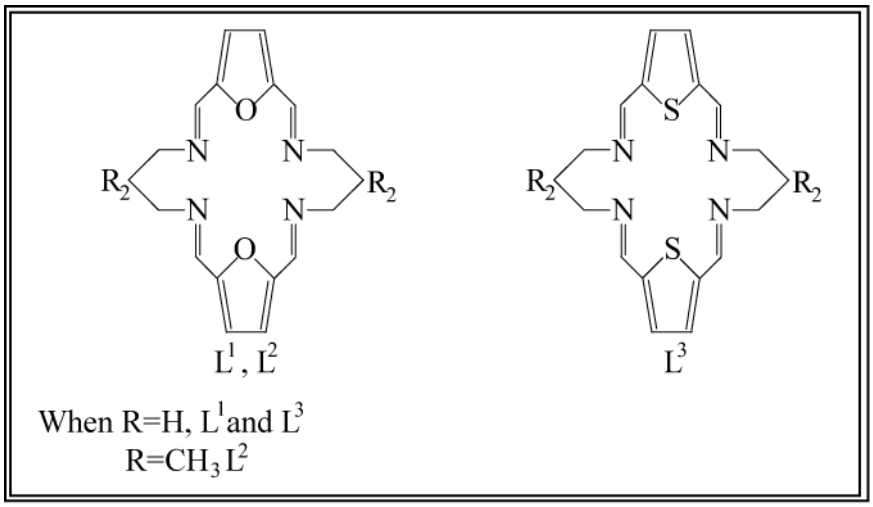



**Fig. 1.19**

(Macrocyclic complex of $[Cu_2(L^1)(OH_2)(ClO_4)_2]$)

Reaction of $Ln(ClO_4)_3$ with SMMT give the $[Ln(SMMT)_2ClO_4]$ complex (where Ln=La, Ce, Pr. Nd. Sm, Eu, Gd, Tb, Dy and Y) and [SMMT=4-slicylideneamino-3-mercapto-6-methyl-1,2,3,4-triazine(4H)-5-one][103].

The above complexes showed the conductance values (12.25-10.44) $ohm^{-1}$ $cm^2$ $mol^{-1}$ which is non-electrolytes[104]. The complexes showed IR bands at 1150, 1080, 660 $cm^{-1}$ for the vibration $v_1$ $v_2$ $v_3$ respectively[105]. The band at 1150 $cm^{-1}$ are split bands and corresponds to $v_1$ and $v_3$ modes of unidentely coordinate ions. as in Fig. 1.20;

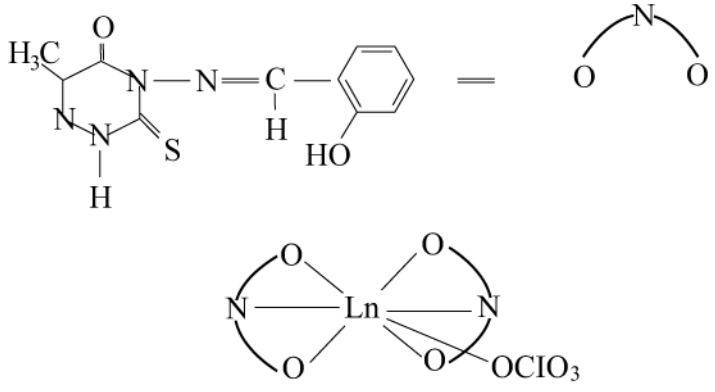
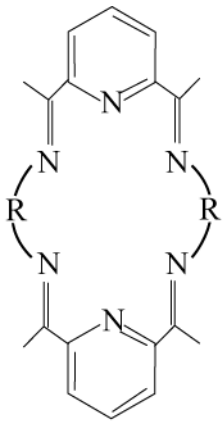



**Fig. 1.20**

(Macrocyclic complex of $[Ln(SMMT)_2ClO_4]$)

Synthesis of dinuclear copper(II) complexes with tetraimine shiff-base macrocyclic ligands derived from 2,6 diacety1 pyridine and 1, n-diamino-n'-hydroxyalkanes (nn'=3,2,4,2 and 5,3) complex $[Cu_2(HL^4)(H_2O)^2(ClO_4)_3]$ and $[Cu_2(H_2O)(ClO_4)_3]$ are indicated the coordination of perchlorate to the metal[106] ion. as shown fig 1.21;




$H_2L^4$;R = $CH_2CH_2CH(OH)CH_2CH_2$

**Fig. 1.21**

(Macrocyclic complex $[Cu_2(HL^4)(H_2O)^2(ClO_4)_3]$)

The Tetrahydroborate reduction of the schift base prepared by template cyclizations on lead(II) complexes[107]. as shown fig 1.22;

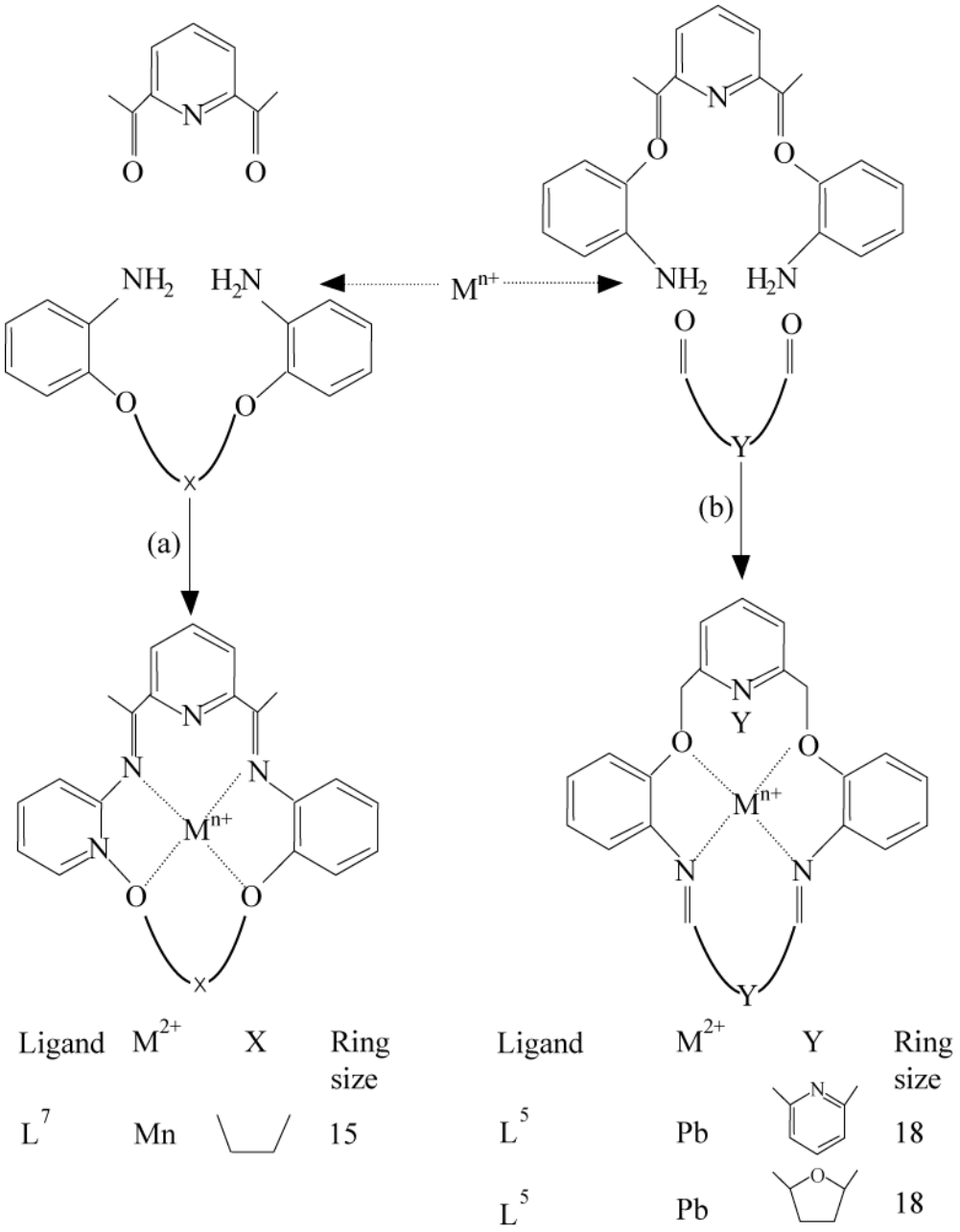



**Fig. 1.22**

(cyclizations reaction on lead(II) complexes)

The above complex coordinate the perchlorate to the metal, because of the suggest IR band at (1098-625) $cm^{-1}$.

In the literature it is reported that $[NiL^9(ClO_4)]$, $[CuL^9\ (ClO_4)]$ complexes are good agreement of coordinated perchlorate bond to the metal. (Were

$L^9$=1,8 dibenzoyl-2, 7-diphenyl-3,6 diazaoctane). The complexes showed absorption bond at (1100, 1080, 980) $cm^{-1}$ for $ClO_4$ moiety[106-110].

In the literature it is reported that $[Cu_2L^{10}(ClO_4)_2]$ complexes are perchlorate bond to the metal. On the basis of elemental analysis and electronic spectra and magnetic moment[111]. [Where $L^{10}$ = Bis-{1-hydroxyiminato-1,2-diphenyl (2-iminopyridy)}ethane].

The complex $[Ag(1NHSAL)_2(ClO_4)]$ and $[Ag(lNHHAP)_2ClO_4]$ have been synthesis[112] by adding ligand dropwise and mixed with metal perchlorate solution in the 2:1 molar ratio, (where ligand INHSAL = 2-hydroxybenzal dehyde) 1 NHHAP=2hydroxy acetophenone. On the basis of IR spectra showed a band at (1090-620) $cm^{-1}$ regions these are assignable that perchlorate coordinate to the metal.[113,114]

## 1.10 BIOLOGICAL ACTIVITY OF SOME IMPORTANT COMPOUNDS

Complex compound is very important in bioinorganic chemistry. Over the last decade or so there has been a growing awareness of the importance of wide range of metallic and non metallic elements in biological system[115]. Some 25 elements which are currently throughout to be essential to life, ten can be classified as trace metal ions; Fe, Cu, Zn, Mn, Co, Cr, Sn, V and Ni and four as bulk metal ions; Na, K, Mg and Ca. In addition there is some tentative evidence that Cd and Pb may be required at very low levels. There is also evidence that Sn, As and Br may possibly be essential trace elements. In the following section the out line of the chemistry and biological effects of some of the essential and polluting elements is given bellow.

A number of metal complexes and ligand have been shown to be chemically useful in variety of areas, e.g. As antitumor agent's antiviral agents and in the treatment of illness, for example, in haemocyanins, contain Cu and bind one molecule of $O_2$ for every pair of copper(I) ions. Haemocyanine is found only in molluscs and arthropods. Inorganic chemistry has been interested in developing suitable copper complexes which would minic some of properties of haemocyanin.[116]

In sufficient levels of cobalt in the diet gives rise to a disease, known as bush sickness in Australia and Newzealand. Trace amounts of vitamin $B_{12}$ is essential for the synthesis of hemoglobin by mammals. Implantation of cobalt powder has been reported to cause malignant tumors in muscles.

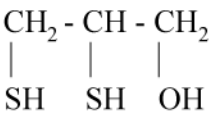
Metal complexation occupy an important role in almost all branches of medical and pharmaceutical activity. Prior to 1980, search for anticancer drug was focussed primarily on organic compounds[117]. However, with the discovery of cis-diaminechloroplatinum(II), which shows excellent antitumour activity, arose keen interest in exploring other inorganic compounds as possible therapeutic agents, Copper, Silver, Led, and Gold complexes are among the most promising inorganic compounds known to posses anticancer activity.

Copper is found in human cells and primarily associated with copper dependent enzymes that are required for normal metabolic process. The complexzation of Co, Fe, Mg, Zn and Cu with nitrogen containing chain in the enzymes are very diverse[118,119] . The antimalarial activities of a series of 2-acetyl pyridine and their Cu, Ni, Fe, Mn, complexes have tested for their antimalarial and antiteukemie properties. These compounds have been found to posses significant antimalarial activities[120]. Brada and Altman found copper containing compounds to be effective in preventing liver tumours. Iron (II) complex being much more active. The antineoplastic antibiotic bleomycin has shown to be more effective as it copper complex Rao et al[121]. investigated antitumor activity and cytotoxicity of blemoycin with some of metal complex using Ehrlich as cites tumors in nice and Ehrich cell in culture. They reported the following order for both antitumour activity and cytotoxicity

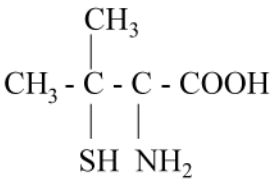
Cu(II)Blm>Zn(II)Blm>Fe(IIBlm>Co(II)Blm.

[Blm=Bleomycin]

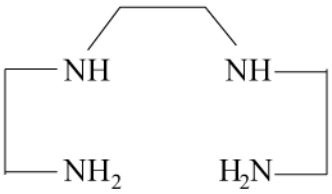
Although ions of cobalt copper, iron, zinc and manganese are essential for mammals, but above metal present in excess, become to Wilson disease leads to an expressive accumulation of copper in the liver, kidney and brain and leads to liver and kidney failure and various neurological abnormalities. The disease involves the administration of various cheating agents which are capable of mobilising the copper as follows:



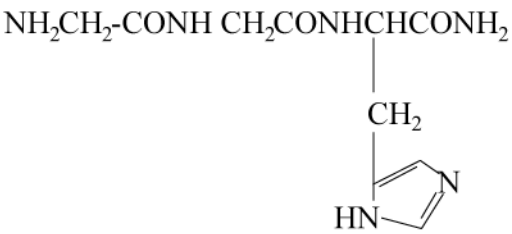

Dimercaarol (BAL)




D-penicillamine




1.8 diamino-16-diazaoctane




glyglyhis $NH_2$

Cancer is a disease characterized by uncontrolled muttriplication and spread with in the organism of apparently abnormal forms of the organism own cell. Many organic carcinogens are also excellent ligand. (Fig. 1.23) and the importance of coordination compounds, in cancer initiation is the subject of considerable debate.

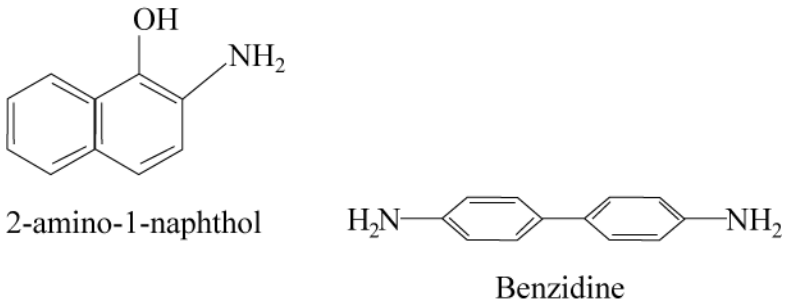



**Fig. 1.23**

(Bidentate Ligand)

From the above discussion it appears that the medicinal activity of metal complexes markedly influence the bio-availability of the drug in our body and provides diverse potential in the biological action of all living systems.

## 1.11 AIM OF THE PRESENT WORK

Macrocyclic complex compounds are an immense filed in chemistry, especially in coordination chemistry and in bio-inorganic chemistry.

In the nature a lot of macrocyclic compounds are known (e.g. haemoglobin, myoglobin, vitamin $B_{12}$ coenzymes where the metal atoms are Fe, Co and the basic macrocyclic unit is porphyrin but have different functional group to the macrocyclies). The chemical properties of some macrocyclic compounds resemble those of antibiotics.[122] For instance, macrolactones resemble the natural metabolites such as nonatine and monactine.

In the recent years considerable attention have been given to the synthesis of macrocyclic complex.[123-125] These complex compounds have been used an model system of biologically important materials, such as porphyrin and

corins. Some of the macrocyclic ligand cannot be easily prepared from the reactants.[126] In that case the complex compounds could be synthesised by template method. The desire macrocyclic ligand can be isolated by stripping the complex compounds.[127,128] Macrocyclic tetraaza complex of $Ni^{2+}$ act as catalyst to reduce $CO_2$ to CO and $Fe^{2+}$, $Mn^{3+}$ porphyrins have been most commonly studied catlyst.[129]

In view of the extensive use as drugs and significant pharmacological activities of macrocyclic complexes and their derivatives, it is desired to synthesize macrocyclic complexes of Ni (II), Cu (II) and Fe (II). The synthesized macrocyclic complexes and their derivatives are expected to have microbial activity.[130-131]

Therefore, considering the rapid increasing importance of macrocyclic ligand and their complexes in biology and in medicine the present work is divided in to two parts:

i. Firstly, synthesis of some new macrocyclic complexes by the reactions of malonodihydrazide with Ni(II), Cu(II) and Fe(II) perchlorate in the presence of formaldehyde, acetaldehyde, butyraldehyde will be characterised by elemental analysis, UV visible and IR spectral analysis, magnetic moment and conductance measurements and some other physical properties.

ii. Secondly, study of antibacterial activity of the synthesised complexes (some test organisms such as, *Salmonella-17, Klebsilla, Shigella dysenteriae, Shigella shiga, Shigella boydii, Shigella sonnei, Shigella flexneri, Escherichia coli, Pseudomonas aeruginosa, Salmonella, Bacillus megaterium, Sarcina lutea, Staphylococcus aureus, Bacillus cereus*) including the investigation of minimum inhibitory concentration of the complexes.

# METHODS AND MATERIALS

## 2.1 PHYSICAL MEASUREMENTS

### 2.1.1 Weighing

The weighing operation was performed on a electronic balance.

### 2.1.2 Melting Point Measurement

Melting point of the ligands and complexes were obtained with an electrothermal melting point, apparatus model No. A.Z 6512.

### 2.1.3 Infrared spectra

Infrared spectra disc were recorded as KBr with a SHIMADZU FTIR-8400 infrared spectrophotometer.

### 2.1.4 Conductivity

The conductivity cell was normally cleaned three times with water and finally rinsed three times with acetone and allowed to dry in air.

Conductivity measurements of the present complexes were carried out in dimethyl sulfoxide (DMSO). The conductivity viz. the molar conductivities were calculated by using the formula.

$\dot{k}$ = (1000/C) × cell constant × observed conductivity

Where ‘C’ represents the concentration of the respectively complex in mol/L.

Generally $10^{-3}$M solutions of complex were employed for this purpose. The conductance measurements were made at room temperature using by digital conductivity meter and a dip type cell with a polarized electrons. The cell was calibrated with 0.01N; 0.001N and 0.0001N potassium chloride solution and it has a cell constant of 1.065. The conductance of the pure solvent was also determined. The observed conductivity was obtained by subtracting the conductance of the pure solvent from the observed conductance of the solution of the complexes.

### 2.1.5 Magnetic moments

i. **Working principle of the balance:**

ii. The magnetic susceptibilities of the isolated complexes were determined by Gouy's method.

The following general expression for mass susceptibility in C.G.S units may be derived in the same manner for the traditional Gouy method.

$$\chi_g = \frac{1}{m}[C(R-R_o)+ \chi_{vair}A] \text{ ----------(i)}$$

Where,

C = Constant of proportionality

R = Susceptibility of the tube with sample

$R_o$= Susceptibility of the empty tube

l = Length of the sample (in cm)

m = Mass of the sample (in gm)

A = Cross-section area of the tub (in $cm^2$)

$\chi_{vair}$= Volume Susceptibility of the displaced air, for powdered sample the air correction term $\chi_{vair}$may normally be ignored.

C the const of proportionality is related to the calibration constant of a given balance by the formula.

$C = C_{Bal}/10^9$ ---------- (ii)

From (i) and (ii), we get

$\chi_g = C_{Bal} \times l \times (R\text{-}R_o)/10^9 \times m$ ---------- (iii)

**ii. Calibration of the balance**

The magnetic susceptibility Balance (M.S.B) must be calibrated at its intended work place. The balance is to be used mainly for solid sample, then a solid calibrant (preferably) [Hg $Co(SCN)_4$] is recommeded since some of the systemic errors in packing may cancel. The constancy of the calibration was checked using a sealed off sample of $MnCl_2$ solution.

**iii. Procedure**

1. The Zero knob of the magnetic susceptibility was turned until numerical display shows zero (000) and calibration sample [$HgCo(SCN)_4$] was inserted into sample holder. It was allowed settled reading the numerical display.
2. Reading was recorded and calibration constant was calculated from the formula.

   $C = Blac = C_{Tube}/(R\text{-}R_o)$

   $= (1766.842)/\{2830\text{-}(\text{-}17\}$

   $= 2.086$ ---------- (iv)

   From (iii) and (ii) we get,

   $\chi_g = 2.086x1x\ (R\text{-}Ro)/10^9 \times m$ ---------- (v)

**iii. Operation of the "Balance"**

1. The range knob was turned to the XI scale then it was allowed to 10 minutes warm up period before use.
2. The zero knob adjusted until the display reads 000. The zero was adjusted on each side.
3. An empty sample tube of known weight was placed into the tube guide and was taken the reading $R_o$.
4. The sample was packed and sample mass, m in grams and the sample length 1 in cm.
5. The packed sample tube was placed the tube guide and was taken the reading R.

The mass susceptibility, $\chi_g$ was calculated using.

$$\chi_g = 2.086 \times 1 \times (R\text{-}R_o)/10^9 \times m$$

The temperature was recorded from thermometer situated in the balance room.

**iv. The Magnetic Moment**

From the measurement of magnetic moment one can find the number of unpaired electrons present in the system and the possible configuration and also the structure.

If substance is placed in a field of intensity H gauss, then B, the magnetic induction of the field within the substance is given by,

$$B = H+4\pi l$$

Where, I = Intensity of magnetism induced by the field.

I / H = is called the volume susceptibility of the substance and is given the symbol $\chi_v$. In most cases a more useful quantity is the magnetic

susceptibility per unit mass susceptibility, $\chi_g$ equal to $\chi_{v/d}$ where d is the density of the substance in gm/cm$^3$. It was convenient to regard $\chi_v$ as dimensionless and $\chi_g$ as having the dimensions of reciprocal density.

The molar susceptibility $\chi_m$ is equal mass susceptibility $\chi_g$ multiplied by formula weight of the substance. ($\chi_m = \chi_g \times$ molecular weight)

Here $\mu_{eff} = 2.828 \sqrt{(\chi_m \times T)}$

The magnetic moment was calculated using the above equation

**Table 2.1: Unpaired spins and magnetic moments**

| No. of Unpaired electrons | Total spin angular moment (S) | Magnetic moments $\mu_s$ (Bohr Magnetons)(B.M) |
|---|---|---|
| 1 | 0.5 | 1.73 |
| 2 | 1 | 2.83 |
| 3 | 1.5 | 3.87 |
| 4 | 2 | 4.90 |
| 5 | 2.5 | 5.92 |

The idea on magnetic measurements can be applied to understand the stereochemistry of metal complex.

### 2.1.6 Analysis of TGA, DTA, XRD:

All the sample of thermal studies and XRD analysis of Dhaka university and and Rajshahi university.

### 2.1.7 Metal estimation:

A known weight of the complex was taken into a conical flask and to it concentrated, $H_2SO_4$ (0.5ml) was added. It was fumed down to dryness and the preocess was repeated. Conc $HNO_3$, (0.5ml) was then added and cone $HClO_4$ (0.5ml) were added too. The mixture was fumed to dryness. The process of the adding acids and funming down to dryness was continued until there was no black materials. Distilled water (100 ml) was added to dissolve the residue and then the metal was estimated complexometrically[92]. Using EDTA (EDTA = Ethylenediamine tetraacetic acid) and DMG (DMG= dimethyl glyoxime) excellent agreement of result were found.

### 2.1.8 Elemental analysis

Micro analysis for carbon hydrogen and nitrogen were obtained by using Kjeldahl Method[93].

## 2.2 PURIFICATION OF THE SOLVENTS[102]

### 2.2.1 Ethanol

About 1.25g of clean and dry magnesium turnings and 0.125g of iodine were placed in dry 500 mL round bottle flask containing 40 mL of reagent Ethanol. The flask was then fitted with a condenser carrying a calcium chloride guard tube on the top. The mixture was warmed until, the iodine had disappcared; heating was continued until all the magnesium was converted into ethoxied, then 250 mL of absolute ethanol was added and the mixture was refluxed for one hour. After cooling, the ethanol was distilled directly into a vessel in which it was stored, by resembling the condenser for

downward distillation via a splash head adapter. Then the dry ethanol was collected into a receiving flask from which it was stored into an air tight bottle.

### 2.2.2 Acetone

The acetone was heated under reflux with successive quantities of potassium per manganese until the violet coloured persisted. It was the dred with anhydrous potassium carbonate, Filtered from the desiccant and distilled. Precaution was taken to exclude moisture. i.e a calcium chloride guard tube was used.

## 2.3 NAME OF THE CHEMICALS/REAGENTS USED AND SUPPLIERS

| | |
|---|---|
| Cadmiam perchlorate Cd $(ClO_4)_2$ | Flleuka |
| Silver perchlorate $Ag(ClO_4)$ | E. Merck |
| Platenum perchlorate $Pt(ClO_4)_3$ | E. Merck |
| Chromium Perchlorate $Cr(ClO_4)_3$ | E. Merck |
| Gold Perchlorate $Au(ClO_4)_3$ | E. Merck |
| Led Perchlorat $Pb(ClO_4)_2$ | E. Merck |
| Zinc Perchlorat Zn(ClO | E. Merck |
| Lanthanum perchlorat $La_2(ClO_4)_3$ | E. Merck |
| Cerium perchlorat $Ce_2(ClO_4)_3$ | Flleuka |
| Gadolinium perchlorat $Gd_2(ClO_4)_3$ | E. Merck |
| Lanthanum carbonate $La_2(CO_3)_3$ | Flleuka |
| Cerium perchlorat $Ce_2(CO_3)_3$ | Flleuka |
| Gadolinium perchlorat $Gd_2(CO_3)_3$ | E. Merck |
| Cadmiam carbonate $CdCO_3$ | Flleuka |
| Silver carbonate $Ag_2CO_3$ | Flleuka |
| Platenum carbonate $Pt_2(CO_3)_3$ | E. Merck |
| Chromium carbonate $Cr_2(CO_3)_3$ | E. Merck |
| Zinc carbonate $ZnCO_3$ | E. Merck |
| Gold carbonate $Au_2(CO_3)_3$ | Flleuka |
| Led carbonate $PbCO_3$ | E. Merck |
| Propionaldehyde ($CH_3$ $CH_2$-CHO) | E. Merck |
| Butyraldehyde ($CH_3$ $CH_2$-$CH_2$CHO) | E. Merck |
| Crotonaldehyde($CH_3$-CH=CH.CHO) | E. Merck |
| Benzaldehyde ($C_6H_5CHO$) | E. Merck |

| | |
|---|---|
| Cinnamaldehyde($C_6H_5$.CH=CHCHO) | E. Merck |
| Ethanol(Absolute) | E. Merck |
| Etheline diamine | E. Merck |
| 1,8 Diaminonaphthaline | E. Merck |
| Urea | E. Merck |
| Thiourea | E. Merck |
| Malonic acid | E. Merck |
| Perchloric acid | E. Merck |
| 2,3 Butanedione | E. Merck |
| 3,4-Haxanedione | E. Merck |
| 2,6-Diamino pyridine | E. Merck |

**Chemicals for Biological activities**

| | |
|---|---|
| NaCl | E. Merck |
| Beef Extract | E. Merck |
| Peptone | E. Merck |
| Agar | E. Merck |
| Disk(Blank) | E. Merck |
| Antibiotic Disc | E. Merck |
| Molar Hilton agar | E. Merck |
| Yeast Extract | E. Merck |
| Bactrotry ptone | E. Merck |
| Bato yeast extract | E. Merc |
| Chloroform | E. Merck |
| Acetone | E. Merck |
| Pet-ether ($40^0$C) | BDH |

| | |
|---|---|
| Absolute ethanol | BDH |
| Dimethyl sulfoxide (DMSO) | E. Merck |
| Formaldehyde | James Burrough Ltd. (England) |
| Acetaldehyde | BDH |
| Cinnamaldehyde | BDH |
| Hydrazine hydrate | E. Merck |
| Dimethylformamide (DMF) | BDH |
| Diethylmalonate | May & Baker Ltd. |

All chemicals were used as supplied except purification of the solvent.

# EXPERIMENTAL

## 3.1 PREPARATION OF COMPLEXES

### 3.1.1 Preparation of malonodihydrazide[105] $C_3H_8N_4O_2$.

Diethyl malonate (8.080 g, 50 mmol) and hydrazine hydrate (5.006 g, 100 mmol) were mixed together in a beaker with constant stirring at ambient temperature. The reaction was carried out without solvent. The precipitation was stared to form when the solution was turned from yellowishting to shite. After the precipitation the product was filtered off on a buckner funnel and washed with ethanol three times and dried in a vacuum desiccator over anhydrous $CaCl_2$. A silky white amorphous product 8.800 g was obtained (60% yield). The melting point of the compound was recorded at $125^0$C-$130^0$C.

The substance was soluble in water and Dimethyl formamide(DMF) and insoluble in ethanol, methanol, dichlomethane, diethylether, petether, carbontetrachloride and n-hexane.

### 3.1.2 Preparation of Metal Perchlorate.

Metal perchlorate was prepared from analytically pure metal carbonate by treatment with 70% perchloric acid (AR). The treatment was done in the following way:

About 100 mL 70% perchloric acid was taken in a 250 mL beaker and than metal carbonate e.g. copper carbonate was added slowly with continuous stirring. The addition of copper carbonate was continued until the bubbles were disappears. Access amount of copper carbonate was added and kept it 12 hours to ensure the complete of the reaction. Than water-alcohol mixture was added with continuous steering and filtered. The resulting solids were filtered, washed with ethanol till free from excess acid and recrystallized several times from ethanol. (Copper perchlorate is partially soluble in alcohol). Copper perchlorate solution was also standardized by iodometric titration. Nickel perchlorate and Iron perchlorate were prepared with the same method.

## 3.2 PREPARATION OF MACROCYCLIC COMPLEXES OF Pb(II).

### 3.2.1 Preparation of $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Lead(II) perchlorate (1.542 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A blue precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.2.2 Preparation of $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

To the solution of malonodihydrazide (0.528 g, 4 mmol in 10 ml water) acetaldehyde (0.176 g, 4 mmol in 10 mL water) was added. To the above mixture with Lead(II) perchlorate hexhahydrate (1.028) g, 2 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for 12 hours and cooled down. A gray colour precipitate was formed. The precipitation was filtered off on a buckner funnel was washed with water and ethanol three times. The product was dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $255^0$C and yield was 0.860 g (60%). The compound was soluble in DMSO and insoluble acetone, alcohol, chloroform.

### 3.2.3 Preparation of [Pb($C_{14}H_{28}N_8O_4$)($ClO_4$)$_2$] complex 3

To the solution malonodihydrazide, $C_3H_8N_4O_2$ (0.528 g, 4 mmol 10 in mL water) and cinnamaldehyde (0.528 g, 4 mmol) solution was added. To above minture Lead(II) perchlorate (1.028 g, 2 mmol in 10 mL water) and the whole mixture was refluxed with constant stirring for 3 hours and cooled down. The yellow precipitate was formed. The product was filtered off on a buckner funnel and washed with water and ethanol three times. The product was dried in a vacuum desiccator over andydrous $CaCl_2$.

The melting point of the compound was $250^0$C and yield was 0.92 g (60%). The compound was soluble in DMSO and insoluble in chloroform, dimethylformamide and acetone.

### 3.2.4 Preparation of [Pb($C_{14}H_{24}N_8O_4$)($ClO_4$)$_2$] complex 4.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Lead(II) perchlorate (1.542 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A blue precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.2.5 Preparation of [Pb($C_{24}H_{28}N_8O_4$)($ClO_4$)$_2$] complex 5.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Lead(II) perchlorate (1.542 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A blue precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.2.6 Preparation of [Pb($C_{20}H_{24}N_8O_4$)($ClO4$)$_2$] complex 6.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Lead(II) perchlorate (1.542 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A blue precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

## 3.3 PREPARATION OF MACROCYCLIC COMPLEXES OF Zn(II).

### 3.3.1 Preparation of $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Zinc(II) perchlorate (0.792 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A light yellow precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.3.2 Preparation of $[Zn(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

To the solution of malonodihydrazide (0.528 g, 4 mmol in 10 ml water) acetaldehyde (0.176 g, 4 mmol in 10 mL water) was added. To the above mixture with Zinc(II) perchlorate (0.528 g, 2 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for 12 hours and cooled down. A read colour precipitate was formed. The precipitation was filtered off on a buckner funnel was washed with water and ethanol three times. The product was dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $255^0$C and yield was 0.860 g (60%). The compound was soluble in DMSO and insoluble acetone, alcohol, chloroform.

### 3.3.3 Preparation of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

To the solution malonodihydrazide, $C_3H_8N_4O_2$ (0.528 g, 4 mmol 10 in mL water) and cinnamaldehyde (0.528 g, 4 mmol) solution was added. To above minture Zinc(II) perchlorate (0.528 g, 2 mmol in 10 mL water) and the whole mixture was refluxed with constant stirring for 3 hours and cooled down. The yellow precipitate was formed. The product was filtered off on a buckner funnel and washed with water and ethanol three times. The product was dried in a vacuum desiccator over andydrous $CaCl_2$.

The melting point of the compound was $250^0$C and yield was 0.92 g (60%). The compound was soluble in DMSO and insoluble in chloroform, dimethylformamide and acetone.

### 3.3.4 Preparation of $[Zn(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Zinc(II) perchlorate (0.792 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A white precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.3.5 Preparation of [$Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2$] complex 5.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Zinc(II) perchlorate (0.793 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A yellow precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.3.6 Preparation of [$Zn(C_{20}H_{24}N_8O_4)(ClO_4)_2$] complex 6.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$ (0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Zinc(II) perchlorate ( 0.793 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A light yellow precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

## 3.4 PREPARATION OF MACROCYCLIC COMPLEXES OF La(II).

### 3.4.1 Preparation of $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Lanthanum (II) perchlorate (1.014 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A white precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.4.2 Preparation of $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

To the solution of malonodihydrazide (0.528 g, 4 mmol in 10 ml water) acetaldehyde (0.176 g, 4 mmol in 10 mL water) was added. To the above mixture with Lanthanum (II) perchlorate (0.676 g, 2 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for 12 hours and cooled down. A yellow colour precipitate was formed. The precipitation was filtered off on a buckner funnel was washed with water and ethanol three times. The product was dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $255^0$C and yield was 0.860 g (60%). The compound was soluble in DMSO and insoluble acetone, alcohol, chloroform.

### 3.4.3 Preparation of [La($C_{14}H_{28}N_8O_4$)($ClO_4$)$_2$] complex 3

To the solution malonodihydrazide, $C_3H_8N_4O_2$ (0.528 g, 4 mmol 10 in mL water) and cinnamaldehyde (0.528 g, 4 mmol) solution was added. To above minture Lanthanum (II) perchlorate (0.676 g, 2 mmol in 10 mL water) and the whole mixture was refluxed with constant stirring for 3 hours and cooled down. The white precipitate was formed. The product was filtered off on a buckner funnel and washed with water and ethanol three times. The product was dried in a vacuum desiccator over andydrous $CaCl_2$.

The melting point of the compound was $250^0$C and yield was 0.92 g (60%). The compound was soluble in DMSO and insoluble in chloroform, dimethylformamide and acetone.

### 3.4.4 Preparation of [La($C_{14}H_{24}N_8O_4$)($ClO_4$)$_2$] complex 4.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Lanthanum (II) perchlorate (1.014 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A yellow precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.4.5 Preparation of [La($C_{24}H_{28}N_8O_4$)($ClO_4$)$_2$] complex 5.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Lanthanum (II) perchlorate (1.014 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A white precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.4.6 Preparation of [La($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] complex 6.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Lanthanum (II) perchlorate(1.014 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A yellow precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

## 3.5 PREPARATION OF MACROCYCLIC COMPLEXES OF Cd(II).

### 3.5.1 Preparation of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$ (0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Cadmium (II) perchlorate (0.9342 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A white precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.5.2 Preparation of $[Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

To the solution of malonodihydrazide (0.528 g, 4 mmol in 10 ml water) acetaldehyde (0.176 g, 4 mmol in 10 mL water) was added. To the above mixture with Cadmium (II) perchlorate (0.6228 g, 2 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for 12 hours and cooled down. A yellow colour precipitate was formed. The precipitation was filtered off on a buckner funnel was washed with water and ethanol three times. The product was dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $255^0$C and yield was 0.860 g (60%). The compound was soluble in DMSO and insoluble acetone, alcohol, chloroform.

### 3.5.3 Preparation of $[Cd(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

To the solution malonodihydrazide, $C_3H_8N_4O_2$ (0.528 g, 4 mmol 10 in mL water) and cinnamaldehyde (0.528 g, 4 mmol) solution was added. To above minture Cadmium (II) perchlorate (0.6228 g, 2 mmol in 10 mL water) and the whole mixture was refluxed with constant stirring for 3 hours and cooled down. The read precipitate was formed. The product was filtered off on a buckner funnel and washed with water and ethanol three times. The product was dried in a vacuum desiccator over andydrous $CaCl_2$.

The melting point of the compound was $250^0$C and yield was 0.92 g (60%). The compound was soluble in DMSO and insoluble in chloroform, dimethylformamide and acetone.

### 3.5.4 Preparation of $[Cd(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Cadmium (II) perchlorate (0.9342 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A blue precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.5.5 Preparation of [Cd($C_{24}H_{28}N_8O_4$)($ClO_4$)$_2$] complex 5.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Cadmium (II) perchlorate (0.9342 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A yellow colour precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.5.6 Preparation of [Cd($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] complex 6.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$ (0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Cadmium (II) perchlorate (0.9342 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A black precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

## 3.6 PREPARATION OF MACROCYCLIC COMPLEXES OF Ag (II).

### 3.6.1 Preparation of [Ag ($C_8H_{16}N_8O_4$)($ClO_4$)$_2$] complex 1.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$ (0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Silver(II) perchlorate (0.921 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A white precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.6.2 Preparation of [Ag($C_{10}H_{20}N_8O_4$)($ClO_4$)$_2$] complex 2

To the solution of malonodihydrazide (0.528 g, 4 mmol in 10 ml water) acetaldehyde (0.176 g, 4 mmol in 10 mL water) was added. To the above mixture with Silver(I) perchlorate (0.614 g, 2 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for 12 hours and cooled down. A yellow colour precipitate was formed. The precipitation was filtered off on a buckner funnel was washed with water and ethanol three times. The product was dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $255^0$C and yield was 0.860 g (60%). The compound was soluble in DMSO and insoluble acetone, alcohol, chloroform.

### 3.6.3 Preparation of $[Ag(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

To the solution malonodihydrazide, $C_3H_8N_4O_2$ (0.528 g, 4 mmol 10 in mL water) and cinnamaldehyde (0.528 g, 4 mmol) solution was added. To above minture Silver(I) perchlorate (0.614 g, 2 mmol in 10 mL water) and the whole mixture was refluxed with constant stirring for 3 hours and cooled down. The yellow precipitate was formed. The product was filtered off on a buckner funnel and washed with water and ethanol three times. The product was dried in a vacuum desiccator over andydrous $CaCl_2$.

The melting point of the compound was $250^0$C and yield was 0.92 g (60%). The compound was soluble in DMSO and insoluble in chloroform, dimethylformamide and acetone.

### 3.6.4 Preparation of $[Ag(C_{14}H_{24}N_8O_4)](ClO_4)_2]$ complex 4.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Silver(I) perchlorate (0.921 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A yellow precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.6.5 Preparation of $[Ag(C_{24}H_{28}N_8O_4)](ClO_4)_2]$complex 5.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$ (0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Silver(I) perchlorate (0.921 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A yellow precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

### 3.6.6 Preparation of $[Ag(C_{20}H_{24}N_8O_4)](ClO_4)_2]$complex 6.

To the aqueous malonodihydrazide, $C_3H_8N_4O_2$(0.792 g, 6 mmol in 10 mL water) formaldehyde solution (0.48 g, 6 mmol 37%) was added. To the above solution Silver(I) perchlorate hexahydrate (0.921 g, 3 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A blue precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

## 3.7 TEMPLATE SYNTHESIS OF NEW MACROCYCLIC COMPLEXES OF METAL (II) IONS.

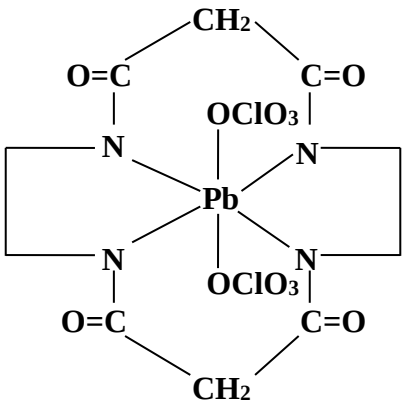
### 3.7.1 Preparation of $[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ complex 1.

To the aqueous Lead(II) perchlorate (1.082 g, 2 mmol in 10 mL water) and ethylene diamine (0.24 g, 4 mmol in 10 mL water) was added. To the above solution malonic acid (0.416 g, 4 mmol in 10 mL water) was added and the whole mixture was refluxed with constant stirring for two hours and cooled down. A blue precipitate was formed immediately. The product was washed with ethanol for three times and dried in a vacuum desiccator over anhydrous $CaCl_2$.

The melting point of the compound was $270^0$C and yield was 1.894 g (80%). The compound was soluble in DMSO and insoluble in acetone, ethanol, water and chloroform.

**Reaction of the complex:**

$NH_2\text{-}CH_2CH_2\text{-}NH_2 + HOOC\text{-}CH_2\text{-}COOH + Pb(ClO_4)_2 \longrightarrow$



# RESULT AND DISCUSSION

## 4.1 MACROCYCLIC COMPLEXES OF Pb(II)

Reactions of malonodihydrazide with Pb(II) perchlorate hexahydrate in presence of formaldehyde, acetaldehyde, butanal dehyde, crotonaldehyde, cinnamaldehyde, and benzaldehyde give some 16 member macrocyclic complex as described in sec.3.2

Complexes (1-6) are characterized on the basis of elemental analysis, magnetic moment & conductance measurements, UV-visible spectra & infrared studies, thermal studies and other physical properties, like melting point, solubility, colour etc.

Molar conductance data of the complexes (1-6) are shown in Table 4.1.1. The conductance values of the complexes suggested that they are non-electrolytic in nature[24].

The infrared spectra of the complexes (1-6) are shown as spectral data (Table 4.1.4) of the complexes showed a strong and broad band at (3246-3265) $cm^{-1}$ which is assigned for the ν(NH) stretching[25].

Due to coordination the ν(N-H) stretching of the amide group goes to the higher field at (3246, 3265) $cm^{-1}$ region as compared to the starting material malonodihydrazide[26]. In the complexes the terminal-$NH_2$ group of malonodihydrazide condensed with the aldehyde moiety form new secondary1-NH groups which may appear at the same region (or overlape) as to the amide-NH group as a result the ν(N-H) band appear as a strong and

broad band. [The starting material malonodihydrazide have three ν(N-H) bands at (3248, 3213, 3050) $cm^{-1}$. The bands at (3248, 3050) $cm^{-1}$ for the asymmetrie and symmetric ν(N-H) stretching of the terminal-$NH_2$ moiety and 3213 $cm^{-1}$ for amidic (N-H) group]. The complexes showed a broad band at (2920-2972) $cm^{-1}$ is suggested for the ν(C-H) stretching of aliphatic moiety[32]. The complexes showed a strong band at (1649-1674) $cm^{-1}$ which represent the ν(C=O) of NH-NH-CO-$CH_2$ moity[27]. Three or four band at (625-1145) $cm^{-1}$ region also indicated the $\nu_1,\nu_2,\nu_3,\nu_4$ bands of ($ClO^-_4$) moiety. These stretching frequency is suggested the coordination of perchlorate to the metal through the O atom[28]. A medium band at (407-412) $cm^{-1}$ region is tentatively attributed to the ν(M-N) mode[29,30.] indicating the coordination of the ligand to the metal through the nitrogen atom.

The magnetic moment measurement data (Table 4.1.3) of the Pb(II) complexes (1-6) showed (1.56-1.78) B.M. These values correspond to no unpaired electrons of Pb(II) $d^{10}$system suggest the octahedral environment of the complexes which are consistent with the literature value[1]. The elemental analyses (C, H and N) (Table 4.1.2) and metal estimation data (Table 4.1.3) of the complexes are consistent with the proposed formula.

The UV-visible spectra of the complexes (1-6) are shown (Table 4.1.5) band at 320,480 nm, (1-6) at represent the d-d transition of $^4A_{2g}$ (F) $\rightarrow$ $^4T_{1g}$(F), $^4A_{2g}$(F) $\rightarrow$ $^4T_{1g}$(P), which suggested the octahedral geometry of the Pb (II) complexes[31,32].

Thermal studies: The thermal properties of metal (II) complexes were investigated by thermograms (TGA, DTA) and are shown in (Fig 4.13-4.18) and the corresponding thermal analysis is presented in (Table.4.1.6).
In the case of complex (1-6) the decomposition occurs in the (230-325)$^0$C range. There is no mass loss up to 230$^0$C. The first stage of decomposition starts at 230$^0$C and end at 230$^0$C with a corresponding weight loss 25%. Which is accompanied by endothermic effect in the DTA curve in the range 225$^0$C which is accompanied by weight loss confirming the second stage of decomposition is observed at 225-350$^0$C (60% wt. loss).meanwhile the DTA curve exhibits endothermic effect in the range 325$^0$C which is accompanied by weight loss confirming.

On the basis of elemental analysis magnetic moment and conductance measurements, thermal studies UV Visible spectra, infrared spectra and other physical properties the suggested structure of the complexes are octahedral in nature as in Fig.4.19.

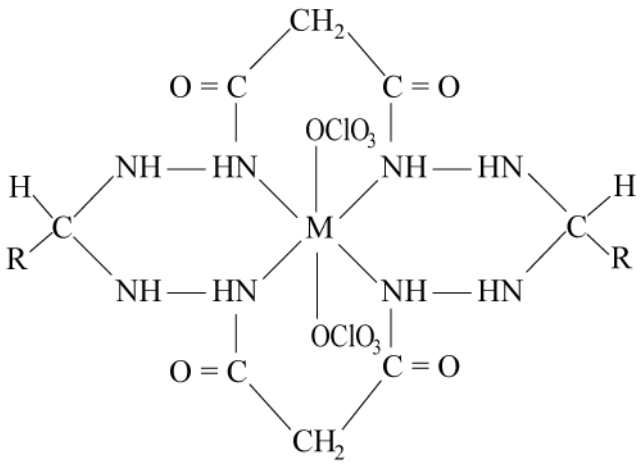



Fig. 4.19

M =Pb (II), where R=H(1),$CH_3$(2),$CH_3CH=CH$,-(3)$C_6H_5CH=CH_2$-(4),$CH_3$-$CH_2$-$CH_2$-(5),$C_6H_6CH$(6)

Table- 4.1.1: Analytical data and other physical properties of compounds (1-6)

| No. | Compounds | % Yield | Colour | Milting point $^0C$ | % M | | Molar conductance ohm$^{-1}$cm$^2$mol$^{-1}$ |
|---|---|---|---|---|---|---|---|
| | | | | | Calculated | Found | |
| 1 | $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 80 | Light blue | 225 | 17.87 | 17.80 | 3.23 |
| 2 | $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 60 | Off whit | 250 | 15.75 | 15.72 | 1.93 |
| 3 | $[Pb\ (C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 75 | Yellow | 190 | 11.21 | 11.23 | 2.87 |
| 4 | $[Pb(\ C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 70 | Light yellow | 210 | 8.59 | 8.60 | 1.79 |
| 5 | $[Pb\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 85 | Whit | 265 | 21.63 | 21.61 | 2.54 |
| 6 | $[Pb\ (C_{14}H_{\ 28}N_8O_4)(ClO_4)_2]$ | 75 | Light blue | 250 | 15.49 | 15.40 | 1.78 |

Table- 4.1.2: Elemental analysis data of compounds (1-6)

| No. | Compounds | %C | | %H | | %N | |
|---|---|---|---|---|---|---|---|
| | | Calculated | Found | Calculated | Found | Calculated | Found |
| 1 | $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 16.18 | 16.20 | 2.86 | 2.86 | 20.07 | 20.00 |
| 2 | $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 36.13 | 36.15 | 3.51 | 3.51 | 14.04 | 14.03 |
| 3 | $[Pb\ (C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 19.65 | 19.60 | 2.89 | 2.89 | 20.27 | 20.2 |
| 4 | $[Pb(\ C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 20.72 | 20.74 | 3.44 | 3.44 | 19.29 | 19.27 |
| 5 | $[Pb\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 19.38 | 19.35 | 3.23 | 3.23 | 22.62 | 22.60 |
| 6 | $[Pb\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 41.19 | 41.20 | 4.00 | 4.00 | 16.01 | 16.00 |

Table- 4.1.3: Magnetic moment data of compounds (1-6)

| No. | Compounds | Sample length, *l* in cm | Weight of the sample, m in gm | Susceptibility of the empty tube, Ro | Susceptibility of the sample with tube ,R | Mass Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | Molecular weight,M | Molar Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | μeff B.M |
|---|---|---|---|---|---|---|---|---|---|
| 1 | $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0695 | -48 | -22 | 0.0082 | 694 | 5.755 | 0.117 |
| 2 | $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2.1 | 0.0692 | -47 | -23 | 0.0072 | 722 | 5.252 | 0.111 |
| 3 | $[Pb\ (C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0596 | -40 | -21 | 0.0052 | 778 | 5.245 | 0.098 |
| 4 | $[Pb(\ C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0559 | -46 | -24 | 0.0046 | 774 | 3.574 | 0.092 |
| 5 | $[Pb\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0630 | -42 | -20 | 0.0052 | 898 | 4.669 | 0.105 |
| 6 | $[Pb\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 1.7 | 0.0589 | -46 | -23 | 0.0048 | 942 | 4.525 | 0.103 |

Table- 4.1.4: Important infrared spectral bands of compounds (1-6)

| No. | Compounds | $\nu$(C-H) $cm^{-1}$ | $\nu$(C=O $cm^{-1}$) | $\nu$(N-H) $cm^{-1}$ | $\nu$(M-N) $cm^{-1}$ | $\nu(ClO_4)$ $cm^{-1}$ |
|---|---|---|---|---|---|---|
| 1 | $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 3047 | 1664 | 3251 | 420 | 1180,1089,623 |
| 2 | $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2920 | 1649 | 3282 | 430 | 1105,979,623 |
| 3 | $[Pb\ (C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 2960 | 1640 | 3250 | 416 | 1150,1060,620 |
| 4 | $[Pb(\ C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 2967 | 1650 | 3261 | 408 | 1160,1040,623 |
| 5 | $[Pb\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 3040 | 1660 | 3254 | 409 | 1140,1080,621 |
| 6 | $[Pb\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 3070 | 1680 | 3270 | 406 | 1170,1090,625 |

Table- 4.1.5: U.V- Visible adsorption maxima of compounds (1-6)

| No. | Compounds | λ max (n,m) |
|---|---|---|
| 1 | $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ ] | 300,520 |
| 2 | $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2$ | 290,500 |
| 3 | $[Pb\ (C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 290,520 |
| 4 | $[Pb(\ C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 290,520 |
| 5 | $[Pb\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 292,490 |
| 6 | $[Pb\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 290,495 |

Table- 4.1.6: Thermal analysis data of compounds (1-6)

| No. | Compounds | %M Ligang | | | %M Metal oxide(MO) | | |
|---|---|---|---|---|---|---|---|
| | | $Tem^0C$ | Calculated | Found | $Tem^0C$ | Calculated | Found |
| 1 | $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 135.02 | 83.22 | 83.20 | 599.00 | 37.66 | 37.60 |
| 2 | $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 135.00 | 86.21 | 86.20 | 599.00 | 36.14 | 36.10 |
| 3 | $[Pb\ (C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 130.90 | 87.14 | 87.10 | 550.60 | 33.72 | 33.60 |
| 4 | $[Pb(\ C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 130.45 | 88.91 | 88.50 | 450.00 | 29.06 | 29.00 |
| 5 | $[Pb\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 140.50 | 88.23 | 88.20 | 530.00 | 30.86 | 30.40 |
| 6 | $[Pb\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 137.45 | 87.07 | 87.00 | 540.00 | 33.89 | 33.30 |

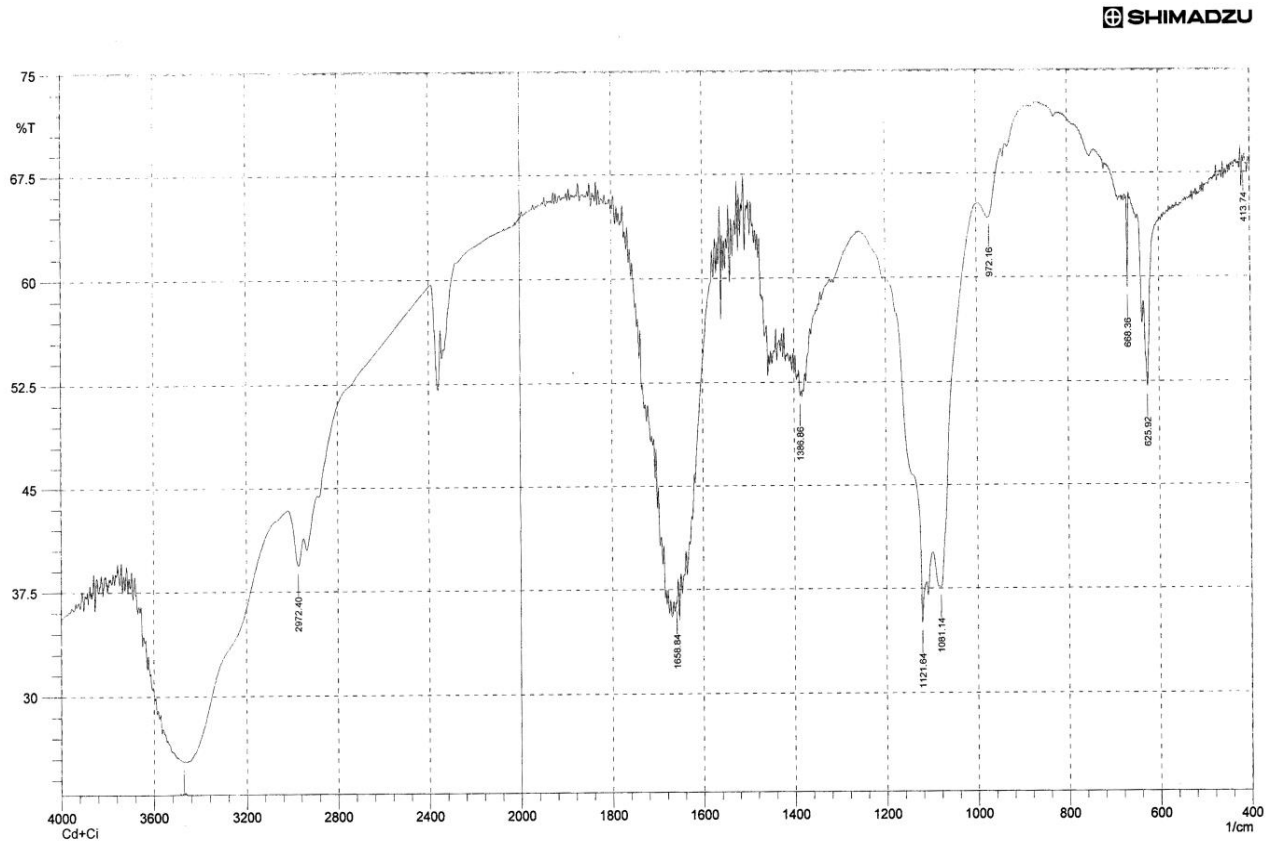



Fig-4.1:Infrared spectrum $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex1

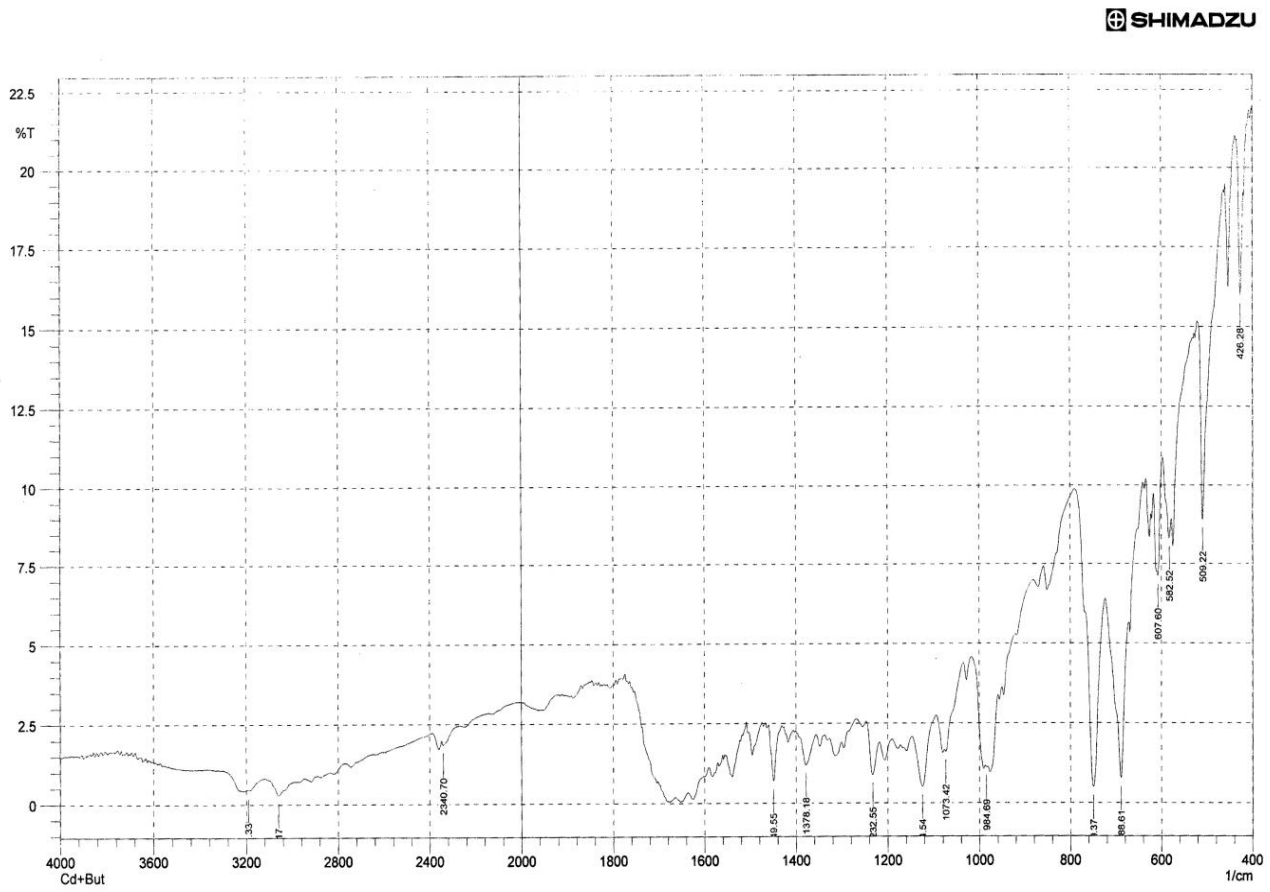



Fig-4.2: Infrared spectrum $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

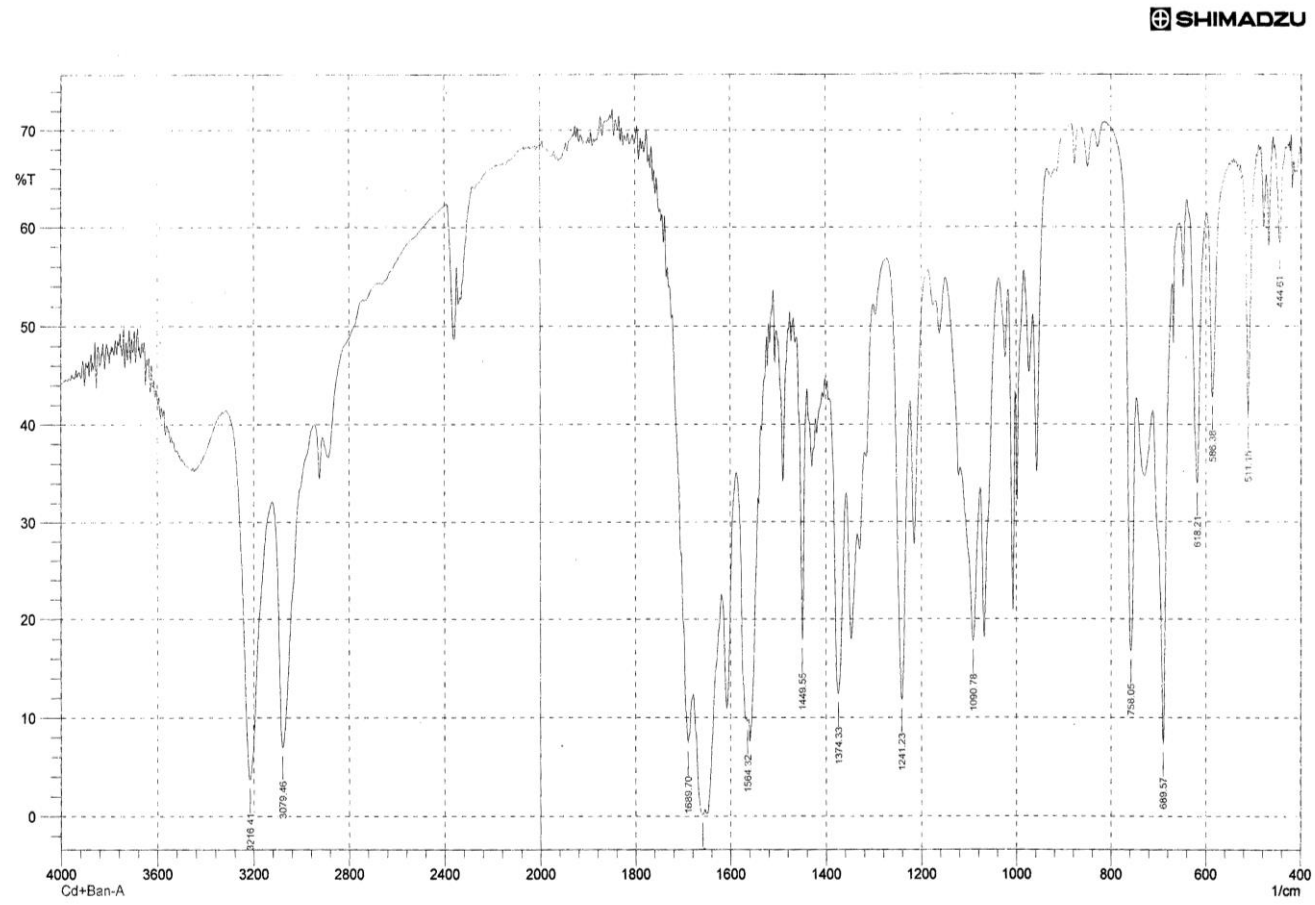



Fig-4.3: Infrared spectrum $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

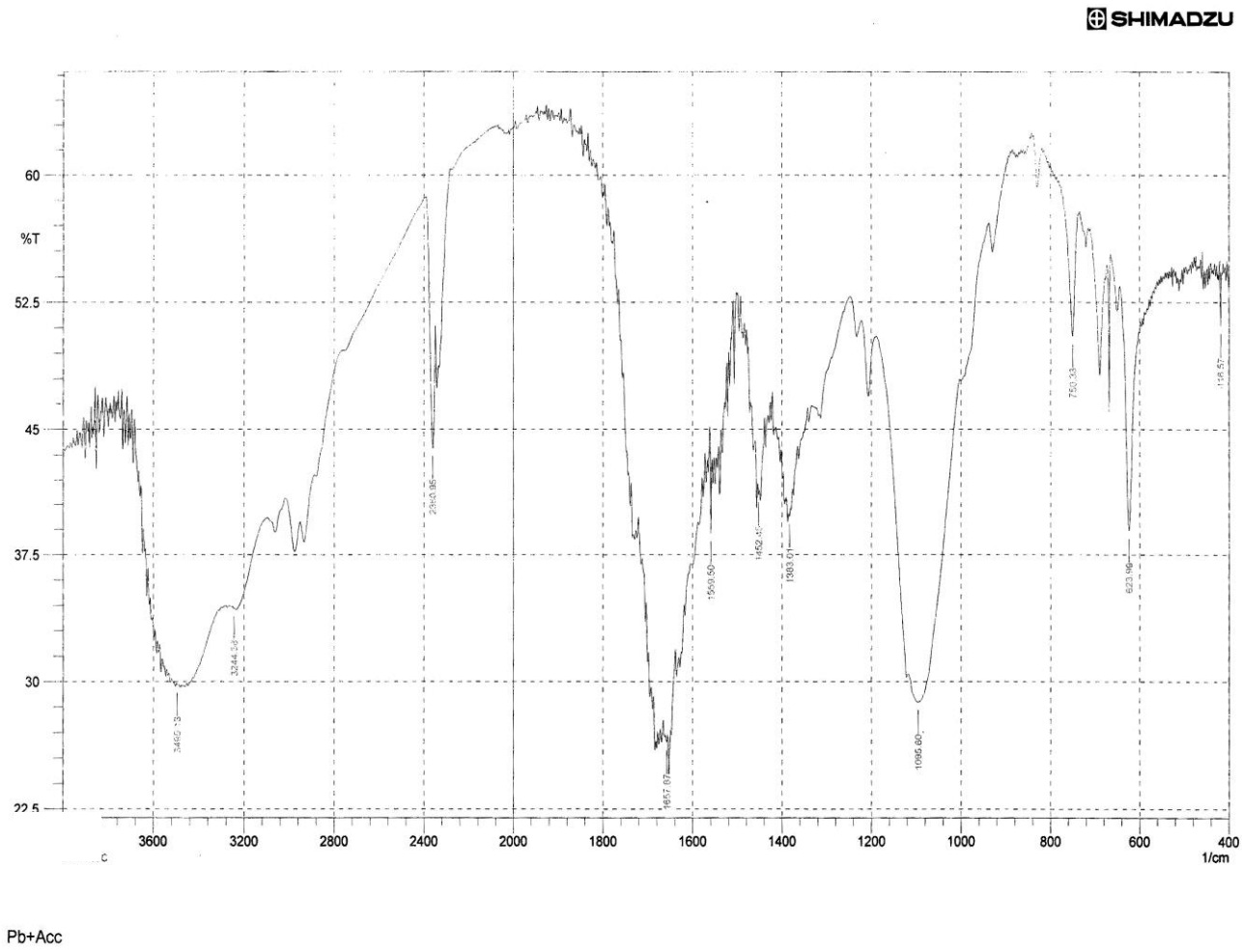



Fig-4.4: Infrared spectrum $[Pb(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4.

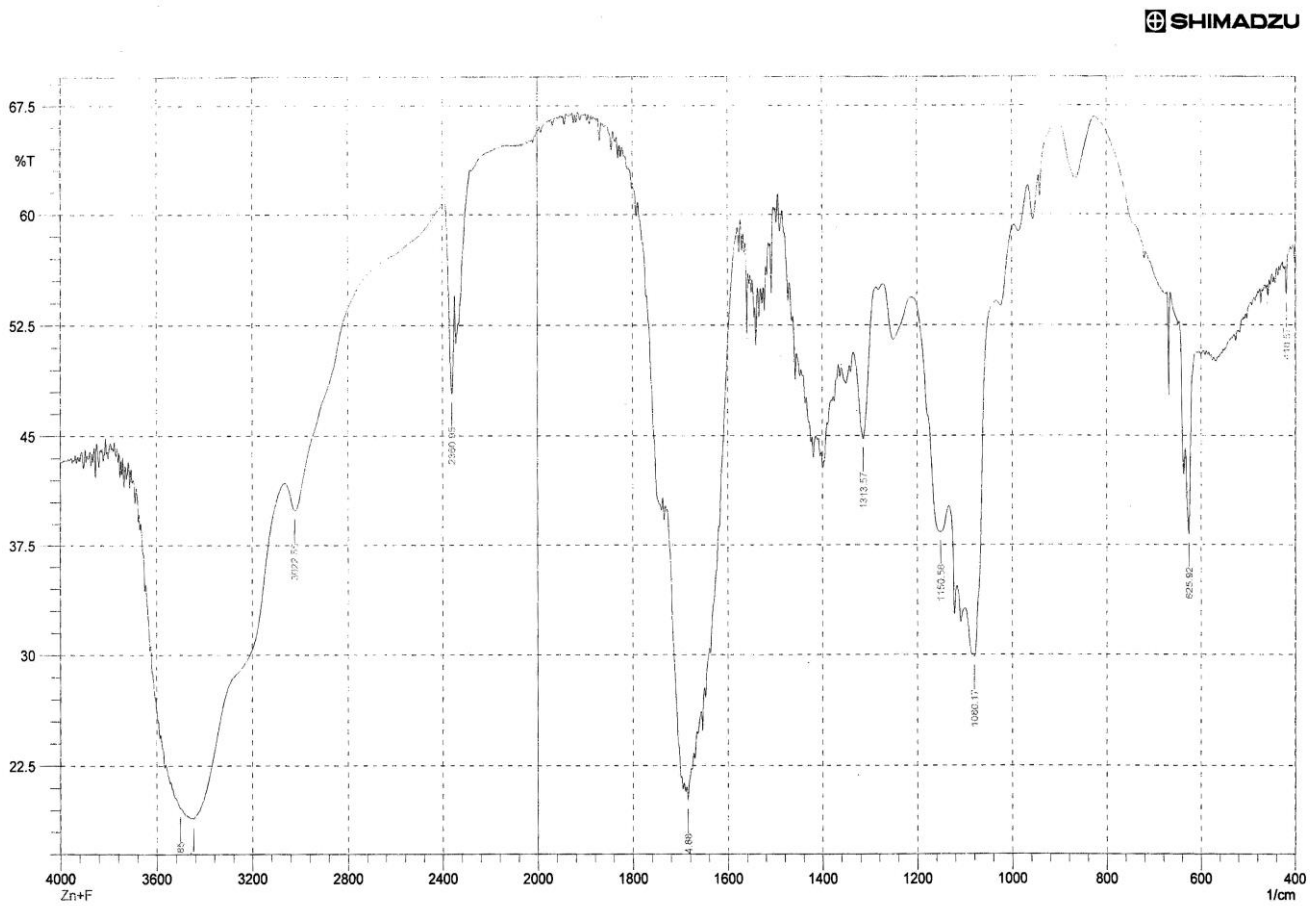



Fig-4.5:Infrared spectrum $[Pb(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5.

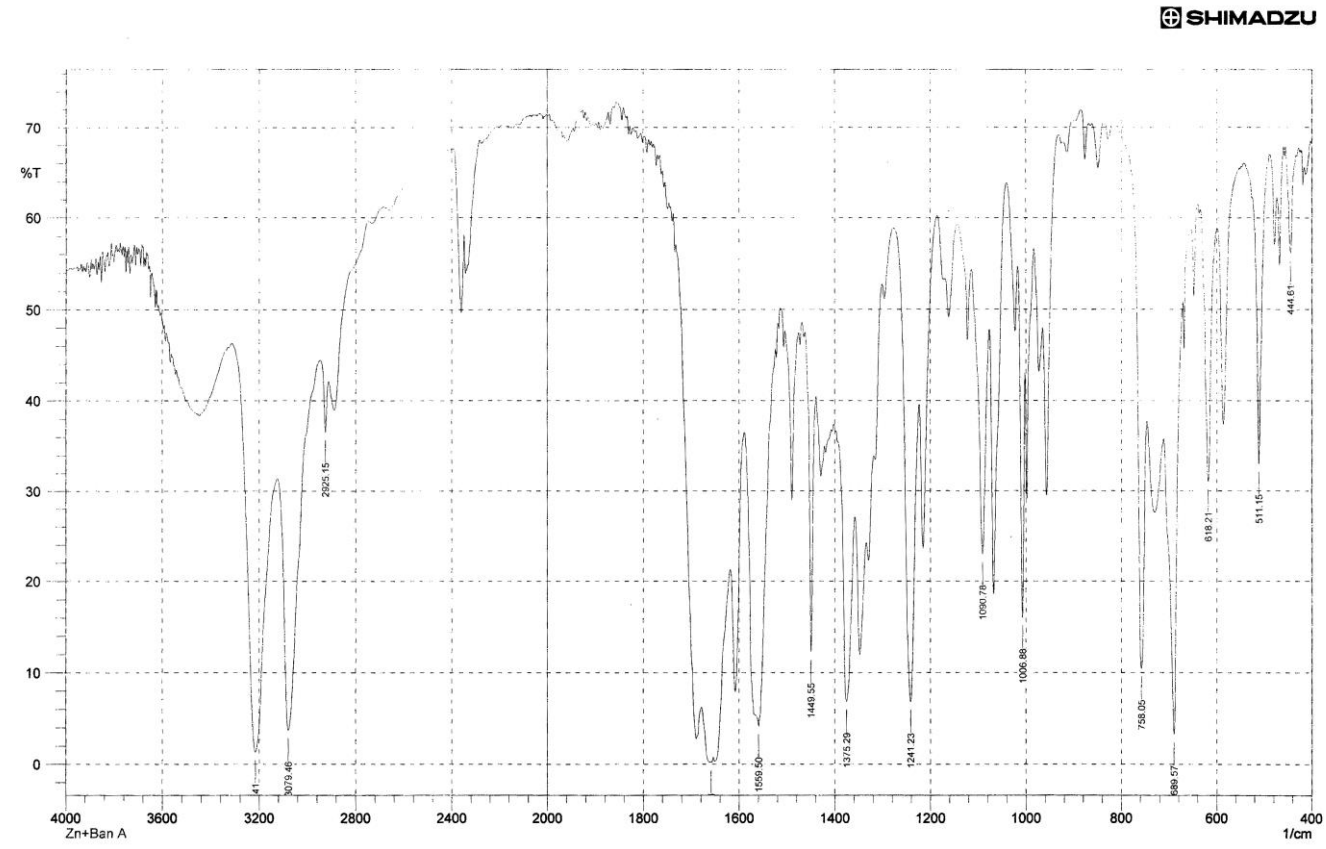



Fig-4.6:Infrared spectrum $[Pb(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex 6.

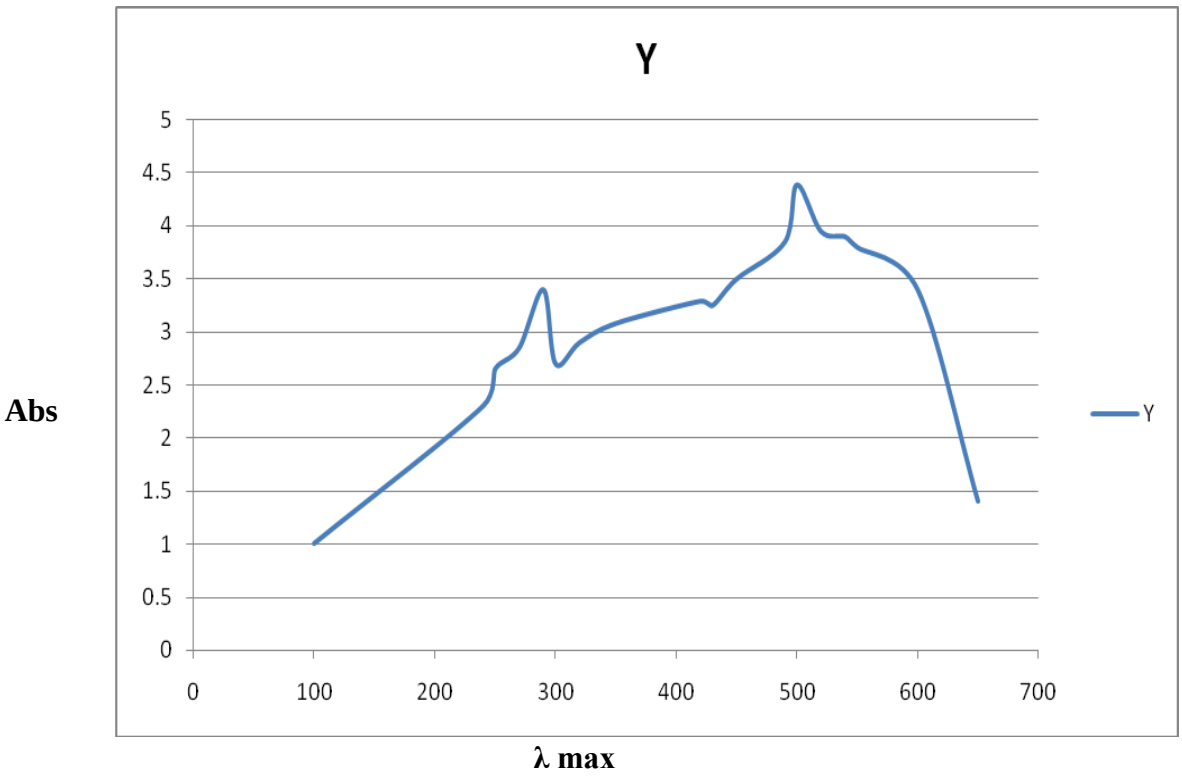



Fig-4.7:UV- Visible spectrum $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1

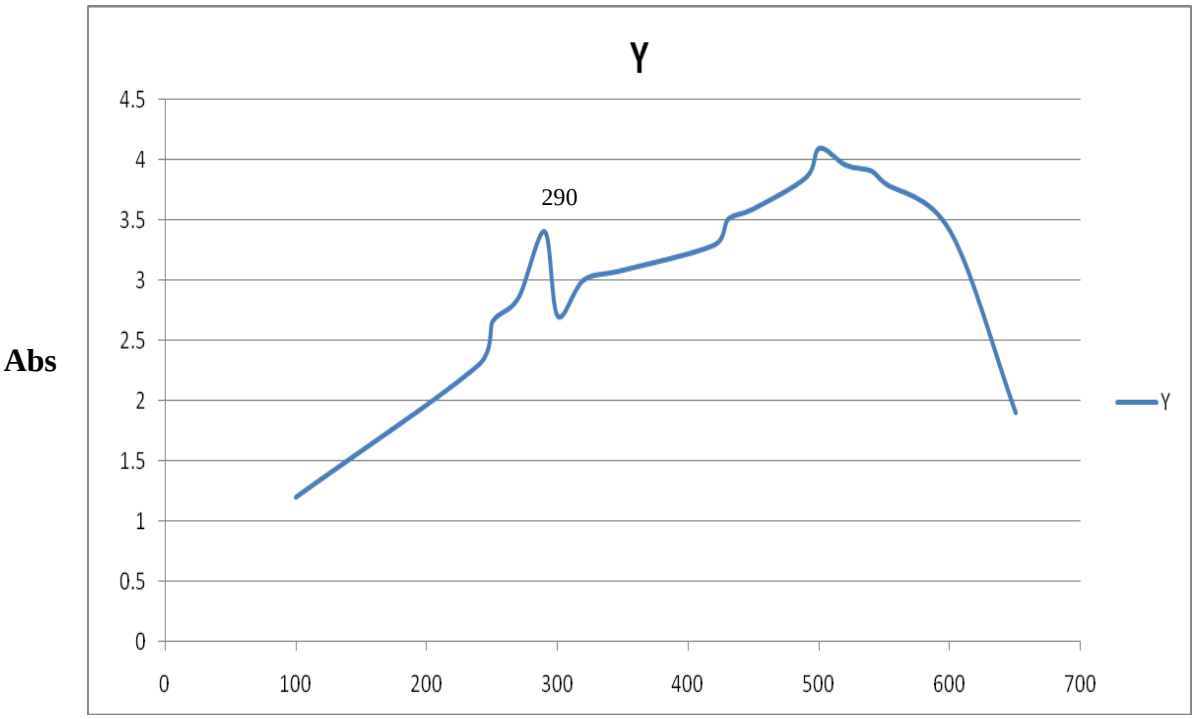



Fig-4.8 :UV- Visible spectrum of $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

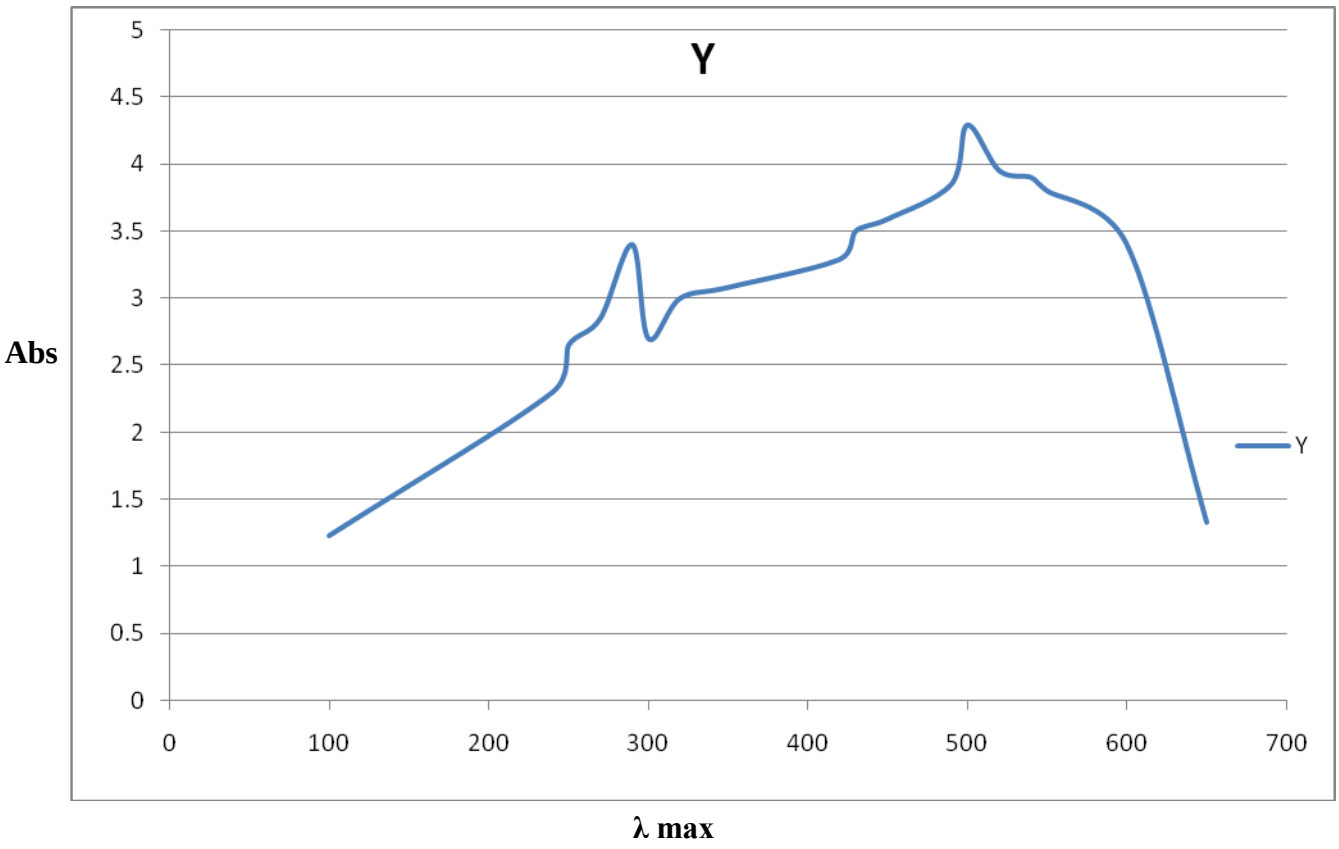



Fig-4.9 :UV- Visible spectrum of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

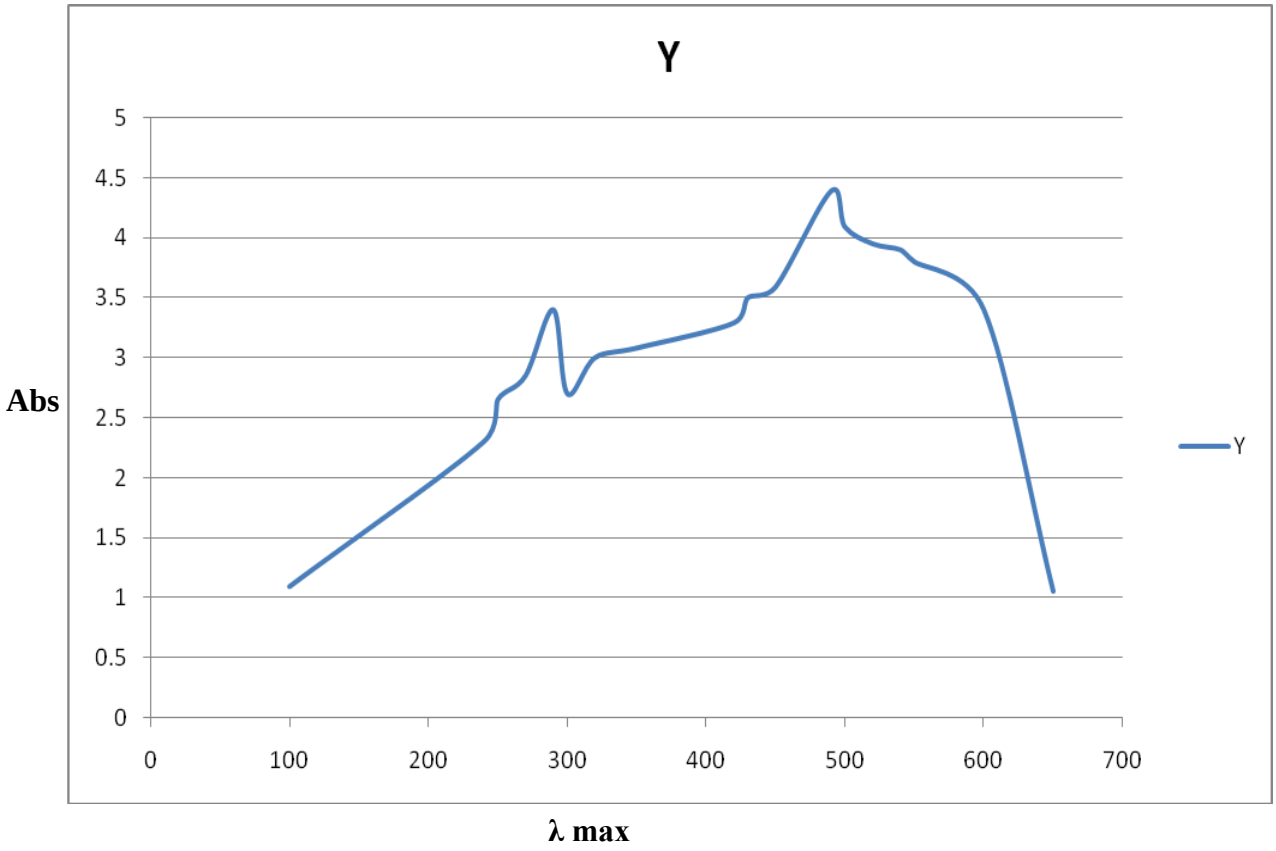



Fig-4.10 :UV- Visible spectrum Of $[Pb(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4.

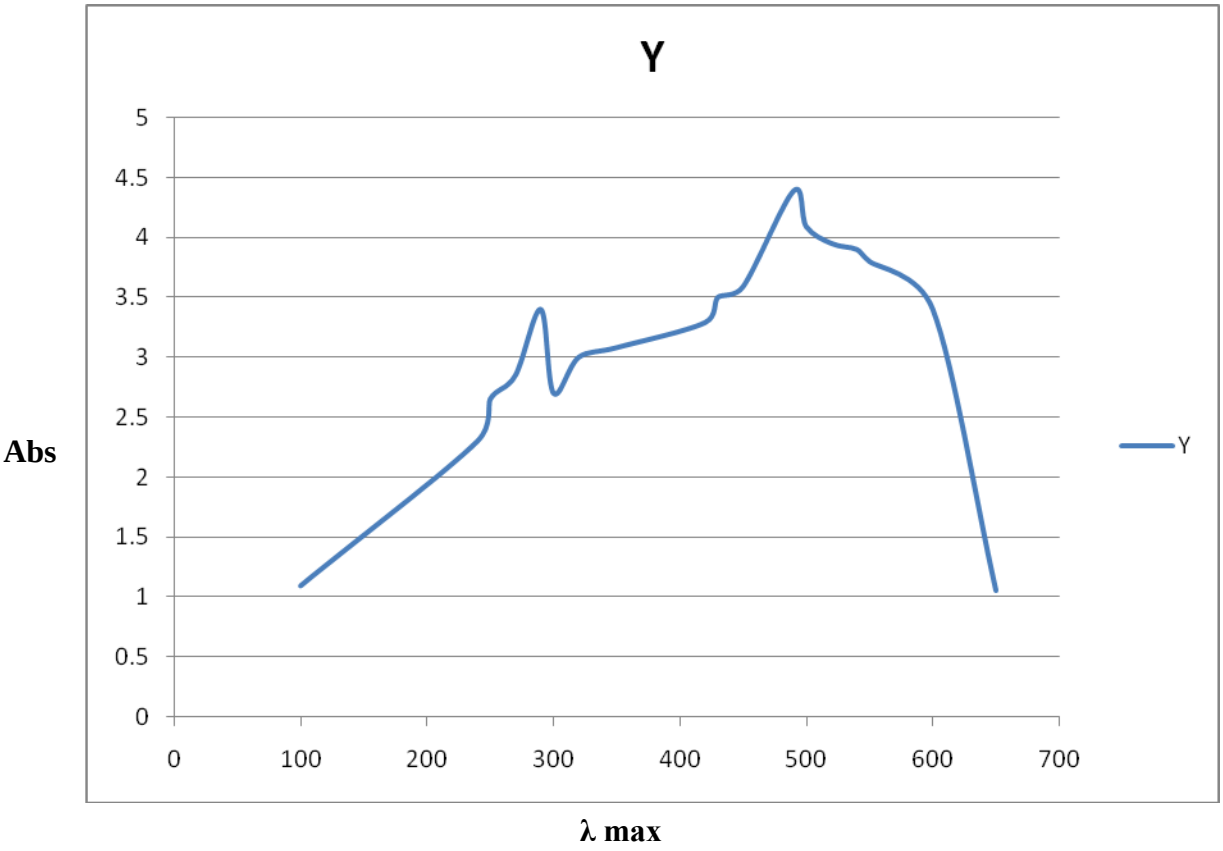



Fig-4.11 :UV- Visible spectrum of $[Pb(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5**.**

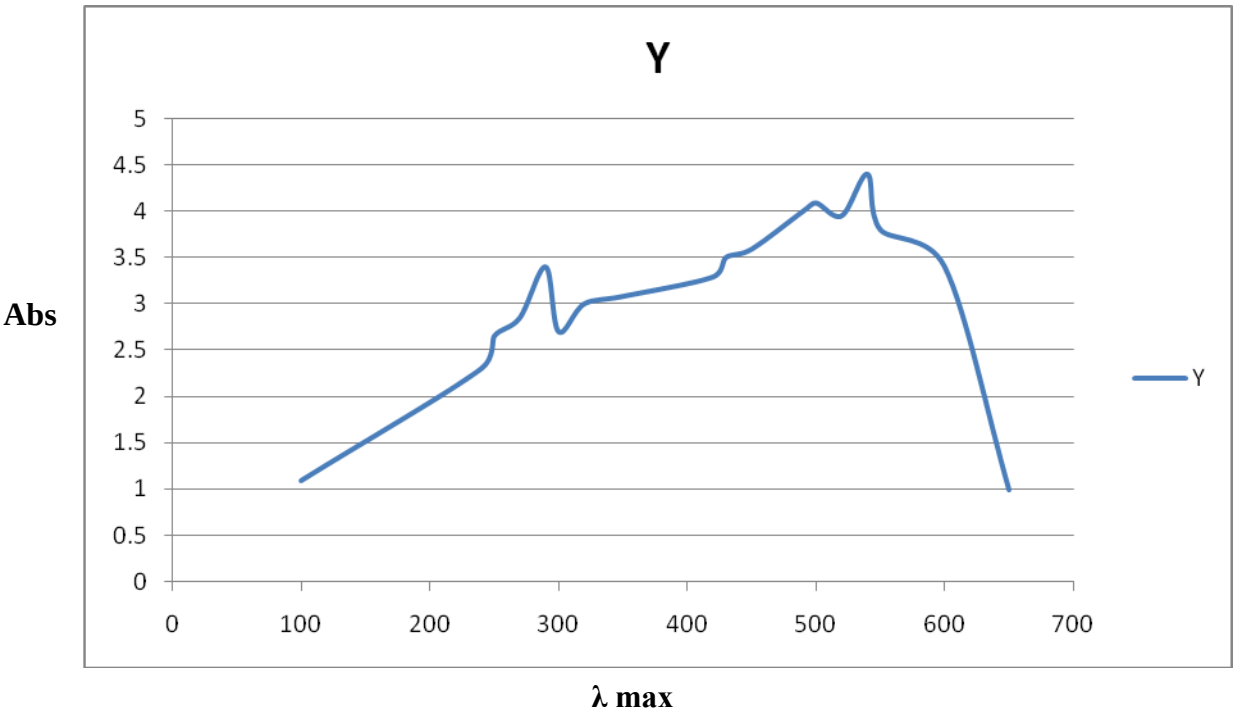



Fig-4.12 : UV- Visible spectrum of $[Pb(C_{20}H_{24}N_8O_4)(ClO4)_2]$ complex 6.

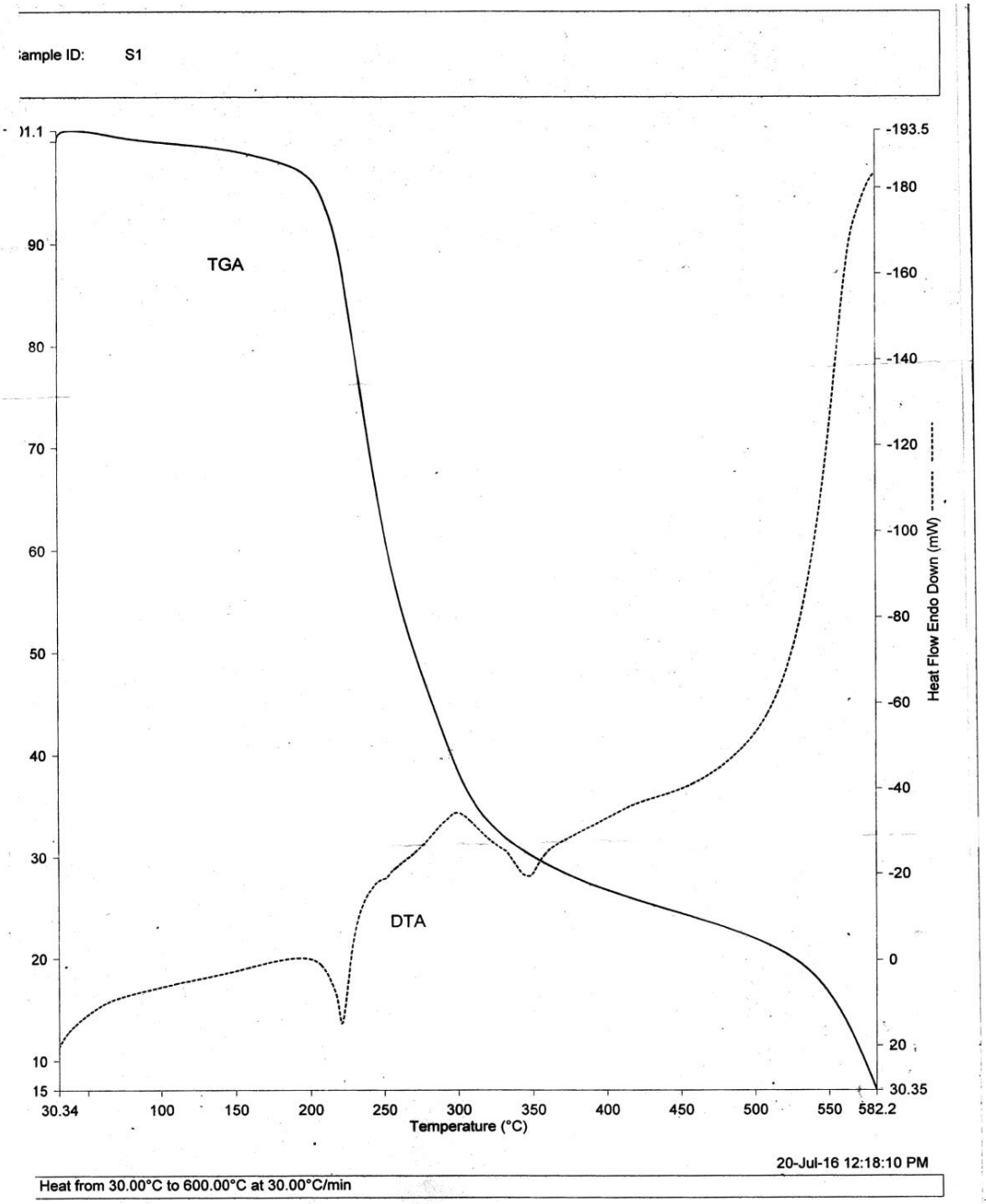



Fig-4.13: TGA & DTA spectrum $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

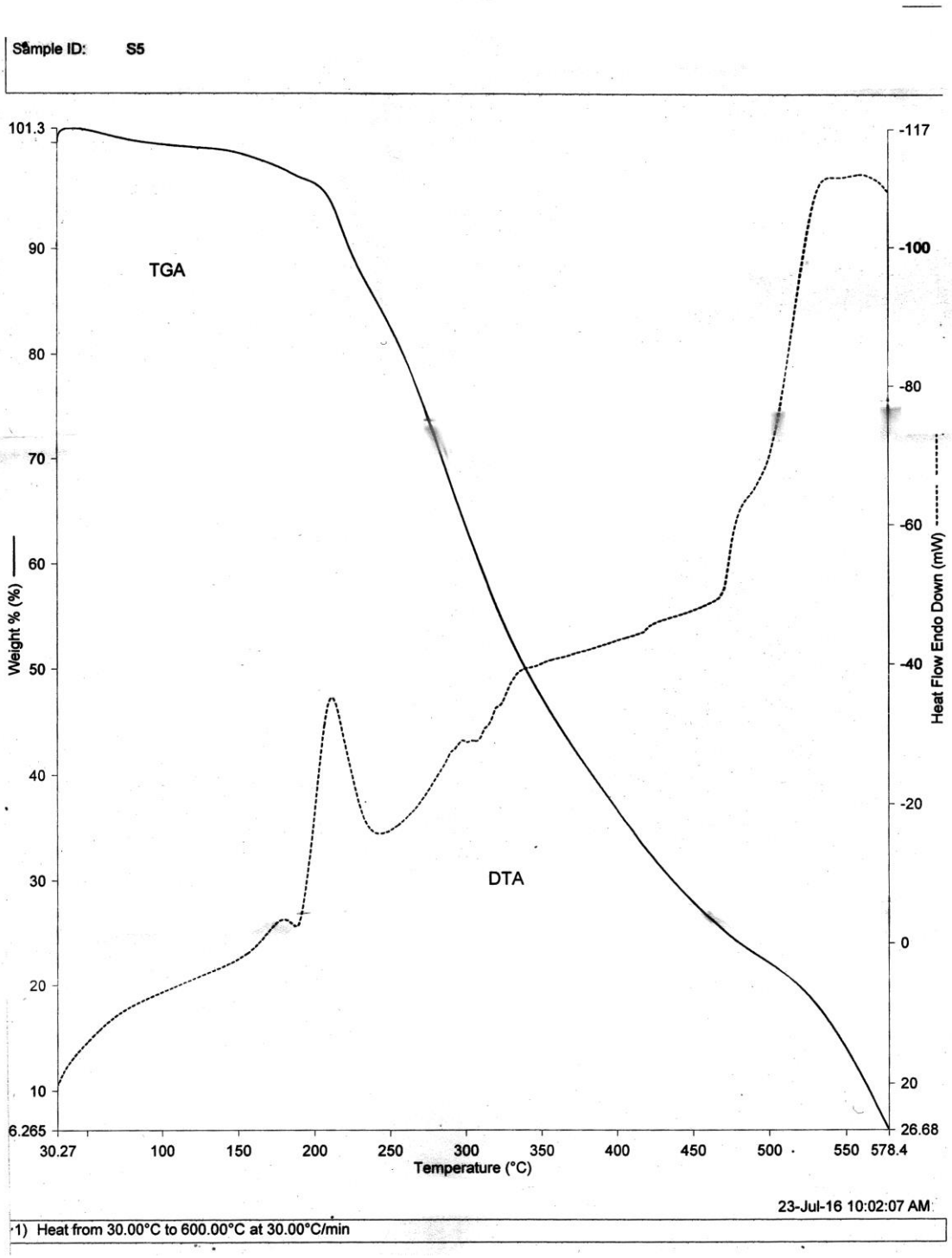



Fig-4.14: TGA & DTA spectrum $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

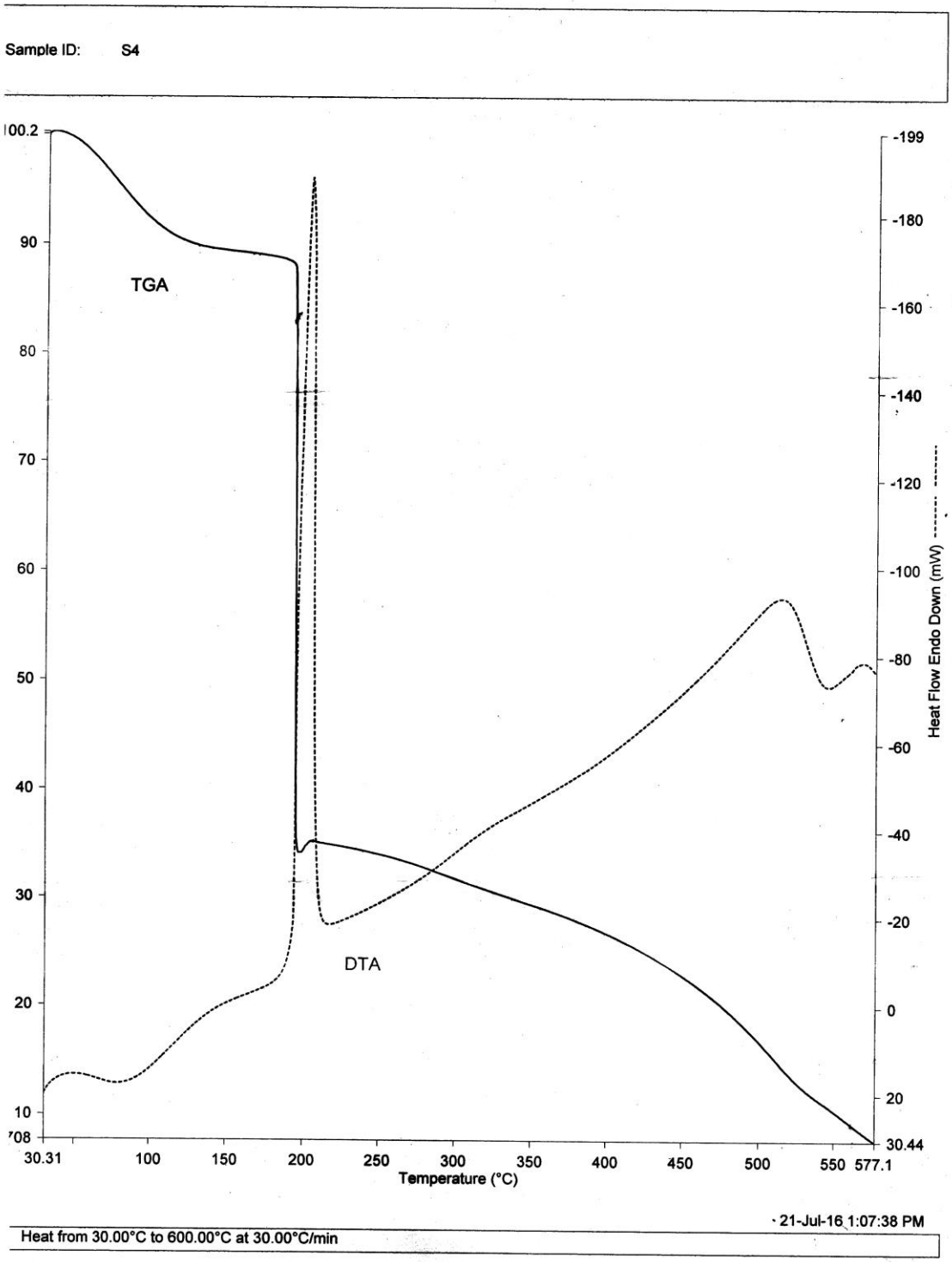



Fig-4.15: TGA & DTA spectrum $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

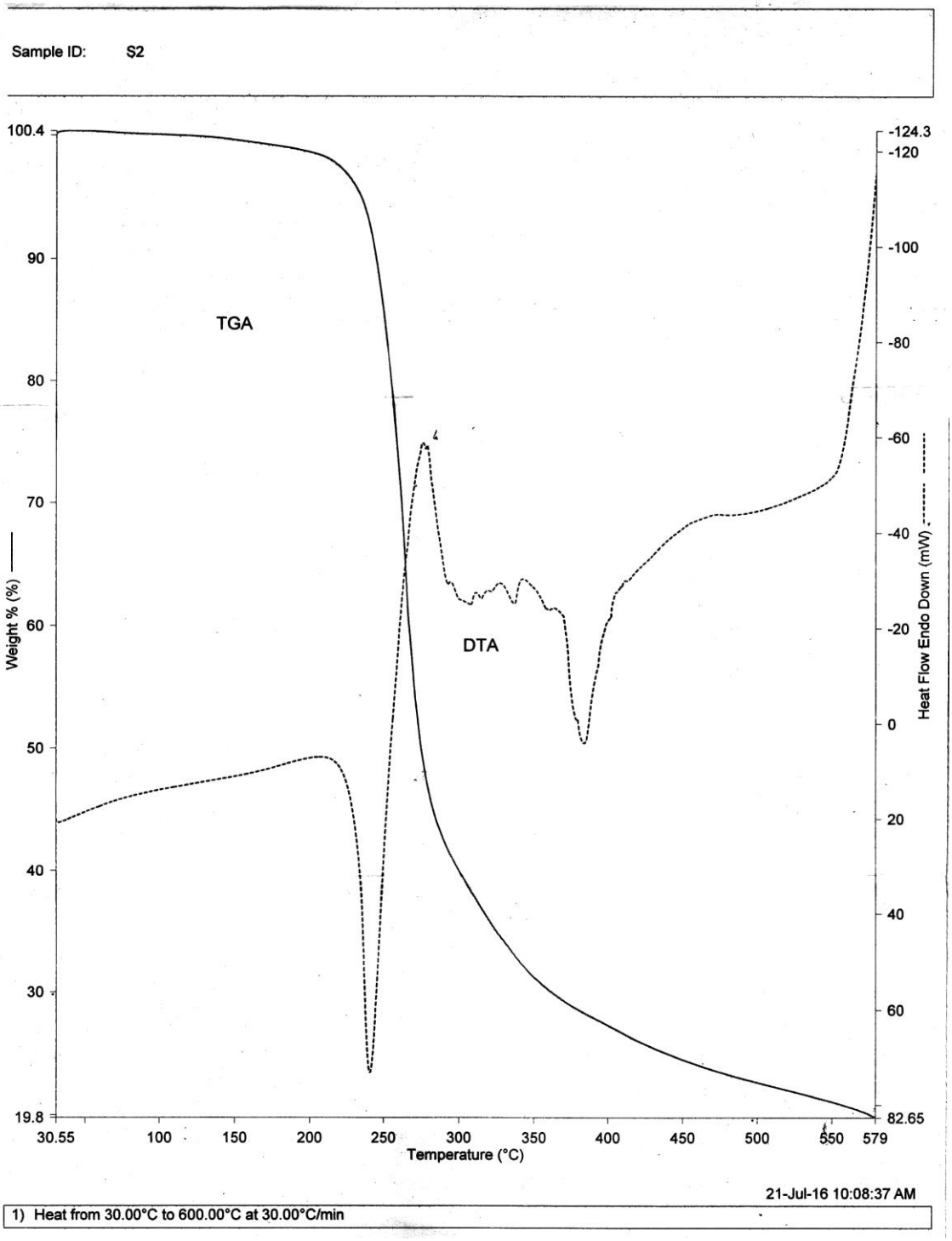



Fig-4.16: TGA & DTA spectrum $[Pb(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4

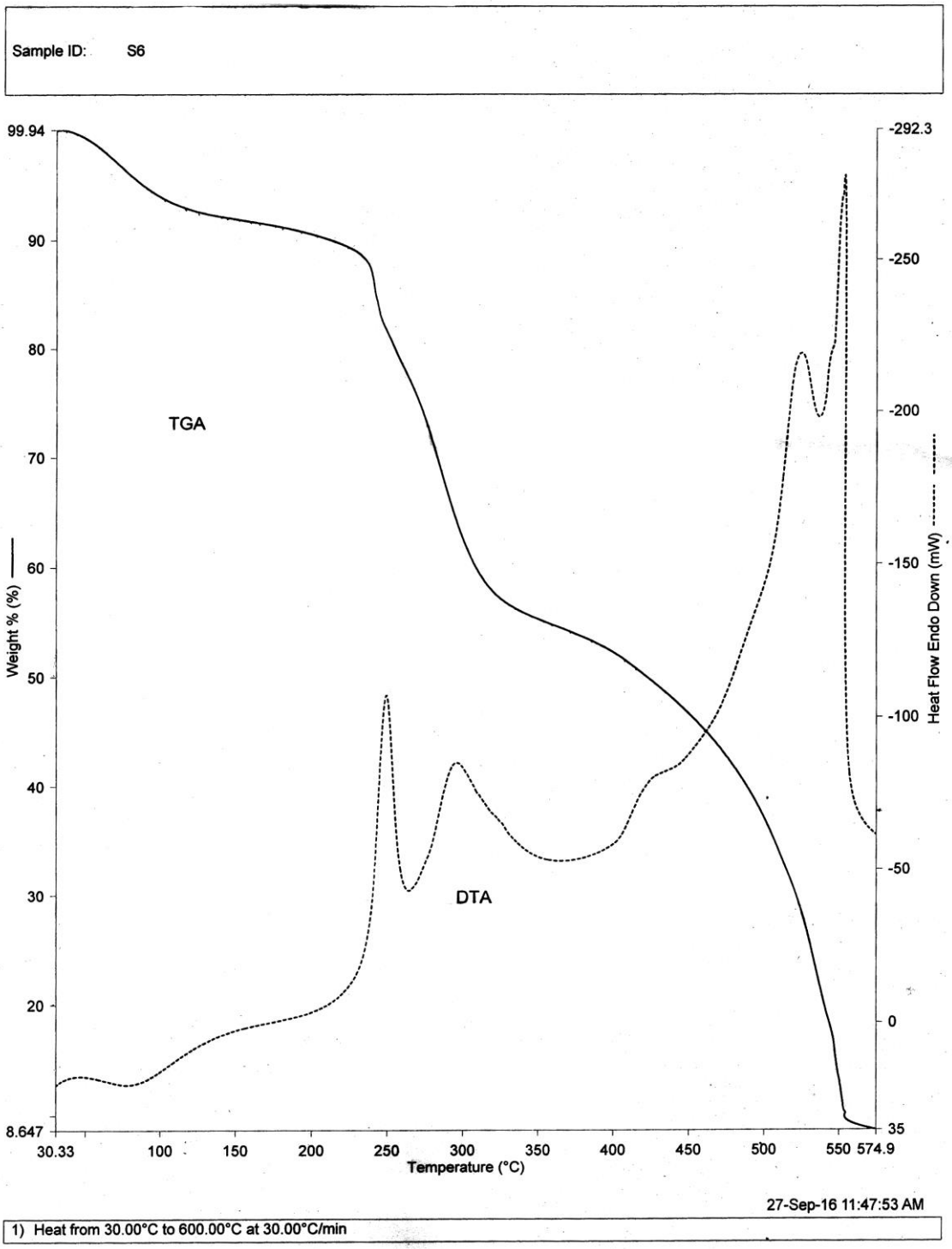



Fig-4.17: TGA & DTA spectrum $[Pb(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5.

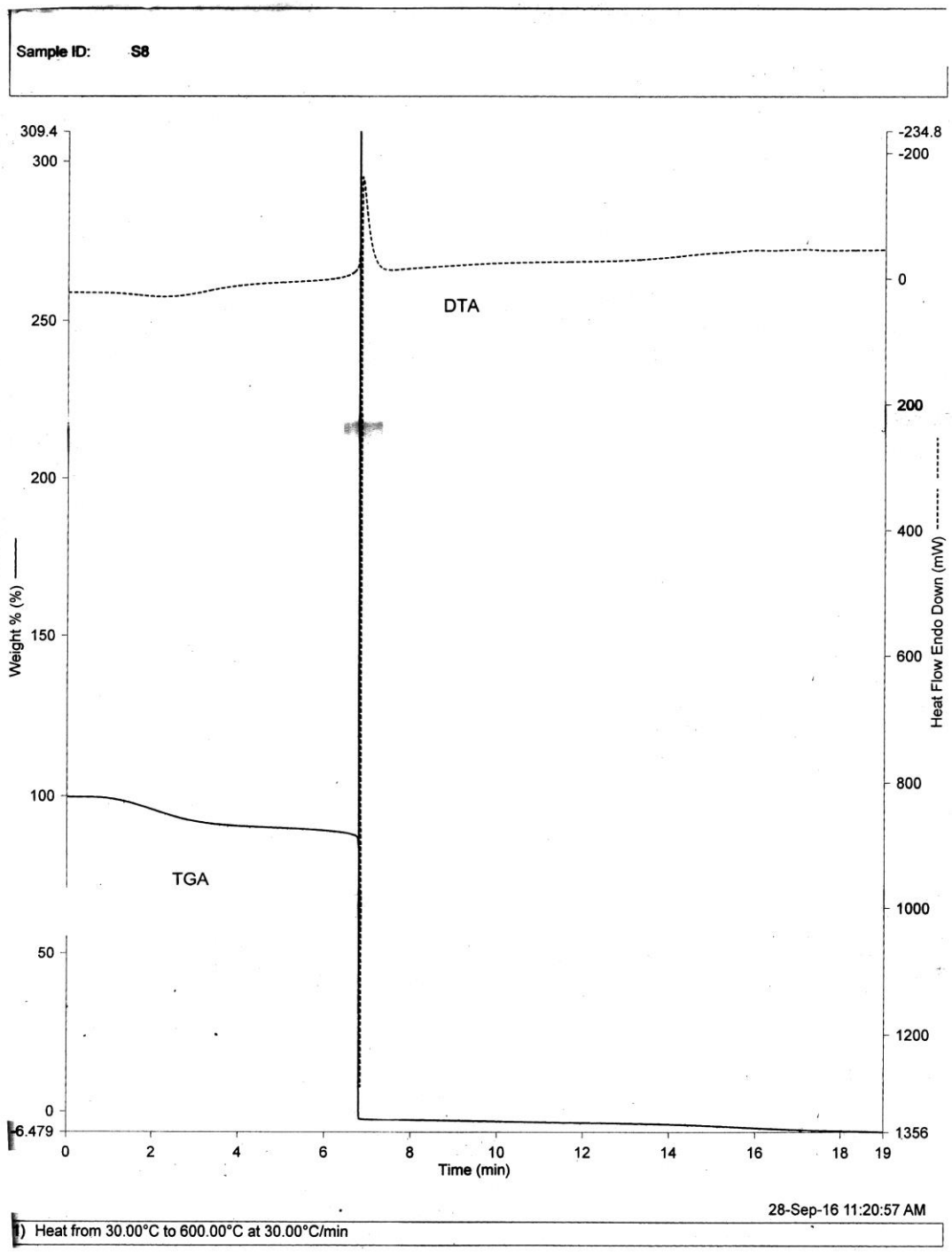


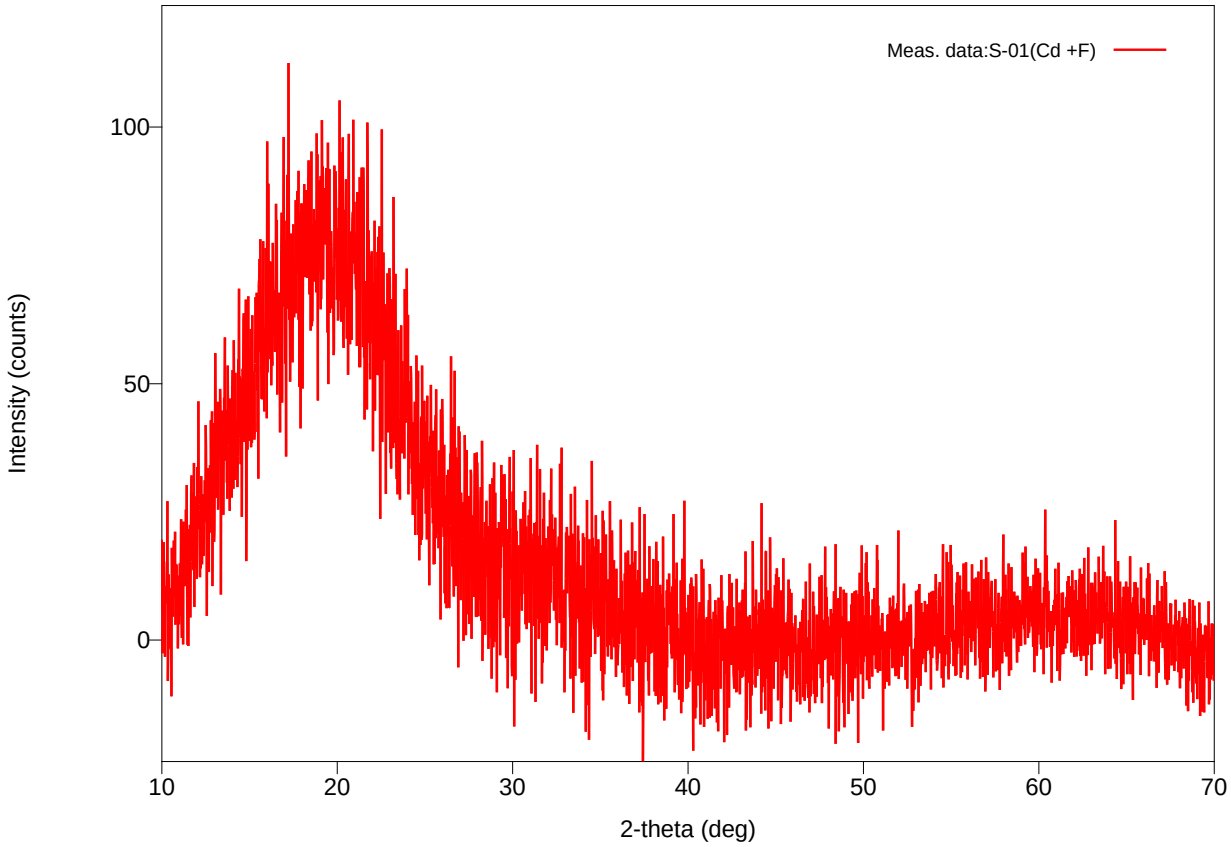

Fig-4.18: TGA & DTA spectrum $[Pb(C_{20}H_{24}N_8O_4)(ClO4)_2]$ complex 6.

# Peak List

## General information

| | | | |
|---|---|---|---|
| Analysis date | 2017/03/05 12:32:52 | | |
| Sample name | S-01(Pb +F) | Measurement date | 2017/03/05 11:57:13 |
| File name | S-01(Pb +F).raw | Operator | User1 |
| Comment | | | |

## Measurement profile



# RESULT AND DISCUSSION

## 4.2 MACROCYCLIC COMPLEXES OF Zn(II)

Reactions of malonodihydrazide with Zn(II) perchlorate hexahydrate in presence of formaldehyde, acetaldehyde, butanal dehyde,crotonaldehyde, cinnamaldehyde, and benzaldehyde give some 16 member macrocyclic complex as described in sec.3.3

Complexes (1-6) are characterized on the basis of elemental analysis, magnetic moment & conductance measurements, UV-visible spectra & infrared studies, thermal studies and other physical properties, like melting point, solubility, colour etc.

Molar conductance data of the complexes (1-6) are shown in Table 4.2.1. The conductance values of the complexes suggested that they are non-electrolytic in nature[24].

The infrared spectra of the complexes (1-6) are shown as spectral data (Table 4.2.4) of the complexes showed a strong and broad band at (3246-3265) $cm^{-1}$ which is assigned for the ν(NH) stretching[25].

Due to coordination the ν(N-H) stretching of the amide group goes to the higher field at (3246, 3265) $cm^{-1}$ region as compared to the starting material malonodihydrazide[26]. In the complexes the terminal-$NH_2$ group of malonodihydrazide condensed with the aldehyde moiety form a new secondary[1]-NH group which may appear at the same region (or overlape) as

to the amide-NH group as a result the ν(N-H) band appear as a strong and broad band. [The starting material malonodihydrazide have three ν(N-H) bands at (3248, 3213, 3050) $cm^{-1}$. The bands at (3248, 3050) $cm^{-1}$ for the asymmetrie and symmetric ν(N-H) stretching of the terminal-$NH_2$ moiety and 3213 $cm^{-1}$ for amidic (N-H) group]. The complexes showed a broad band at (2920-2972) $cm^{-1}$ is suggested for the ν(C-H) stretching of aliphatic moiety[32]. The complexes showed a strong band at (1649-1674) $cm^{-1}$ which represent the ν(C=O) of NH-NH-CO-$CH_2$ moity[27]. Three or four band at (625-1145) $cm^{-1}$ region also indicated the $\nu_1,\nu_2,\nu_3,\nu_4$ bands of ($ClO^-_4$) moiety. These stretching frequency is suggested the coordination of perchlorate to the metal through the O atom[28]. A medium band at (407-412) $cm^{-1}$ region is tentatively attributed to the ν(M-N) mode[29,30.] indicating the coordination of the lignad to the metal through the nitrogen atom.

The magnetic moment measurement data (Table 4.2.3) of the Zn(II) complexes (1-6) showed (1.56-1.78) B.M. These values correspond to no unpaired electrons of Pb(II) $d^{10}$system suggest the octahedral environment of the complexes which are consistent with the literature value[1]. The elemental analyses (C, H and N) (Table 4.2.2) and metal estimation data (Table 4.2.3) of the complexes are consistent with the proposed formula.

The UV-visible spectra of the complexes (1-6) are shown (Table 4.2.5) band at 320,480 nm, (1-6) at represent the d-d transition of $^4A_{2g}$ (F) $\rightarrow$ $^4T_{1g}$(F), $^4A_{2g}$(F) $\rightarrow$ $^4T_{1g}$(P), which suggested the octahedral geometry of the Pb (II) complexes[31,32].

Thermal studies**:** The thermal properties of metal (II) complexes were investigated by thermograms (TGA, DTA) and are shown in (Fig 4.2.13-4.2.18) and the corresponding thermal analysis is presented in (Tabl.4.6). In the case of complex (1-6) the decomposition occurs in the (230-325)$^0$C range. There is no mass loss up to 230$^0$C. The first stage of decomposition starts at 230$^0$C and end at 230$^0$C with a corresponding weight loss 25%.Which is accompanied by endothermic effect in the DTA curve in the range 225$^0$C which is accompanied by weight loss confirming the second stage of decomposition is observed at 225-350$^0$C (60% wt. loss).meanwhile the DTA curve exhibits endothermic effect in the range 325$^0$C which is accompanied by weight loss confirming.

On the basis of elemental analysis magnetic moment and conductance measurements, thermal studies UV Visible spectra, infrared spectra and other physical properties the suggested structure of the complexes are octahedral in nature as in Fig.4.2.19

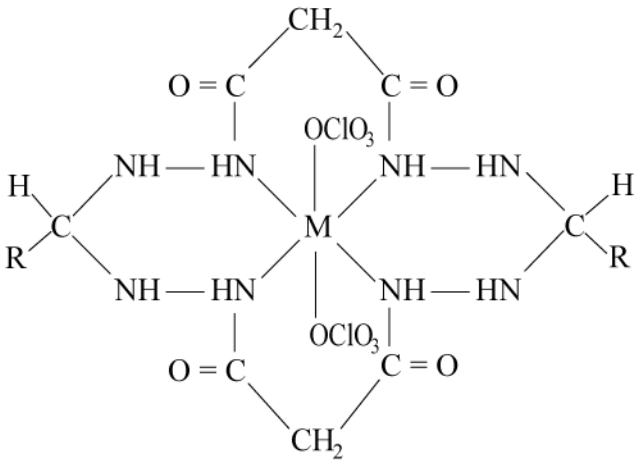



Fig. 4.2.19

M =Zn(II), where R=H(1), $CH_3$(2),$CH_3CH=CH$,-(3) $C_6H_5CH=CH_2$-(4),$CH_3$-$CH_2$-$CH_2$(5), $C_6H_6CH$(6)

Table- 4.2.1: Analytical data and other physical properties of compounds (1-6)

| No. | Compounds | %Yield | Colour | Milting point $^0$C | % M | | Molar conductance ohm$^{-1}$cm$^2$mol$^{-1}$ |
|---|---|---|---|---|---|---|---|
| | | | | | Calculated | Found | |
| 1 | $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 80 | Light yellow | 210 | 11.77 | 11.70 | 11.80 |
| 2 | $[Zn\ (C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 60 | Red | 200 | 11.20 | 111.22 | 12.08 |
| 3 | $[Zn\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 75 | Yellow | 190 | 10.22 | 10.20 | 10.03 |
| 4 | $[Zn\ (\ C_{24}H_{24}N_8O_4)(ClO_4)_2]$ | 70 | white | 215 | 10.28 | 10.25 | 12.03 |
| 5 | $[Zn\ (C_{20}H_{28}N_8O_4)(ClO_4)_2]$ | 85 | yellow | 205 | 9.50 | 9.45 | 13.90 |
| 6 | $[Zn\ (C_{14}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 75 | Light yellow | 203 | 9.44 | 9.50 | 12.31 |

Table- 4.2.2: Elemental analysis data of compounds (1-6)

| No. | Compounds | %C | | %H | | %N | |
|---|---|---|---|---|---|---|---|
| | | Calculated | Found | Calculated | Found | Calculated | Found |
| 1 | $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 18.39 | 18.35 | 2.06 | 2.05 | 21.45 | 21.00 |
| 2 | $[Zn\ (C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 20.68 | 20.60 | 1.72 | 1.72 | 19.31 | 19.03 |
| 3 | $[Zn\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 26.41 | 26.40 | 2.20 | 221 | 17.61 | 17.20 |
| 4 | $[Zn\ (\ C_{24}H_{24}N_8O_4)(ClO_4)_2]$ | 26.58 | 26.50 | 2.22 | 2,20 | 17.72 | 17.70 |
| 5 | $[Zn\ (C_{20}H_{28}N_8O_4)(ClO_4)_2]$ | 42.10 | 42.00 | 3.51 | 3.50 | 16.37 | 16.30 |
| 6 | $[Zn\ (C_{14}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 34.88 | 34.80 | 3.98 | 4.00 | 16.27 | 16.20 |

Table- 4.2.3: Magnetic moment data of compounds (1-6)

| No. | Compounds | Sample length, *l* in cm | Weight of the sample, m in gm | Susceptibility of the empty tube, Ro | Susceptibility of the sample with tube ,R | Mass Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | Molecular weight,M | Molar Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | μeff B.M |
|---|---|---|---|---|---|---|---|---|---|
| **1** | $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0695 | -48 | -22 | 1.71 | 552 | 0.943 | 1.50 |
| **2** | $[Zn\ (C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2.1 | 0.0692 | -47 | -23 | 1.51 | 580 | 0.875 | 1.44 |
| **3** | $[Zn\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0596 | -40 | -21 | 1.46 | 636 | 0.928 | 1.49 |
| **4** | $[Zn\ (\ C_{24}H_{24}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0559 | -46 | -24 | 1.47 | 632 | 0.929 | 1.49 |
| **5** | $[Zn\ (C_{20}H_{28}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0630 | -42 | -20 | 1.31 | 684 | 0.896 | 1.46 |
| **6** | $[Zn\ (C_{14}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 1.7 | 0.0589 | -46 | -23 | 1.38 | 688 | 0.949 | 1.50 |

Table- 4.2.4: Important infrared spectral bands of compounds (1-6)

| No. | Compounds | $\nu$(C-H) $cm^{-1}$ | $\nu$(C=O) $cm^{-1}$ | $\nu$(N-H) $cm^{-1}$ | $\nu$(M-N) $cm^{-1}$ | $\nu(ClO_4)$ $cm^{-1}$ |
|---|---|---|---|---|---|---|
| 1 | $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 3047 | 1664 | 3251 | 420 | 1180,1089,623 |
| 2 | $[Zn\ (C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2920 | 1649 | 3282 | 430 | 1105,979,623 |
| 3 | $[Zn\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 2960 | 1640 | 3250 | 416 | 1150,1060,620 |
| 4 | $[Zn\ (\ C_{24}H_{24}N_8O_4)(ClO_4)_2]$ | 2967 | 1650 | 3261 | 408 | 1160,1040,623 |
| 5 | $[Zn\ (C_{20}H_{28}N_8O_4)(ClO_4)_2]$ | 3040 | 1660 | 3254 | 409 | 1140,1080,621 |
| 6 | $[Zn\ (C_{14}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 3070 | 1680 | 3270 | 406 | 1170,1090,625 |

Table-4.2.5:U.V-Visible Adsorption Maxima of Compounds (1-6)

| No. | Compounds | λ max (n,m) |
|---|---|---|
| 1 | $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 290,540 |
| 2 | $[Zn\ (C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 290.490 |
| 3 | $[Zn\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 293.490 |
| 4 | $[Zn\ (\ C_{24}H_{24}N_8O_4)(ClO_4)_2]$ | 290,500 |
| 5 | $[Zn\ (C_{20}H_{28}N_8O_4)(ClO_4)_2]$ | 290,500 |
| 6 | $[Zn\ (C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 300,540 |

Table- 4.2.6: Thermal Analysis Data Of Compounds (1-6)

| No. | Compounds | %M Ligand | | | %M Metal oxide(MO) | | |
|---|---|---|---|---|---|---|---|
| | | $Tem^0C$ | Calculated | Found | $Tem^0C$ | Calculated | Found |
| 1 | $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 135.02 | 83.22 | 83.20 | 599.00 | 37.66 | 37.60 |
| 2 | $[Zn\ (C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 135.00 | 86.21 | 86.20 | 599.00 | 36.14 | 36.10 |
| 3 | $[Zn\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 130.90 | 87.14 | 87.10 | 550.60 | 33.72 | 33.60 |
| 4 | $[Zn\ (\ C_{24}H_{24}N_8O_4)(ClO_4)_2]$ | 130.45 | 88.91 | 88.50 | 450.00 | 29.06 | 29.00 |
| 5 | $[Zn\ (C_{20}H_{28}N_8O_4)(ClO_4)_2]$ | 140.50 | 88.23 | 88.20 | 530.00 | 30.86 | 30.40 |
| 6 | $[Zn\ (C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 137.45 | 87.07 | 87.00 | 540.00 | 33.89 | 33.30 |

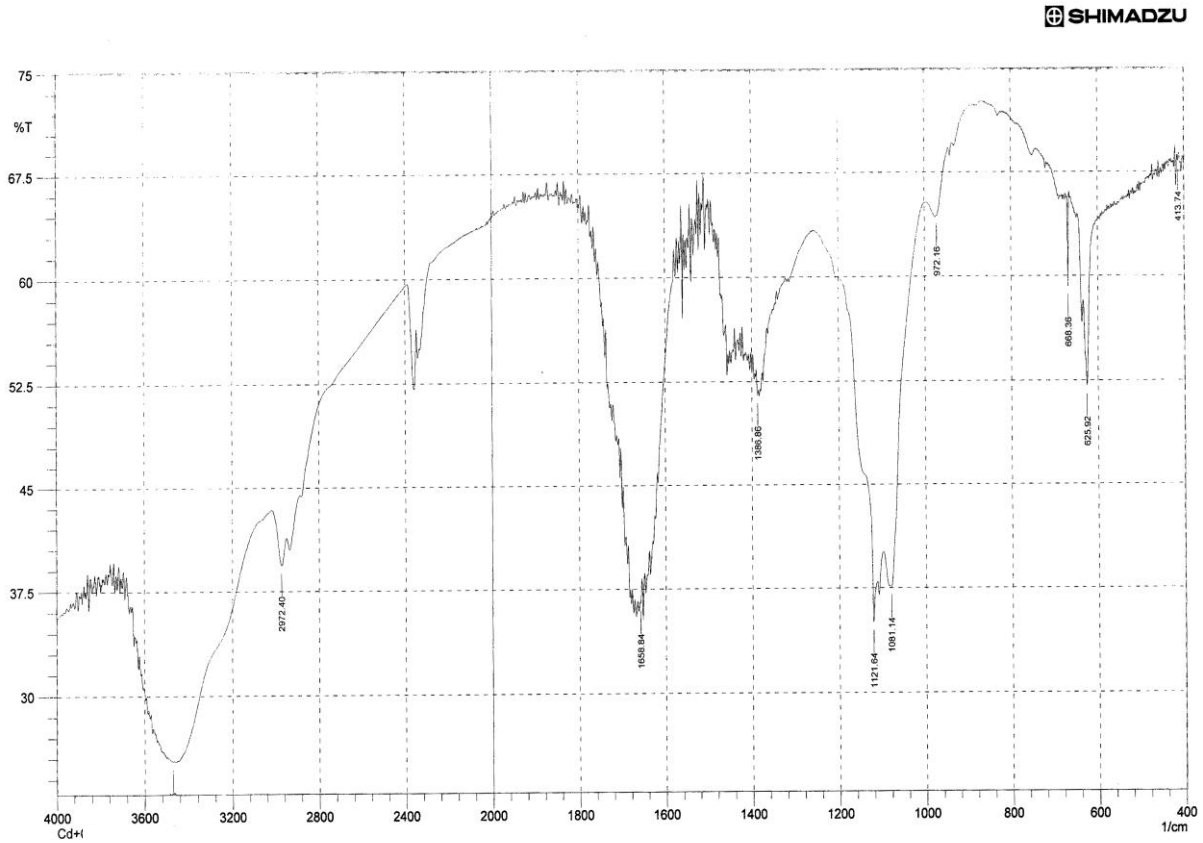



Fig-4.2.1: Infrared spectrum $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$complex 1

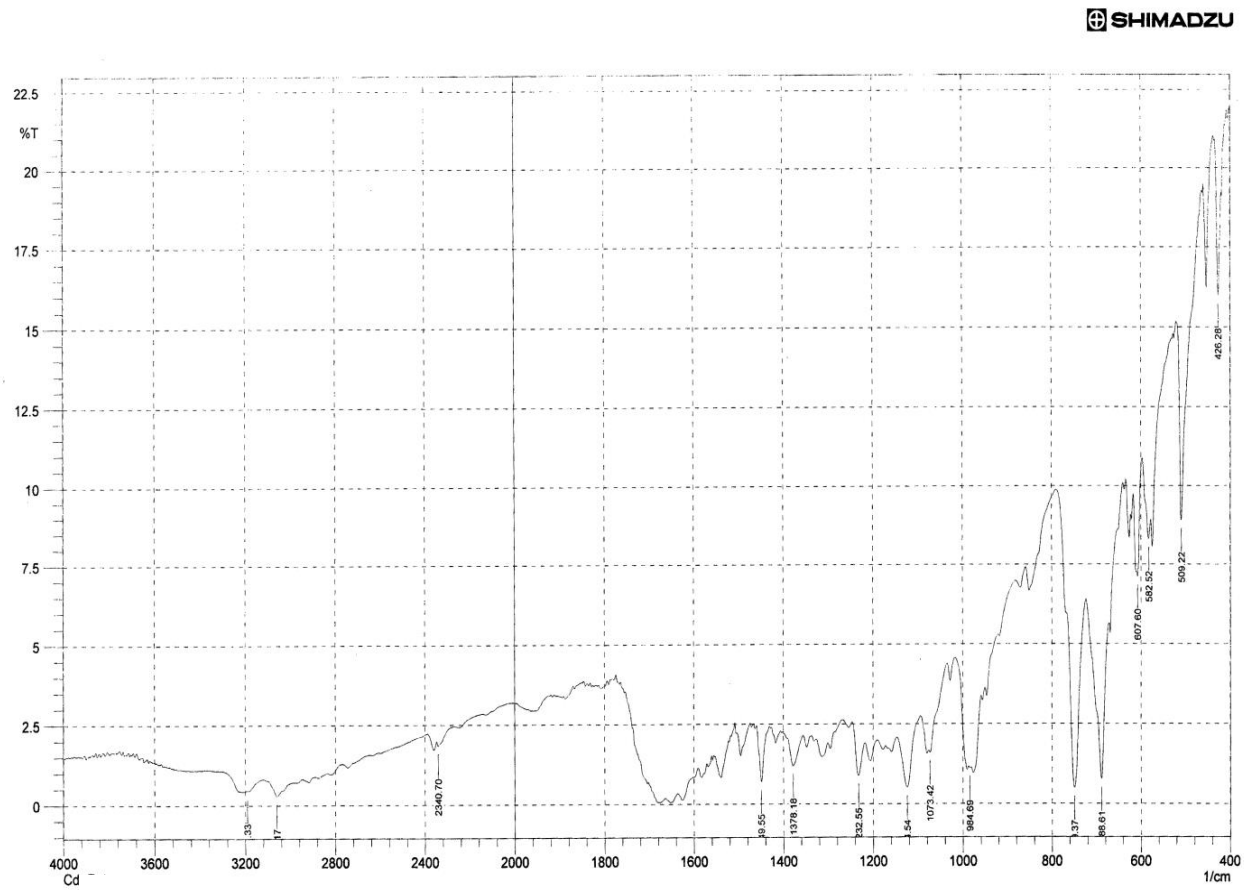



Fig-4.2.2: Infrared spectrum $[Zn(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

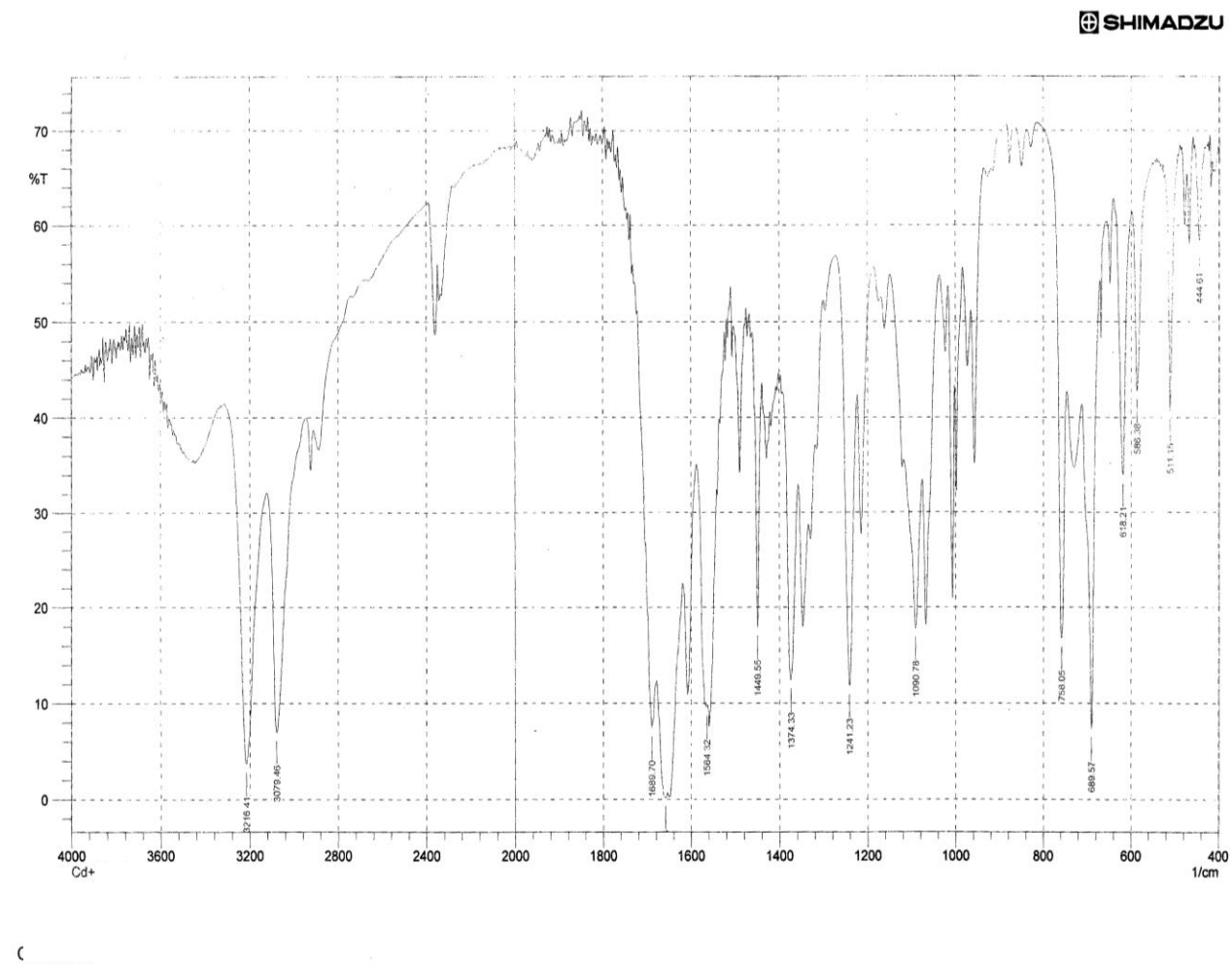



Fig-4.2.3: Infrared spectrum $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

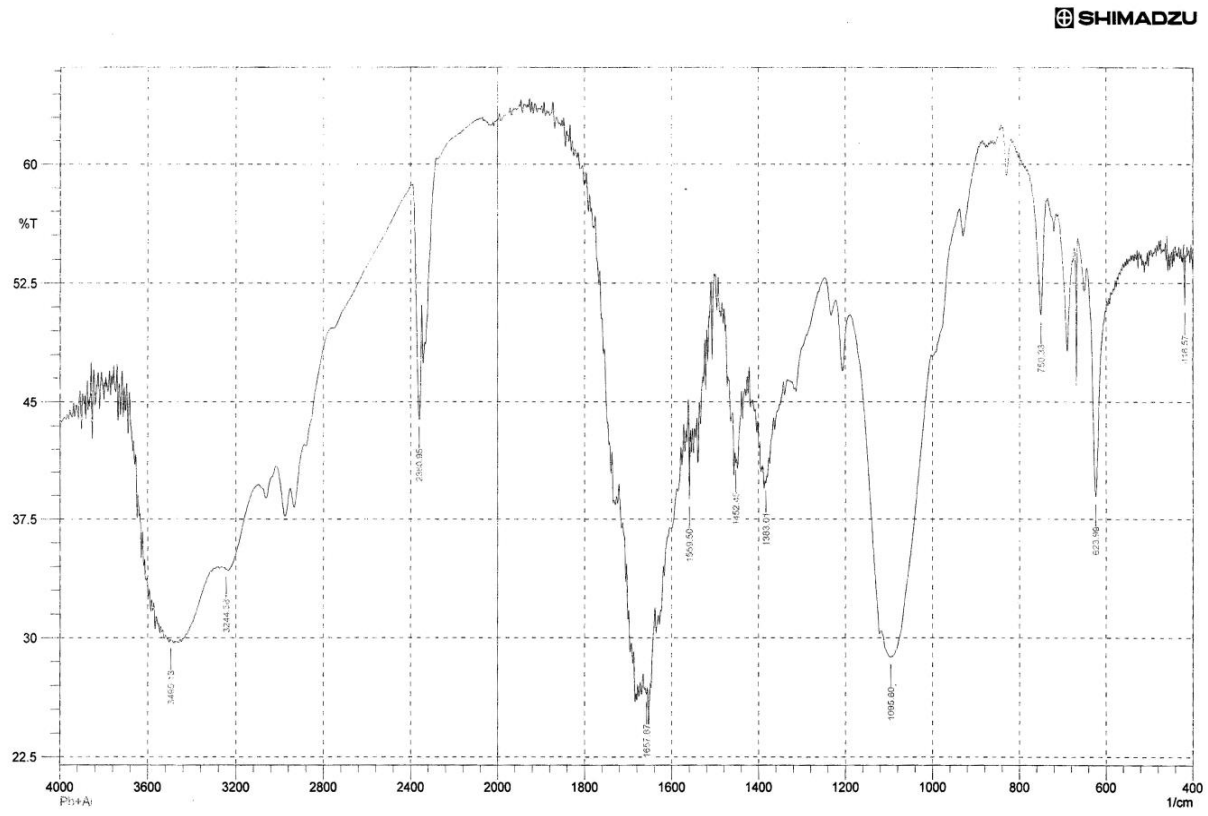



Fig-4.2.4 : Infrared spectrum $[Zn(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4.

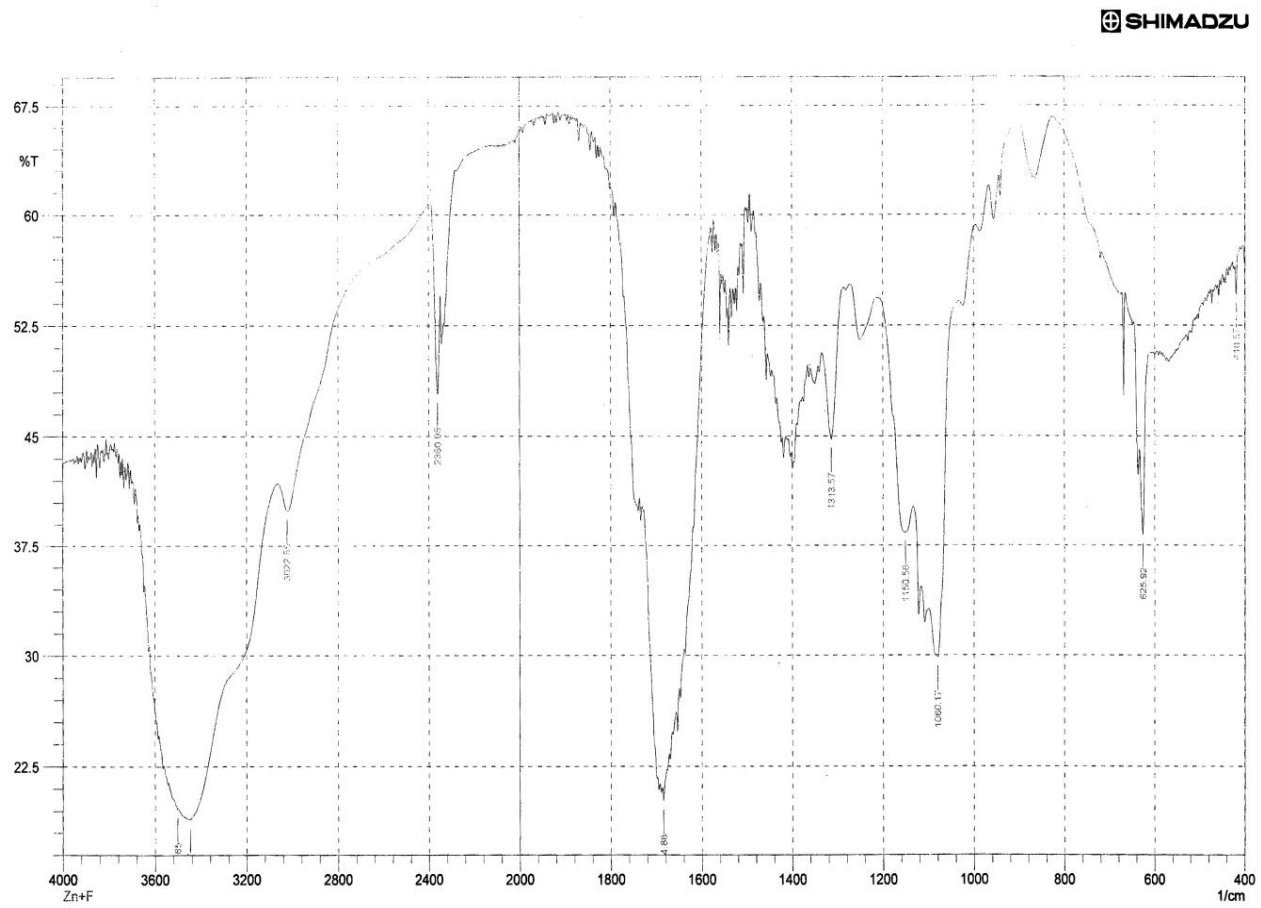



Fig-4.2.5: Infrared spectrum $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5.

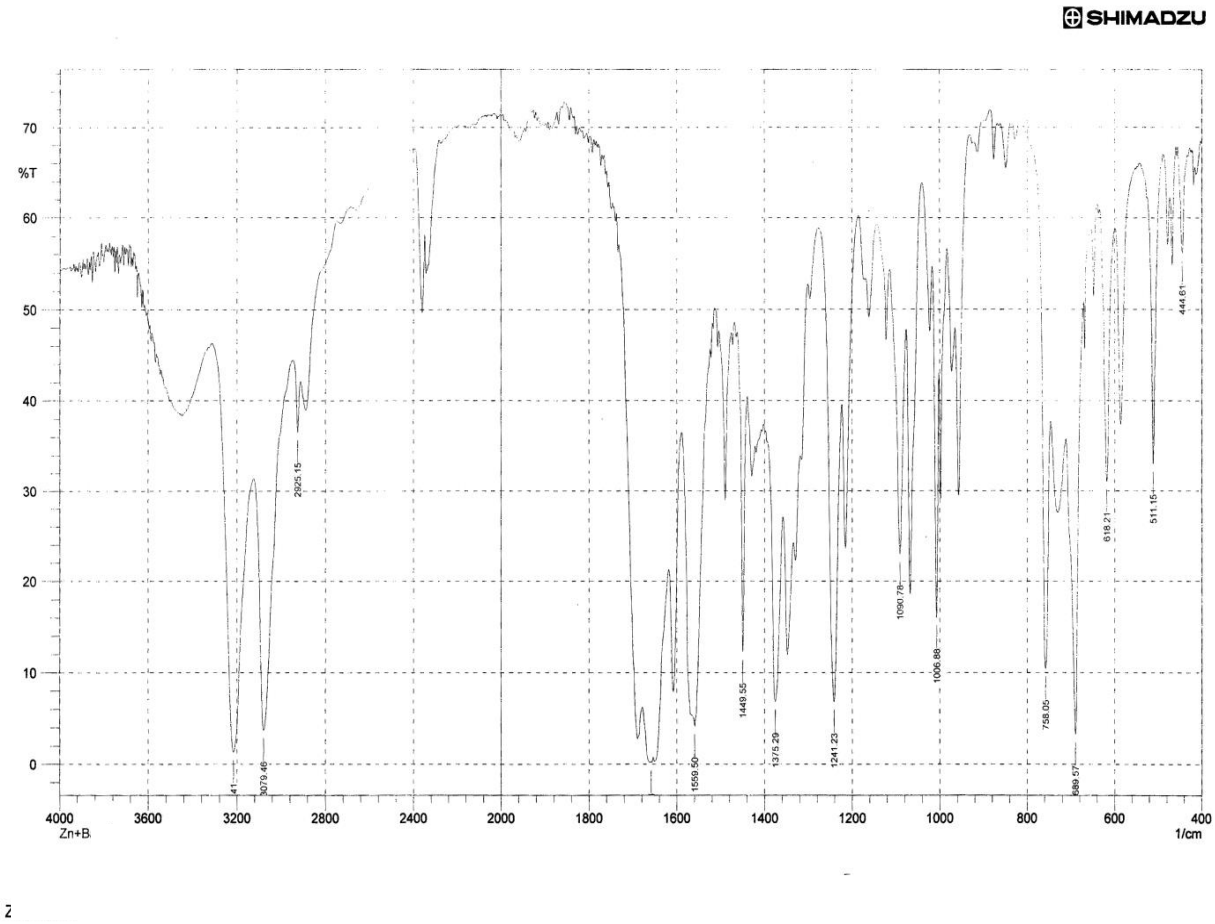



Fig-4.2.6: Infrared spectrum $[Zn(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex 6.

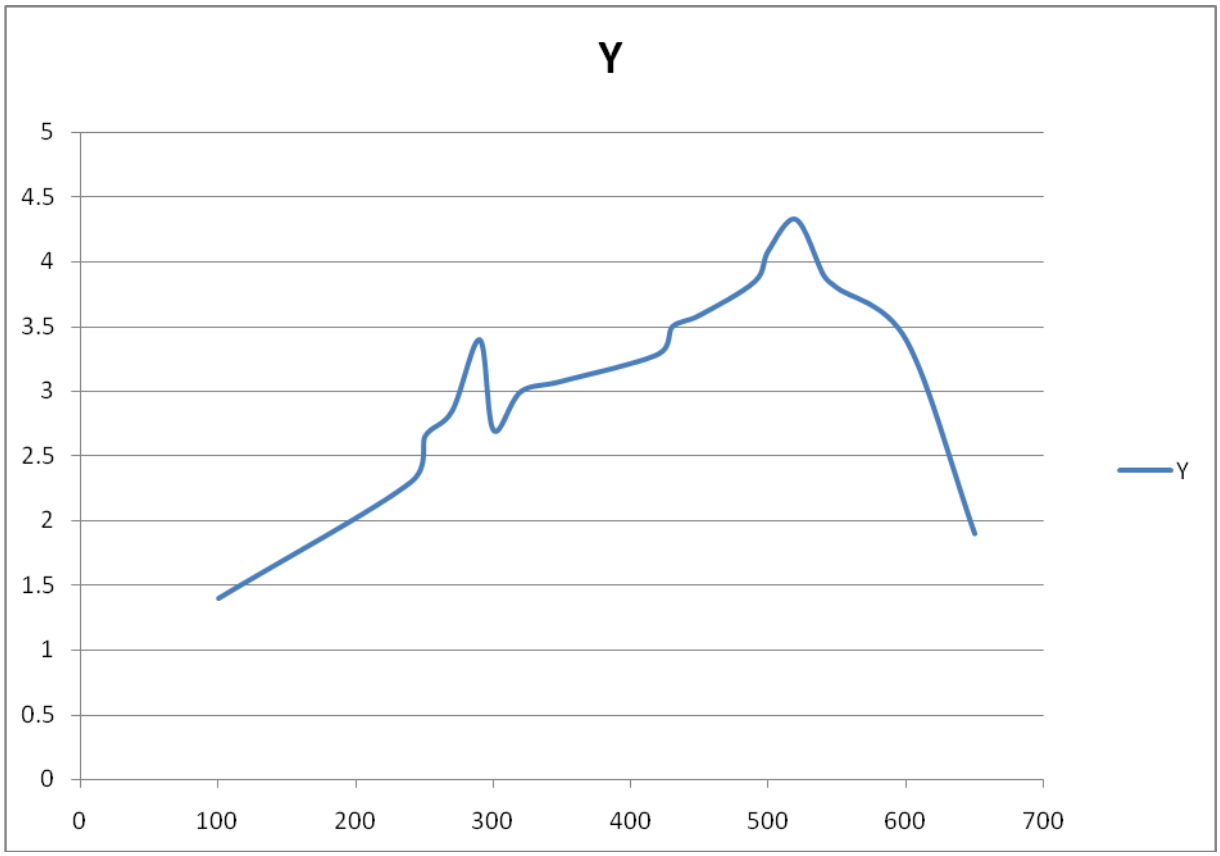



Fig-4.2.7: Infrared spectrum of $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

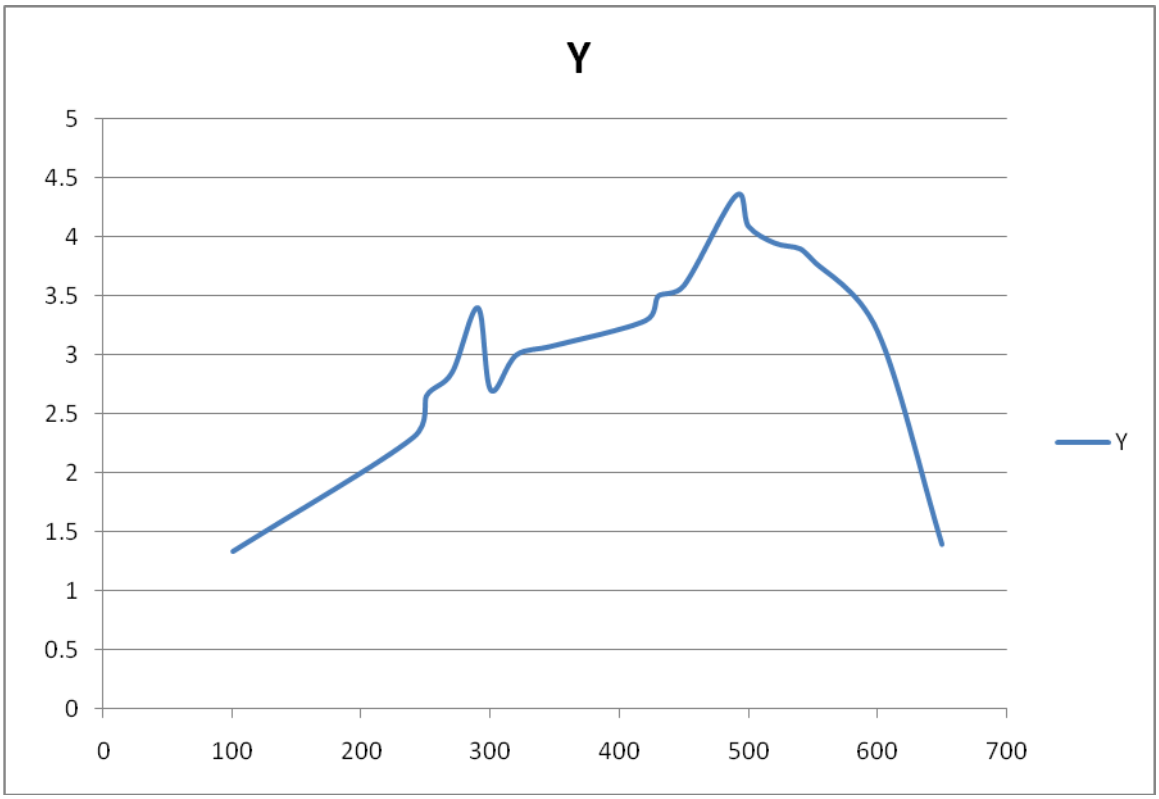



Fig-4.2.8: UV- Visible spectrum of $[Zn(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

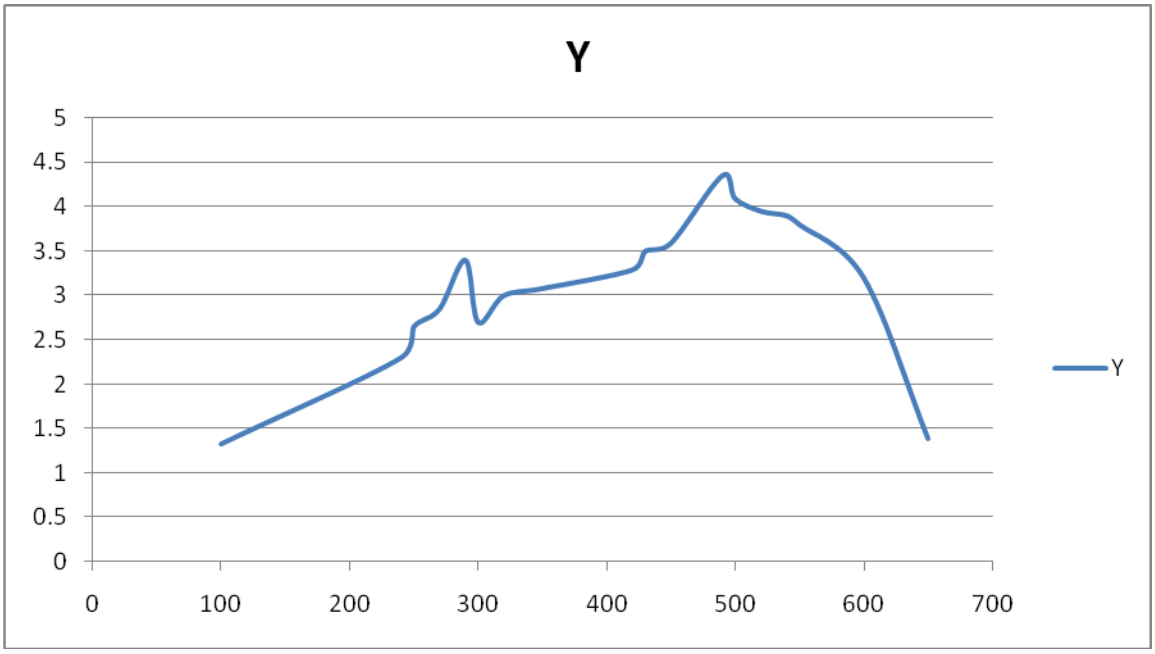



Fig-4.2.9: UV- Visible spectrum of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

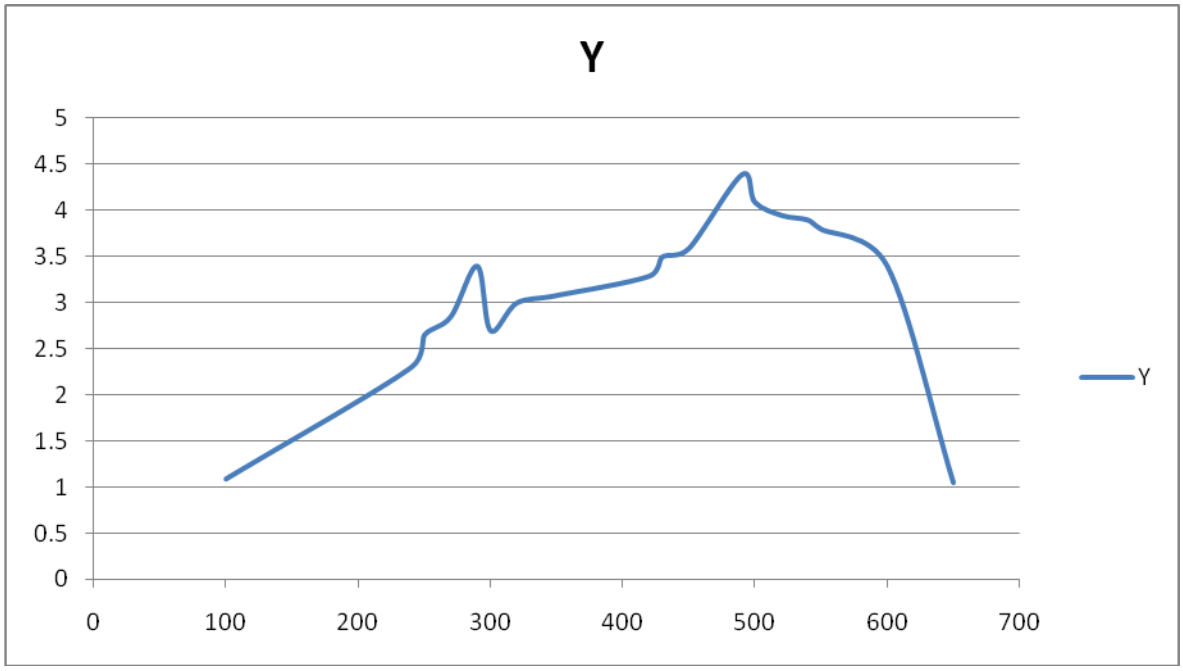



Fig-4.2.10: UV- Visible spectrum of $[Zn(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4.

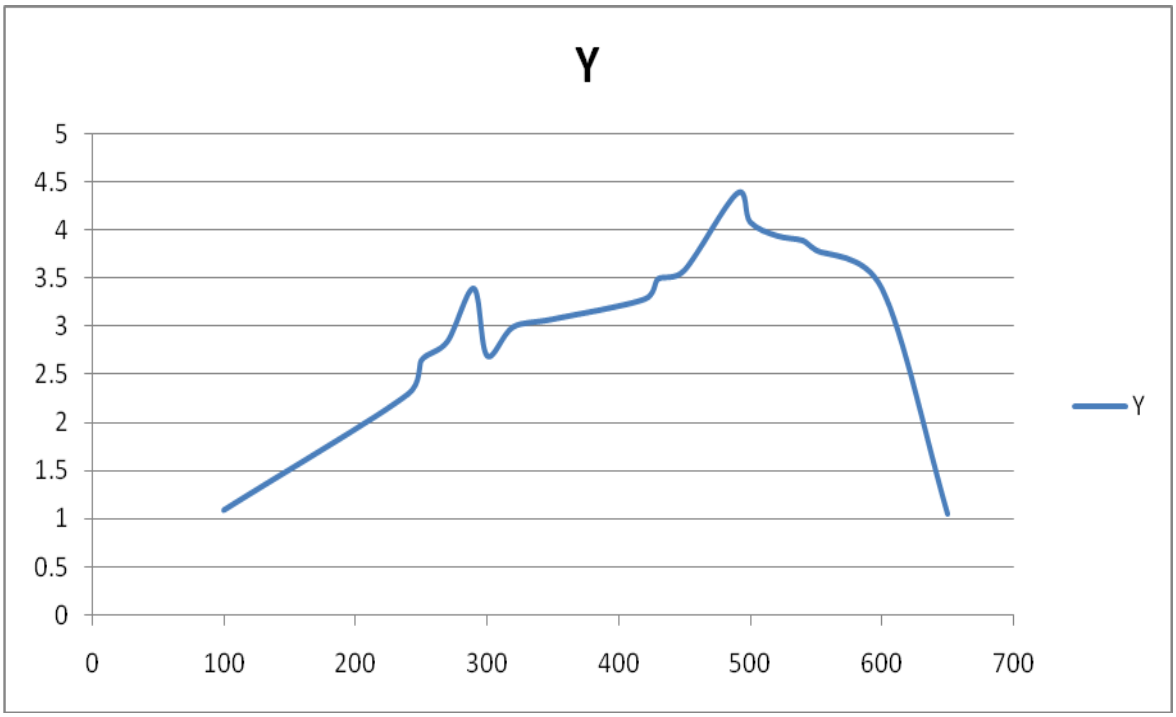



Fig-4.2.11: UV- Visible spectrum of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5

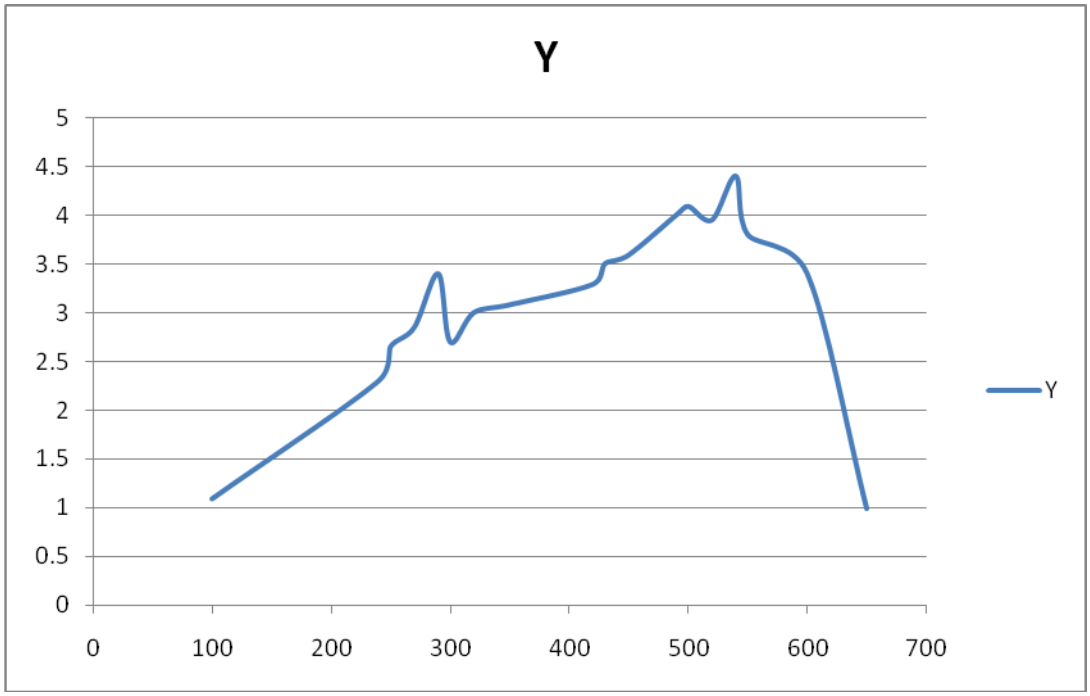



Fig-4.2.12: UV- Visible spectrum of $[Zn(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex 6.

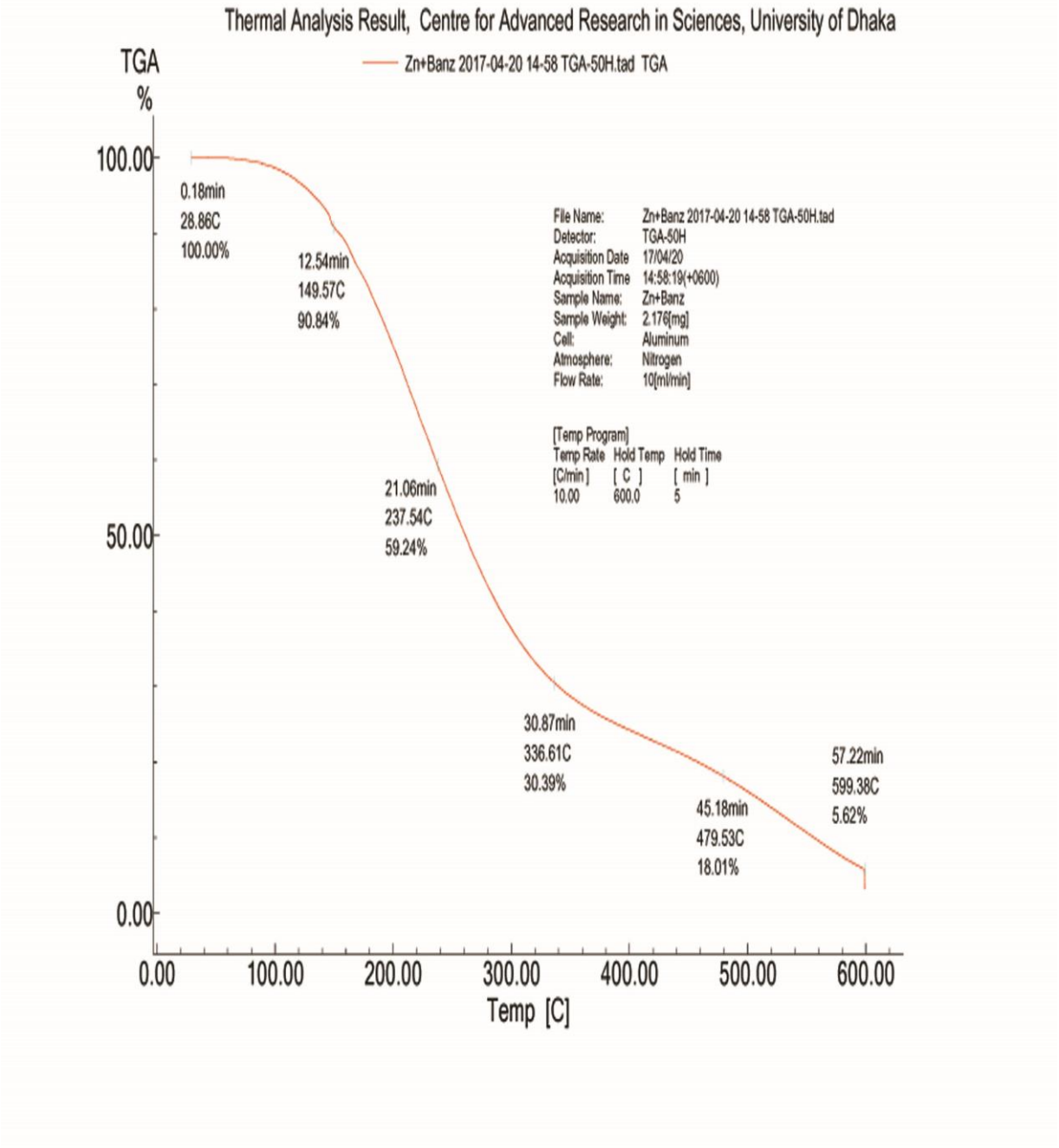



Fig-4.2.13: TGA spectrum $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

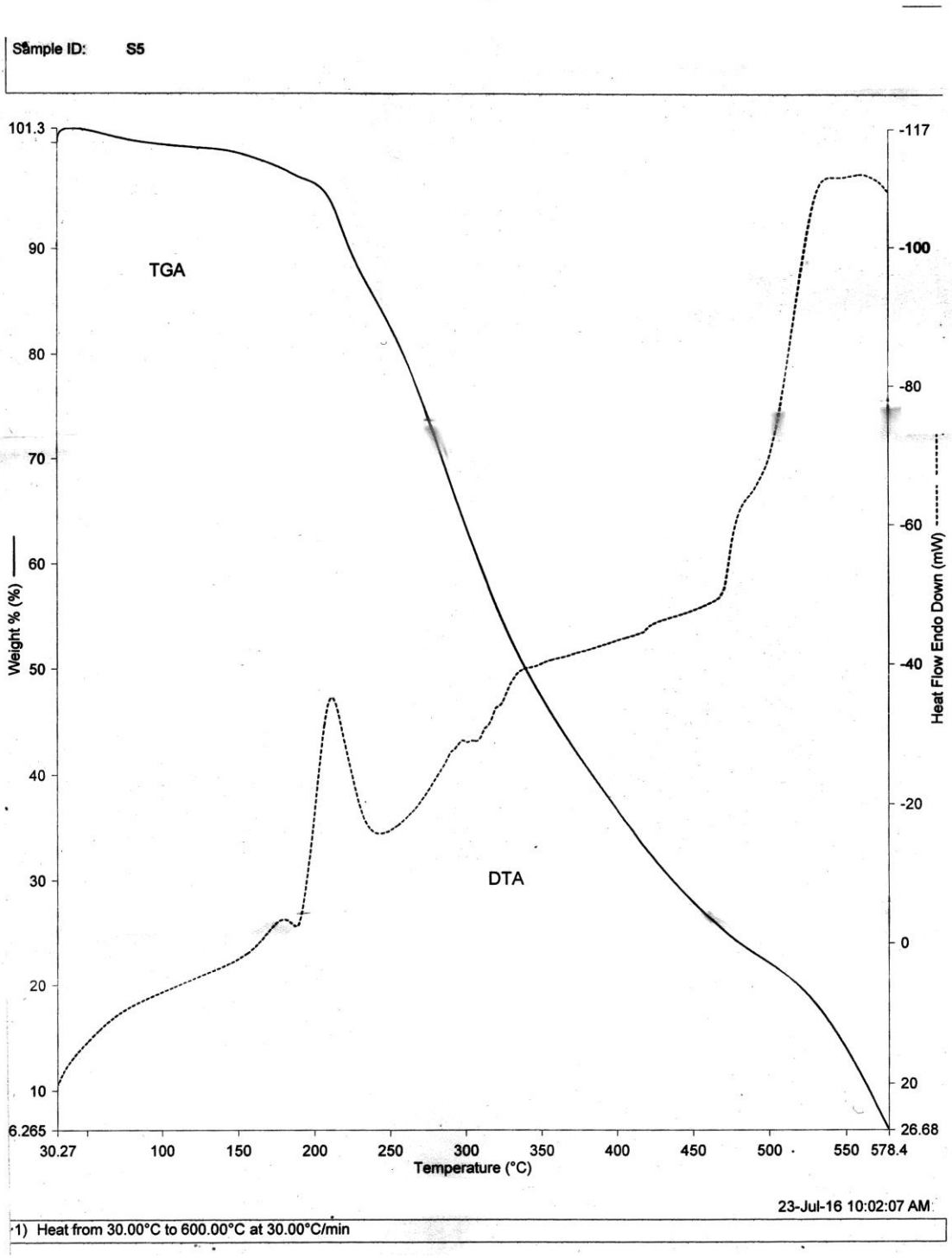



Fig-4.2.14: TGA & DTA spectrum $[Zn(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

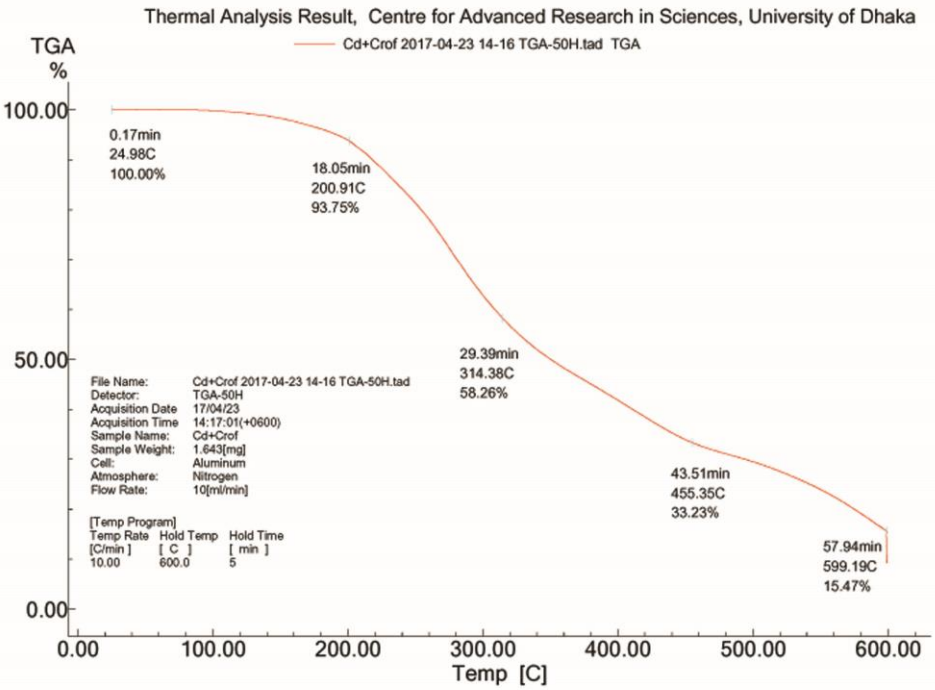



Fig-4.2.15: TGA spectrum $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

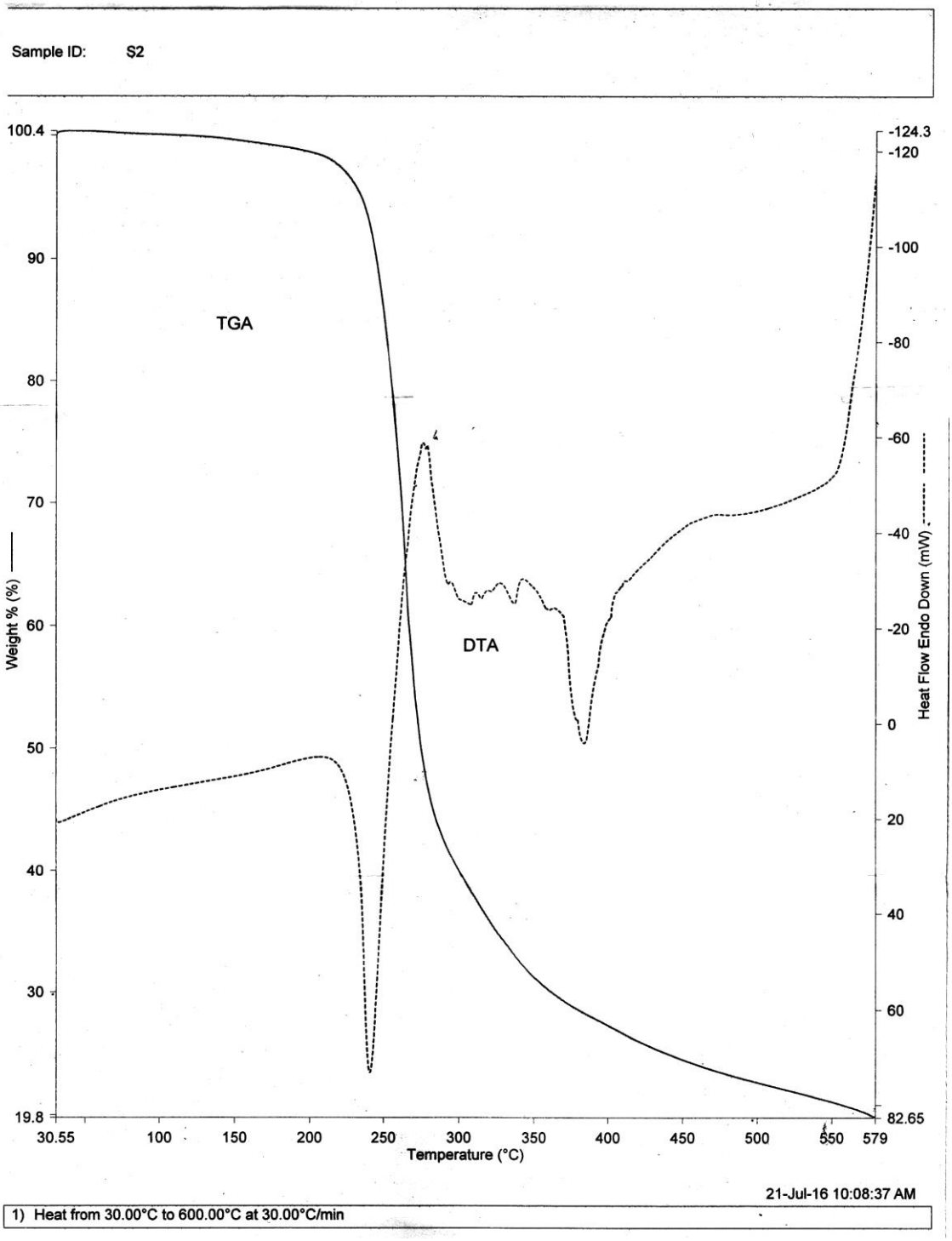



Fig-4.2.16: TGA & DTA spectrum $[Zn(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4

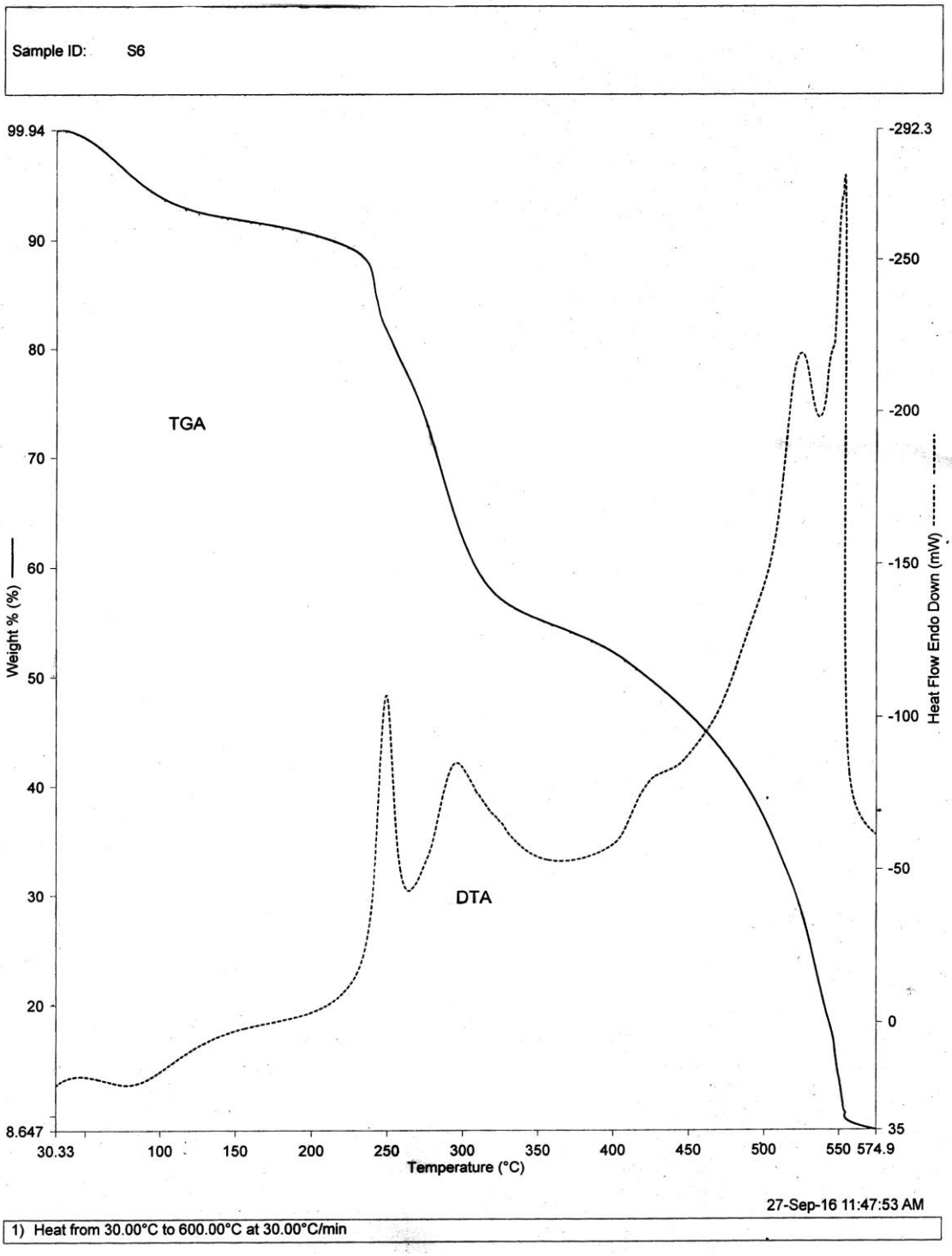



Fig-4.2.17: TGA & DTA spectrum $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5.

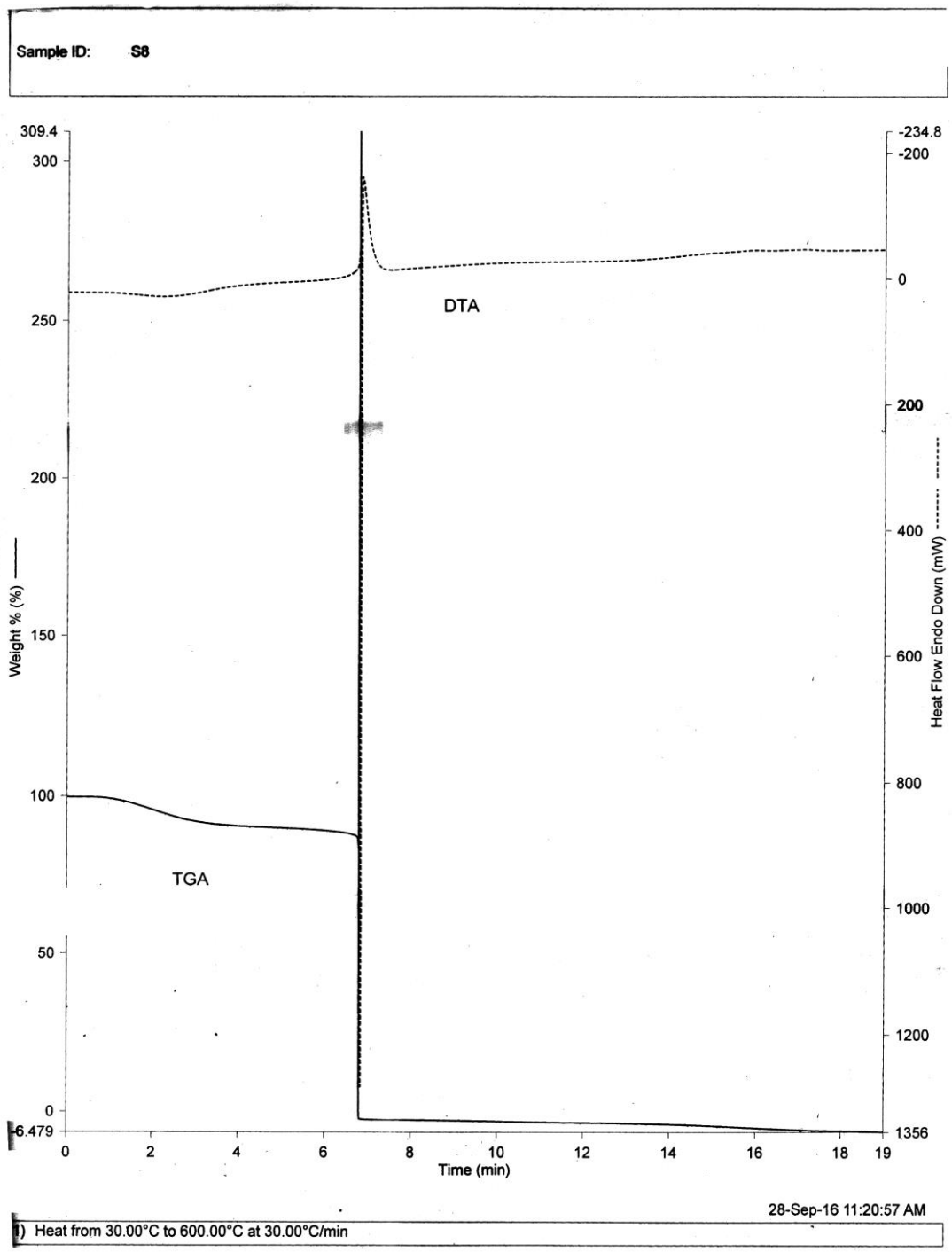


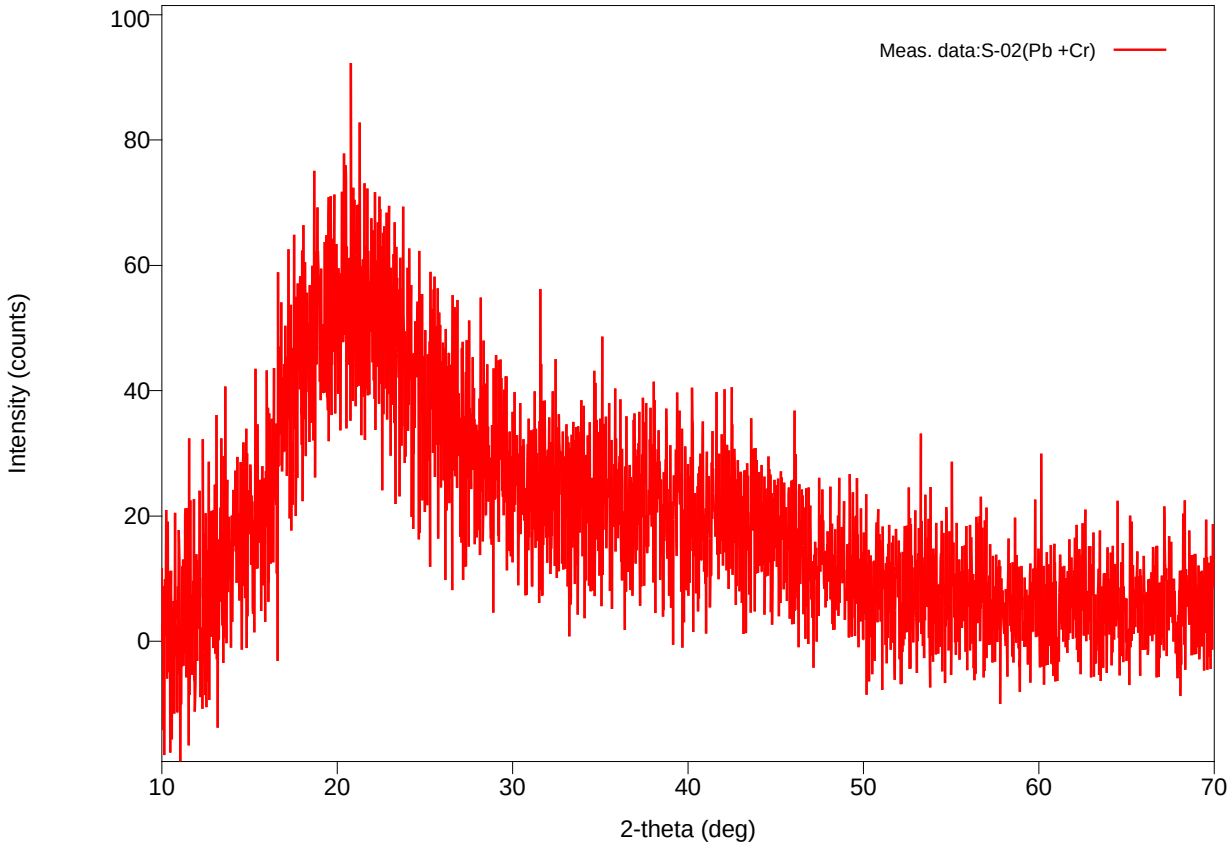

Fig-4.2.18: TGA & DTA spectrum $[Zn(C_{20}H_{24}N_8O_4)(ClO4)_2]$ complex 6.

# Peak List

## General information

| | | | |
|---|---|---|---|
| Analysis date | 2017/03/05 12:58:25 | | |
| Sample name | S-02(Zn +Cr) | Measurement date | 2017/03/05 12:20:22 |
| File name | S-02(Zn +Cr).raw | Operator | User1 |
| Comment | | | |

## Measurement profile



# RESULT AND DISCUSSION

## 4.3 MACROCYCLIC COMPLEXES OF La (II)

Reactions of malonodihydrazide with La(II) perchlorate hexahydrate in presence of formaldehyde, acetaldehyde, butanal dehyde ,crotonaldehyde, cinnamaldehyde and , benzaldehyde give some 16 member macrocyclic complex as described in sec. 3.4

Complexes (1-6) are characterized on the basis of elemental analysis, magnetic moment & conductance measurements, UV-visible spectra & infrared studies ,thermal studies and other physical properties, like melting point, solubility, colour etc.

Molar conductance data of the complexes (1-6) are shown in (Table 4.3.1). The conductance values of the complexes suggested that they are non-electrolytic in nature[24].

The infrared spectra of the complexes (1-6) are shown as spectral data (Table 4.3.4) of the complexes showed a strong and broad band at (3246-3265) $cm^{-1}$ which is assigned for the ν(NH) stretching[25].

Due to coordination the ν(N-H) stretching of the amide group goes to the higher field at (3250, 3282) $cm^{-1}$ region as compared to the starting material malonodihydrazide[26]. In the complexes the terminal-$NH_2$ group of malonodihydrazide condensed with the aldehyde moiety form a new secondary[1]-NH group which may appear at the same region (or overlape) as to the amide-NH group as a result the ν(N-H) band appear as a strong and broad band. [The starting material malonodihydrazide have three ν(N-H) bands at (3250, 3251, 33270) $cm^{-1}$. The bands at (3250, 3382) $cm^{-1}$ for the

asymmetrie and symmetric ν(N-H) stretching of the terminal-$NH_2$ moiety and 3250 $cm^{-1}$ for amidic (N-H) group]. The complexes showed a broad band at (2920-2970) $cm^{-1}$ is suggested for the ν(C-H) stretching of aliphatic moiety[32]. The complexes showed a strong band at (1640-1680) $cm^{-1}$ which represent the ν(C=O) of NH-NH-CO-$CH_2$ moity[27]. Three or four band at (621-1180) $cm^{-1}$ region also indicated the $\nu_1,\nu_2,\nu_3,\nu_4$ bands of ($ClO^-_4$) moiety. These stretching frequency is suggested the coordination of perchlorate to the metal through the O atom[28]. A medium band at (406-420) $cm^{-1}$ region is tentatively attributed to the ν(M-N) mode[29,30.] indicating the coordination of the lignad to the metal through the nitrogen atom.

The magnetic moment measurement data (Table 4.3.3) of the La(II) complexes (1-6) showed (1.56-1.78) B.M. These values correspond to no unpaired electrons of La(II) $d^{10}$system suggest the octahedral environment of the complexes which are consistent with the literature value[1]. The elemental analyses (C, H and N) (Table 4.3.2) and metal estimation data (Table 4.3.3) of the complexes are consistent with the proposed formula.

The UV-visible spectra of the complexes (1-6) are shown (Table 4.3.5) band at 330, 350 nm, (1-6) at represent the d-d transition of $^4A_{2g}$ (F) $\rightarrow$ $^4T_{1g}$(F), $^4A_{2g}$(F) $\rightarrow$ $^4T_{1g}$(P), which suggested the octahedral geometry of the La(II) complexes[31,32].

Thermal studies: The thermal properties of metal (II) complexes were investigated by thermo grams (TGA, DTA) and are shown in (Fig 4.3.13-4.3.18) and the corresponding thermal analysis is presented in (Tabl.4.3.6). In the case of complex (1-6) the decomposition occurs in the (230-325)$^0$C range. There is no mass loss up to 230$^0$C. The first stage of decomposition starts at 230$^0$C and end at 230$^0$C with a corresponding weight loss 25%.

Which is accompanied by endothermic effect in the DTA curve in the range $225^0$C which is accompanied by weight loss confirming the second stage of decomposition is observed at 225-$350^0$C (60% wt. loss).meanwhile the DTA curve exhibits endothermic effect in the range $325^0$C which is accompanied by weight loss confirming.

X-Ray diffraction: The possible geometry of the product $[La(C_{24}H_{28}N_8O_4)(ClO4)_2]$ has been deduced on the basis of x-ray power diffraction studies. The result show that the compound belong to the orthorhombic crystal. The 2θ angles are reported in table 4.3.7.

On the basis of elemental analysis magnetic moment and conductance measurements, thermal studies UV Visible spectra, infrared spectra, x-ray diffraction studies and other physical properties the suggested structure of the complexes are octahedral in nature as in Fig.4.3.19

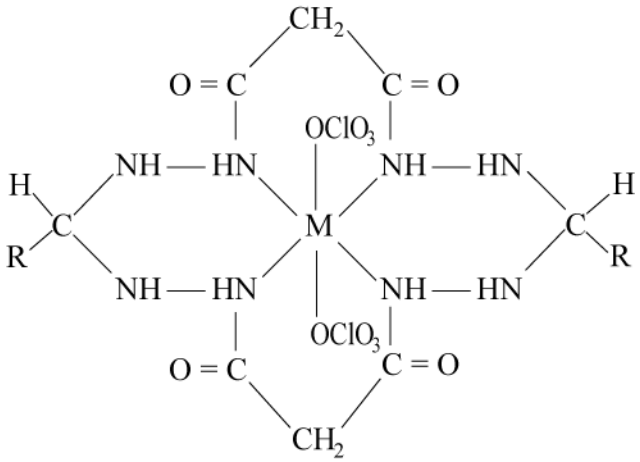



Fig. 4.3.19

M =La (II), where R=H(1), $CH_3$(2),$CH_3CH=CH$,-(3) $C_6H_5CH=CH_2$-(4),$CH_3$-$CH_2$-$CH_2$-(5), $C_6H_6CH$(6)

Table- 4.3.1: Analytical data and other physical properties of compounds (1-6)

| No. | Compounds | %Yield | Colour | Milting point $^0C$ | % M | | Molar conductance ohm$^{-1}$cm$^2$mol$^{-1}$ |
|---|---|---|---|---|---|---|---|
| | | | | | Calculated | Found | |
| 1 | $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 60 | White | 193 | 22.24 | 22.20 | 26.50 |
| 2 | $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 70 | Yellow | 190 | 21.28 | 21.20 | 26.10 |
| 3 | $[La (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 80 | White | 180 | 19.60 | 19.50 | 26.20 |
| 4 | $[La( C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 70 | Yellow | 195 | 19.70 | 19.65 | 27.10 |
| 5 | $[La (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 60 | Whit | 197 | 16.76 | 16.70 | 26.90 |
| 6 | $[La (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 70 | Yellow | 195 | 19.06 | 19.00 | 25.90 |

Table- 4.3.2: Elemental analysis data of compounds (1-6)

| No. | Compounds | %C | | %H | | %N | |
|---|---|---|---|---|---|---|---|
| | | Calculated | Found | Calculated | Found | Calculated | Found |
| 1 | $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 16.18 | 16.20 | 2.86 | 2.86 | 20.07 | 20.00 |
| 2 | $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 36.13 | 36.15 | 3.51 | 3.51 | 14.04 | 14.03 |
| 3 | $[La (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 19.65 | 19.60 | 2.89 | 2.89 | 20.27 | 20.2 |
| 4 | $[La( C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 20.72 | 20.74 | 3.44 | 3.44 | 19.29 | 19.27 |
| 5 | $[La (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 19.38 | 19.35 | 3.23 | 3.23 | 22.62 | 22.60 |
| 6 | $[La (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 41.19 | 41.20 | 4.00 | 4.00 | 16.01 | 16.00 |

Table- 4.3.3: Magnetic Moment Data Of Compounds (1-6)

| No. | Compounds | Sample length, *l* in cm | Weight of the sample, m in gm | Susceptibility of the empty tube, Ro | Susceptibility of the sample with tube ,R | Mass Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | Molecular weight,M | Molar Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | μeff B.M |
|---|---|---|---|---|---|---|---|---|---|
| 1 | $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0695 | -48 | -22 | 1.71 | 593 | 1.19 | 1.69 |
| 2 | $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2.1 | 0.0692 | -47 | -23 | 1.51 | 673 | 1.09 | 1.62 |
| 3 | $[La (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0596 | -40 | -21 | 1.46 | 580 | 1.13 | 1.65 |
| 4 | $[La( C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0559 | -46 | -24 | 1.47 | 756 | 1.32 | 1.78 |
| 5 | $[La (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0630 | -42 | -20 | 1.31 | 494.5 | 1.10 | 1.63 |
| 6 | $[La (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 1.7 | 0.0589 | -46 | -23 | 1.38 | 690.5 | 1.06 | 1.59 |

Table- 4.3.4: Important Infrared Spectral Bands Of Compounds (1-6)

| No. | Compounds | $\nu$(C-H) $cm^{-1}$ | $\nu$(C=O $cm^{-1}$) | $\nu$(N-H) $cm^{-1}$ | $\nu$(M-N) $cm^{-1}$ | $\nu(ClO_4)$ $cm^{-1}$ |
|---|---|---|---|---|---|---|
| 1 | $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 3047 | 1664 | 3251 | 420 | 1180,1089,623 |
| 2 | $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2920 | 1649 | 3282 | 430 | 1105,979,623 |
| 3 | $[La (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 2960 | 1640 | 3250 | 416 | 1150,1060,620 |
| 4 | $[La( C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 2967 | 1650 | 3261 | 408 | 1160,1040,623 |
| 5 | $[La (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 3040 | 1660 | 3254 | 409 | 1140,1080,621 |
| 6 | $[La (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 3070 | 1680 | 3270 | 406 | 1170,1090,625 |

Table- 4.3.5: U.V- Visible Adsorption Maxima Of Compounds (1-6)

| **No.** | **Compounds** | λ max (n,m) |
|---|---|---|
| **1** | $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 300,490 |
| **2** | $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 290,490 |
| **3** | $[La\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 297,500 |
| **4** | $[La(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 290,490 |
| **5** | $[La\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 290,490 |
| **6** | $[La\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 290,550 |

Table- 4.3.6: Thermal Analysis Data of Compounds (1-6)

| No. | Compounds | %M (Ligand) | | | %M Metal oxide(MO) | | |
|---|---|---|---|---|---|---|---|
| | | Tem$^0$C | Calculated | Found | Tem$^0$C | Calculated | Found |
| 1 | $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 135.02 | 83.22 | 83.20 | 599.00 | 37.66 | 37.60 |
| 2 | $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 135.00 | 86.21 | 86.20 | 599.00 | 36.14 | 36.10 |
| 3 | $[La\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 130.90 | 87.14 | 87.10 | 550.60 | 33.72 | 33.60 |
| 4 | $[La(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 130.45 | 88.91 | 88.50 | 450.00 | 29.06 | 29.00 |
| 5 | $[La\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 140.50 | 88.23 | 88.20 | 530.00 | 30.86 | 30.40 |
| 6 | $[La\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 137.45 | 87.07 | 87.00 | 540.00 | 33.89 | 33.30 |

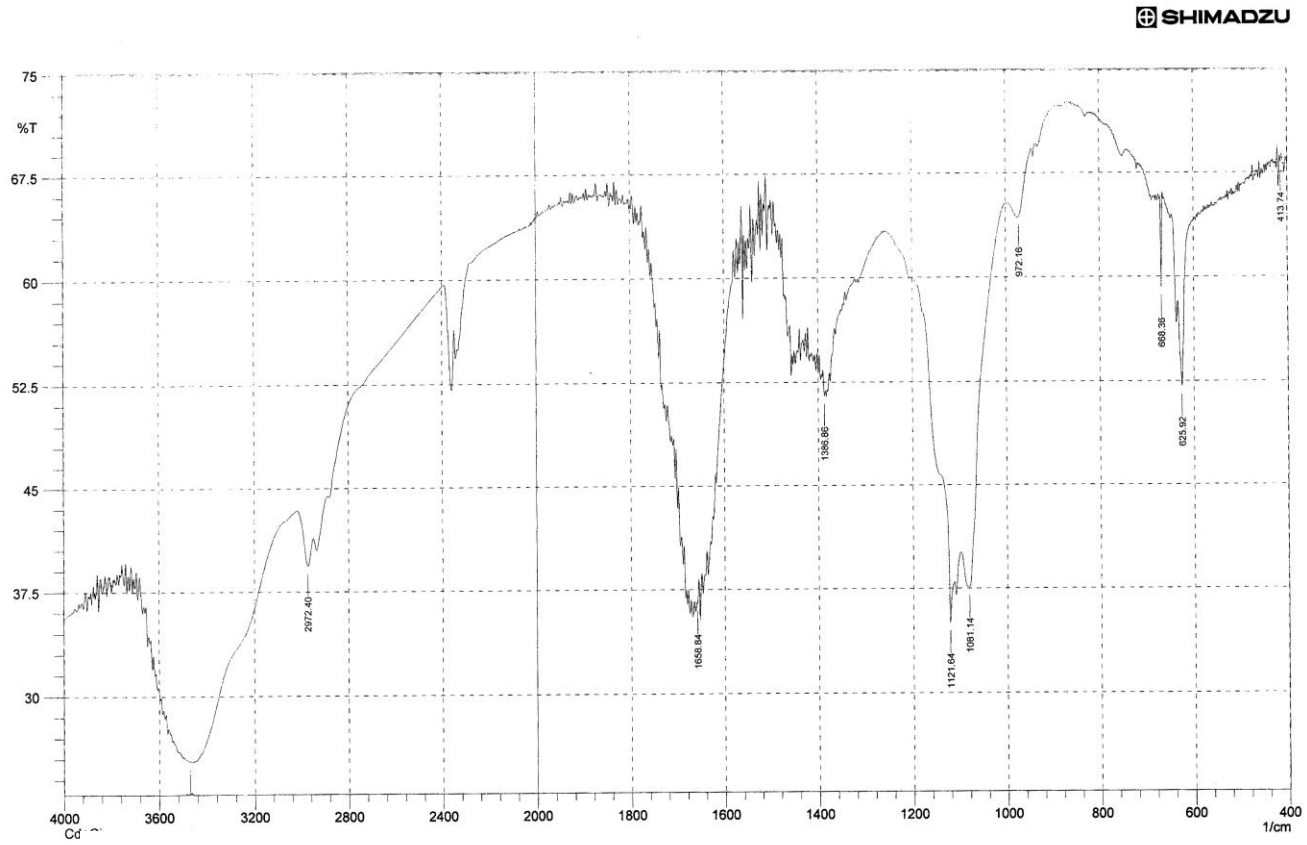



Fig-4.3.1: Infrared spectrum [La $(C_8H_{16}N_8O_4)(ClO_4)_2$]complex 1

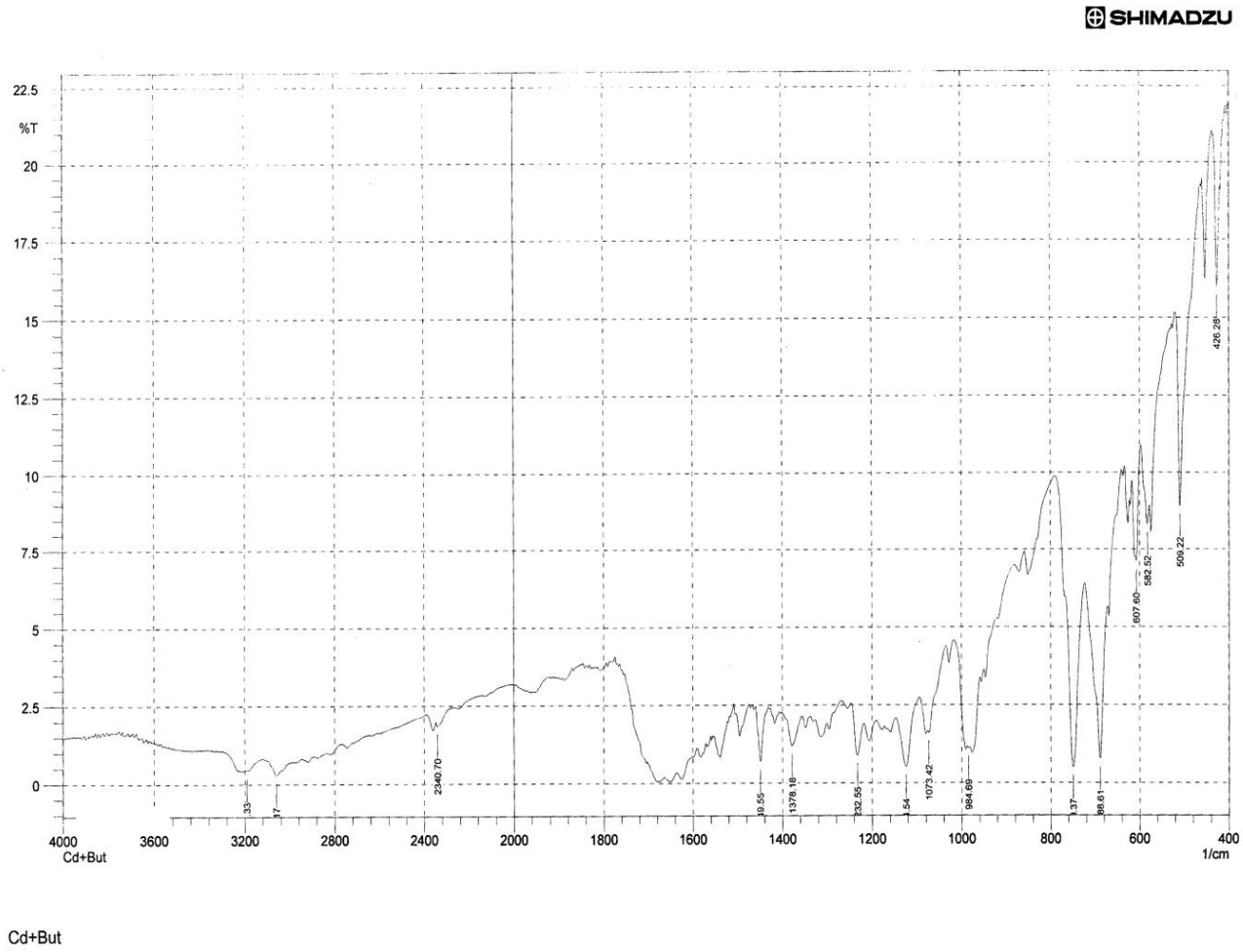



Fig-4.3.2: Infrared spectrum $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

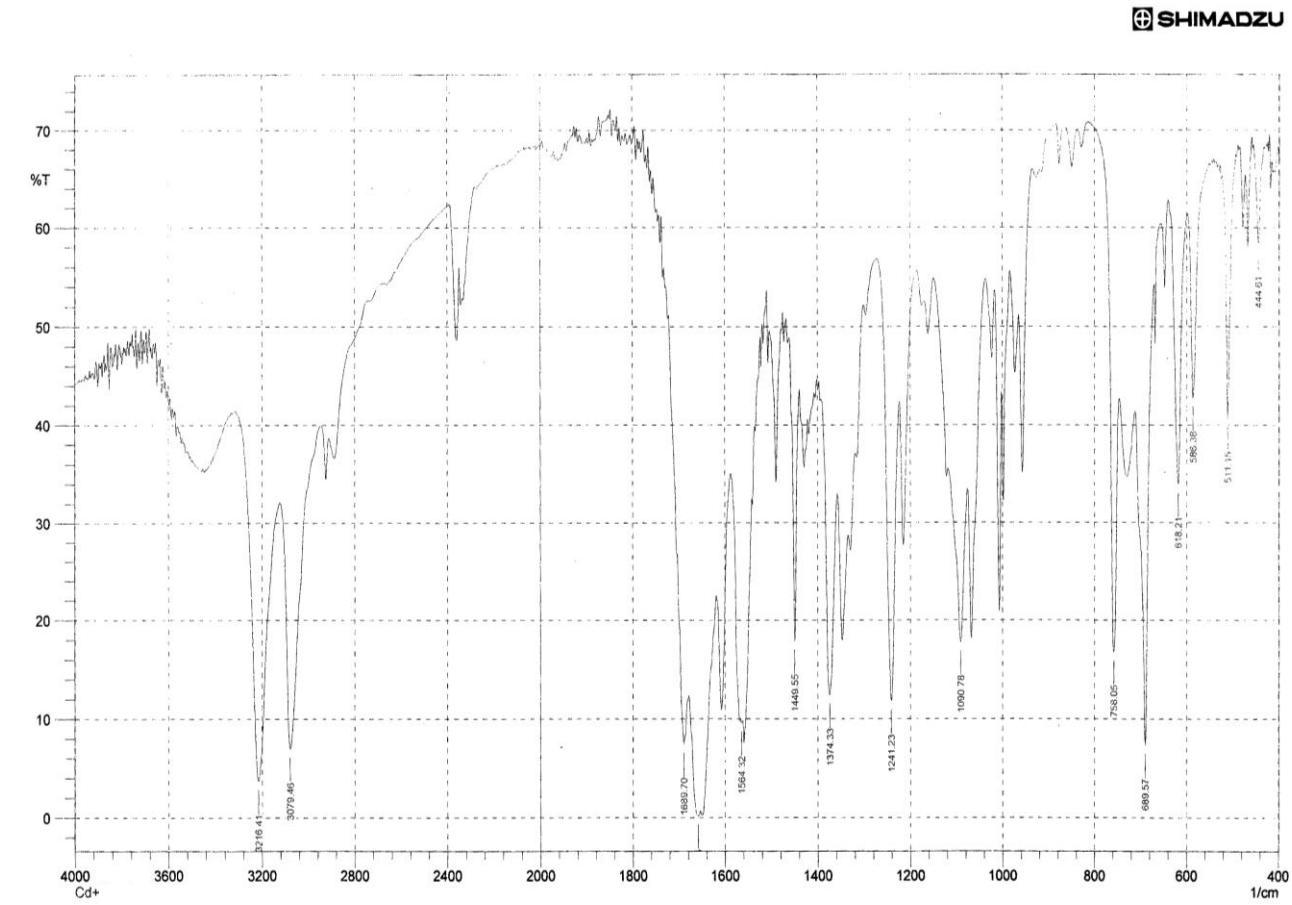
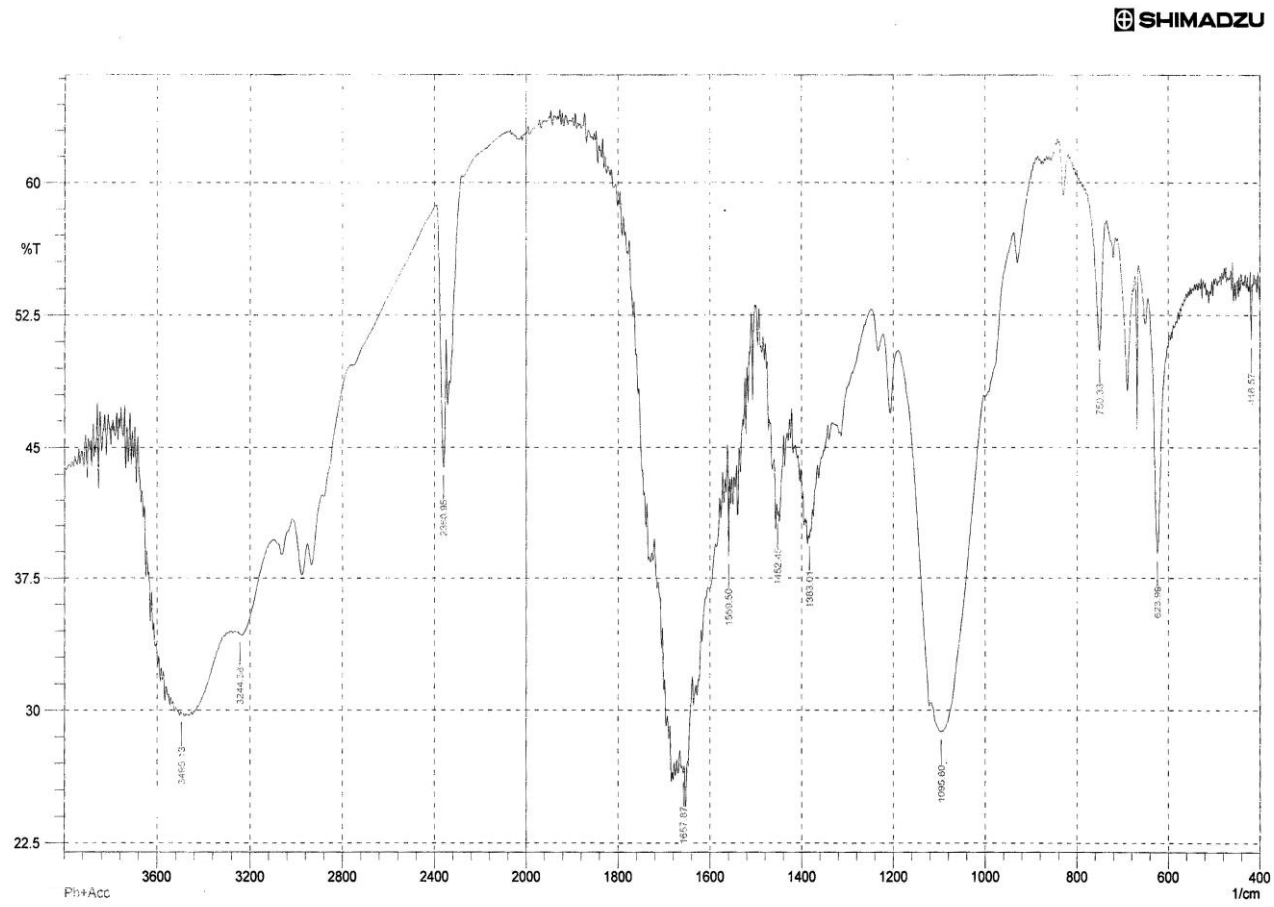



Fig-4.3.3 :Infrared spectrum $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3




Pb+Acc

Fig-4.3.4:Infrared spectrum $[La(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4.

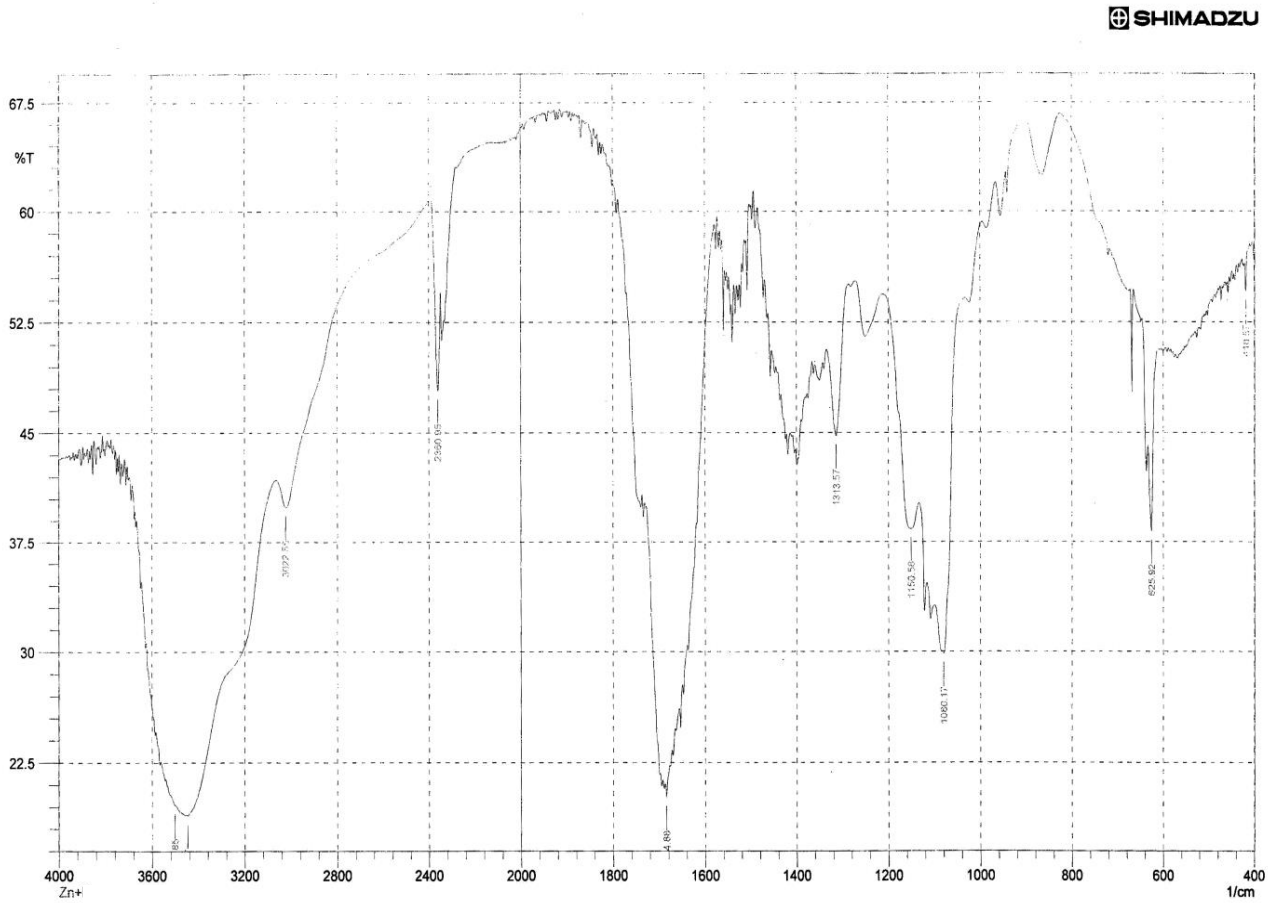



Fig-4.3.5: Infrared spectrum $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5.

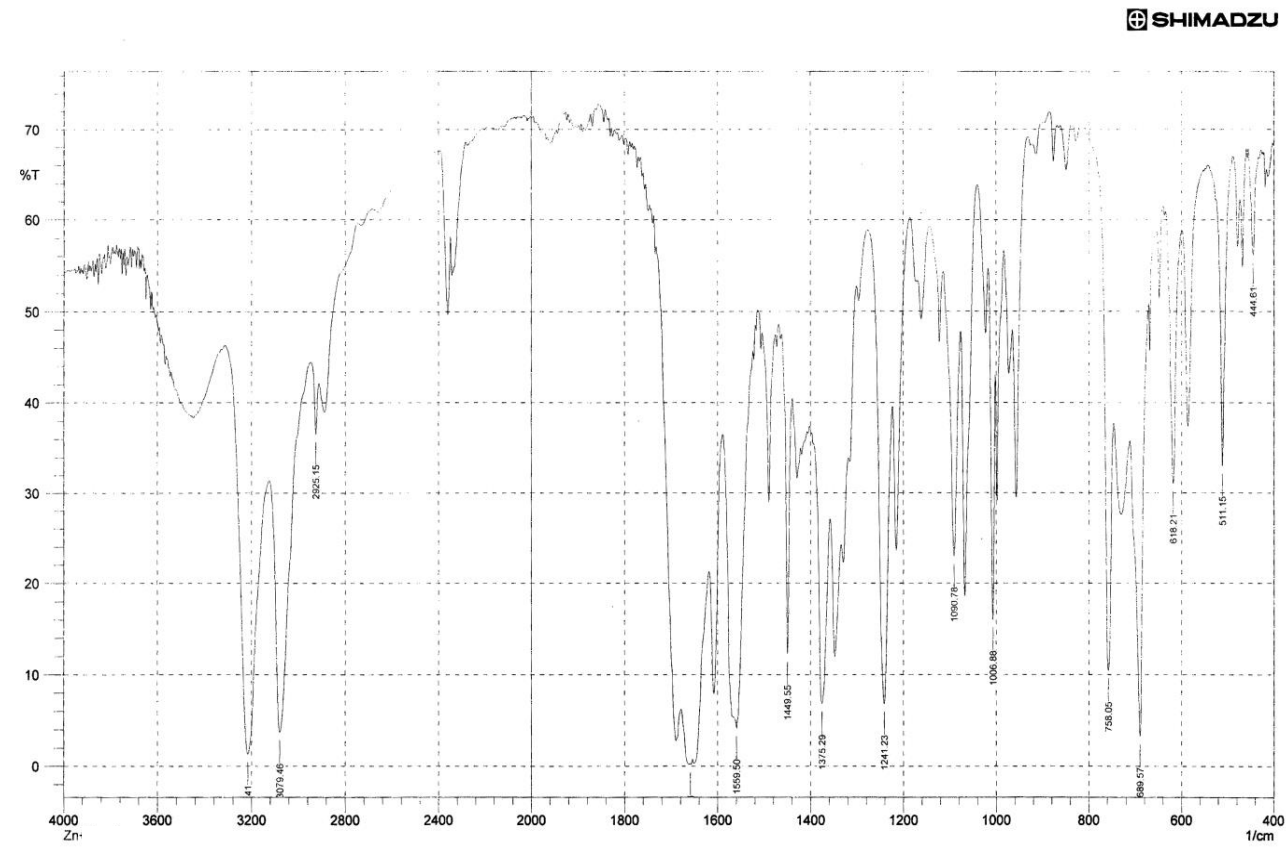



Fig-4.3.6: Infrared spectrum $[La(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex 6.

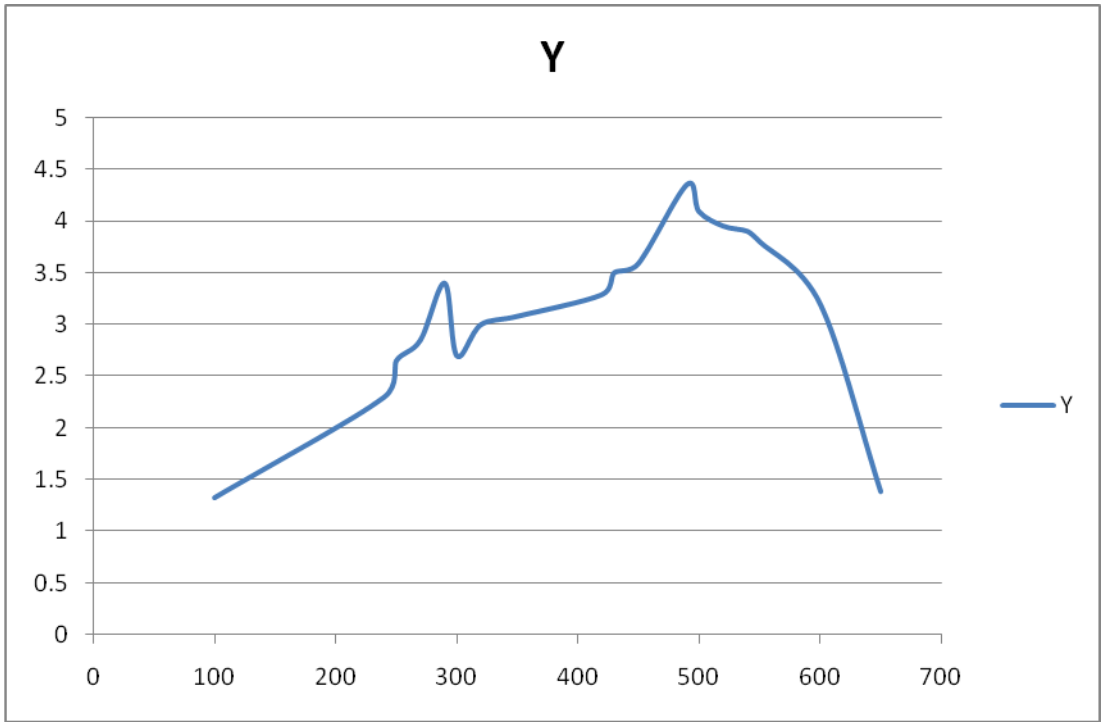



Fig-4.3.7: UV- Visible spectrum of $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

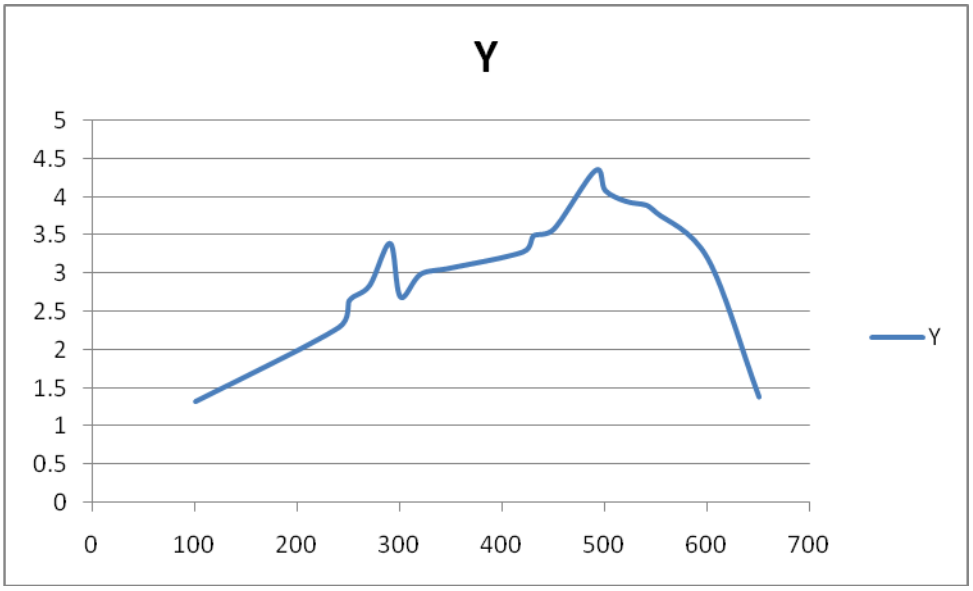



Fig-4.3.8: UV-Visible spectrum of $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

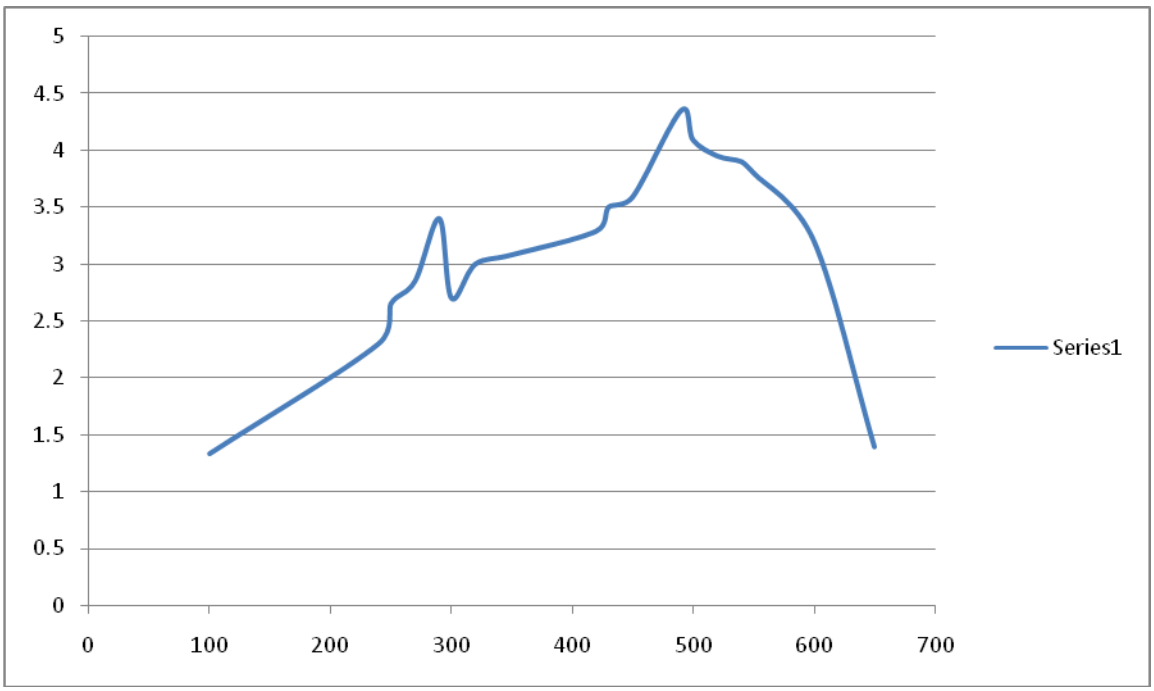



Fig-4.3.9 :UV- Visible spectrum of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

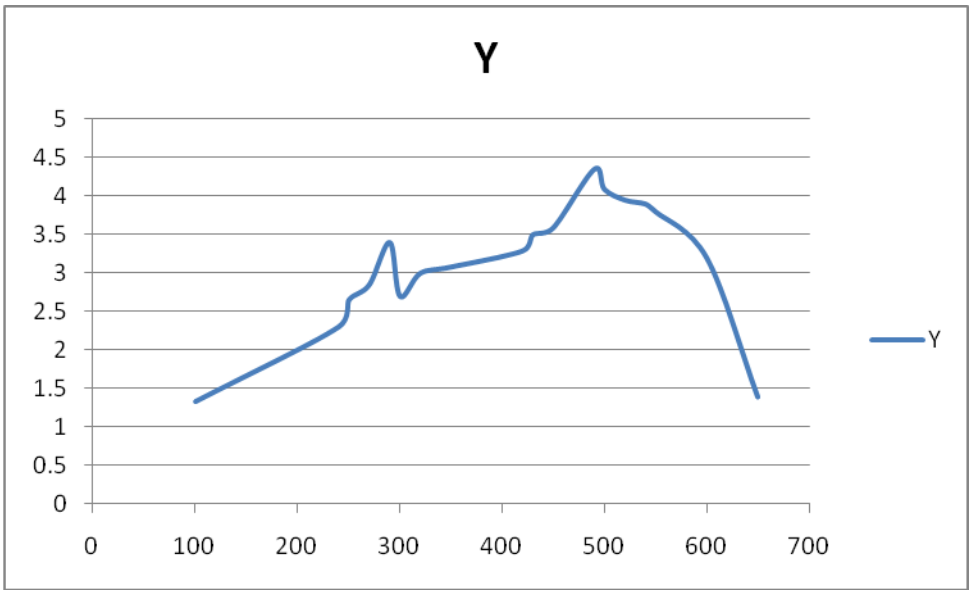



Fig-4.3.10 :UV- Visible spectrum of $[La(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4**.**

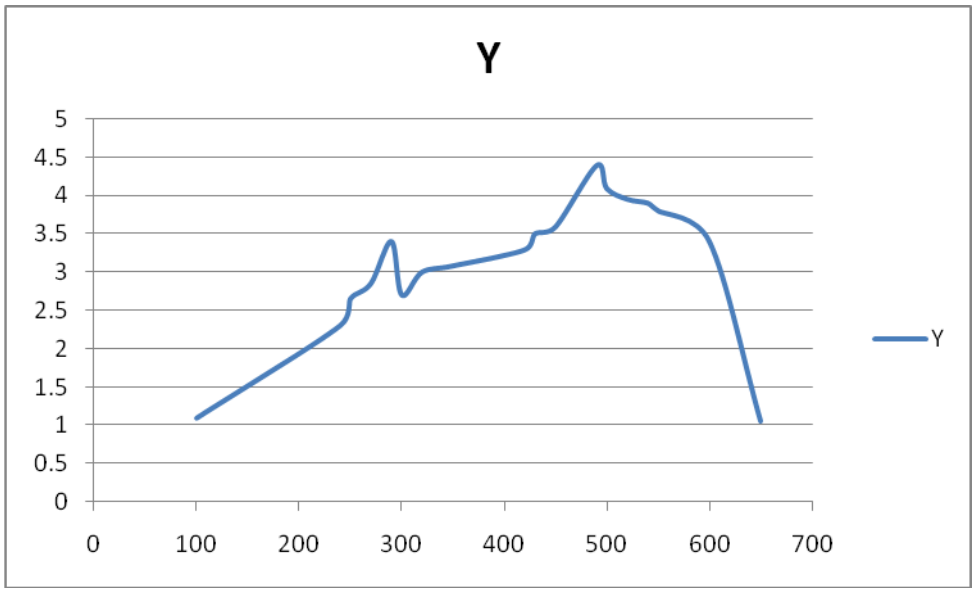



Fig-4.3.11 :UV- Visible spectrum of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5**.**

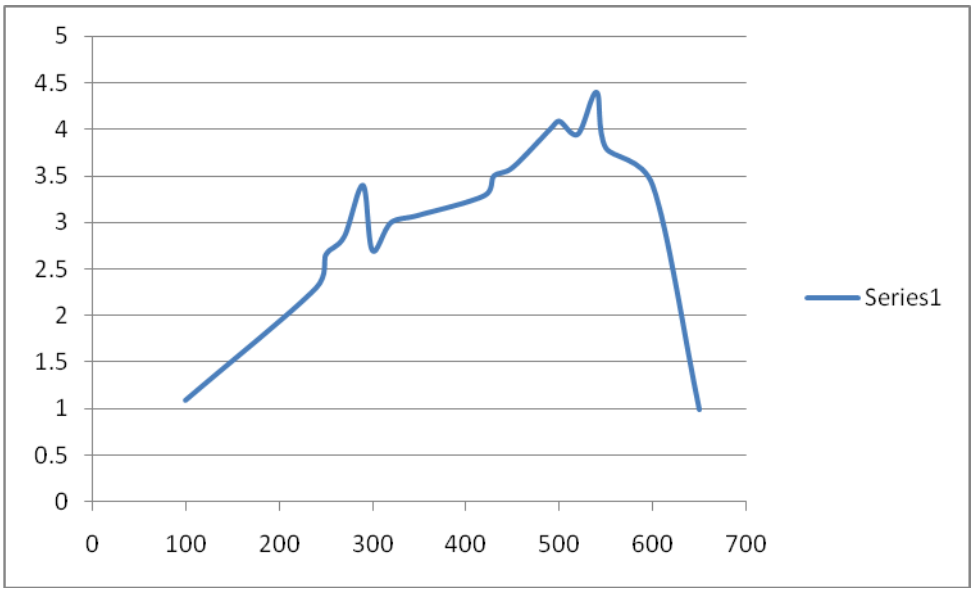



Fig-4.3.12 : UV- Visible spectrum of $[La(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex 6

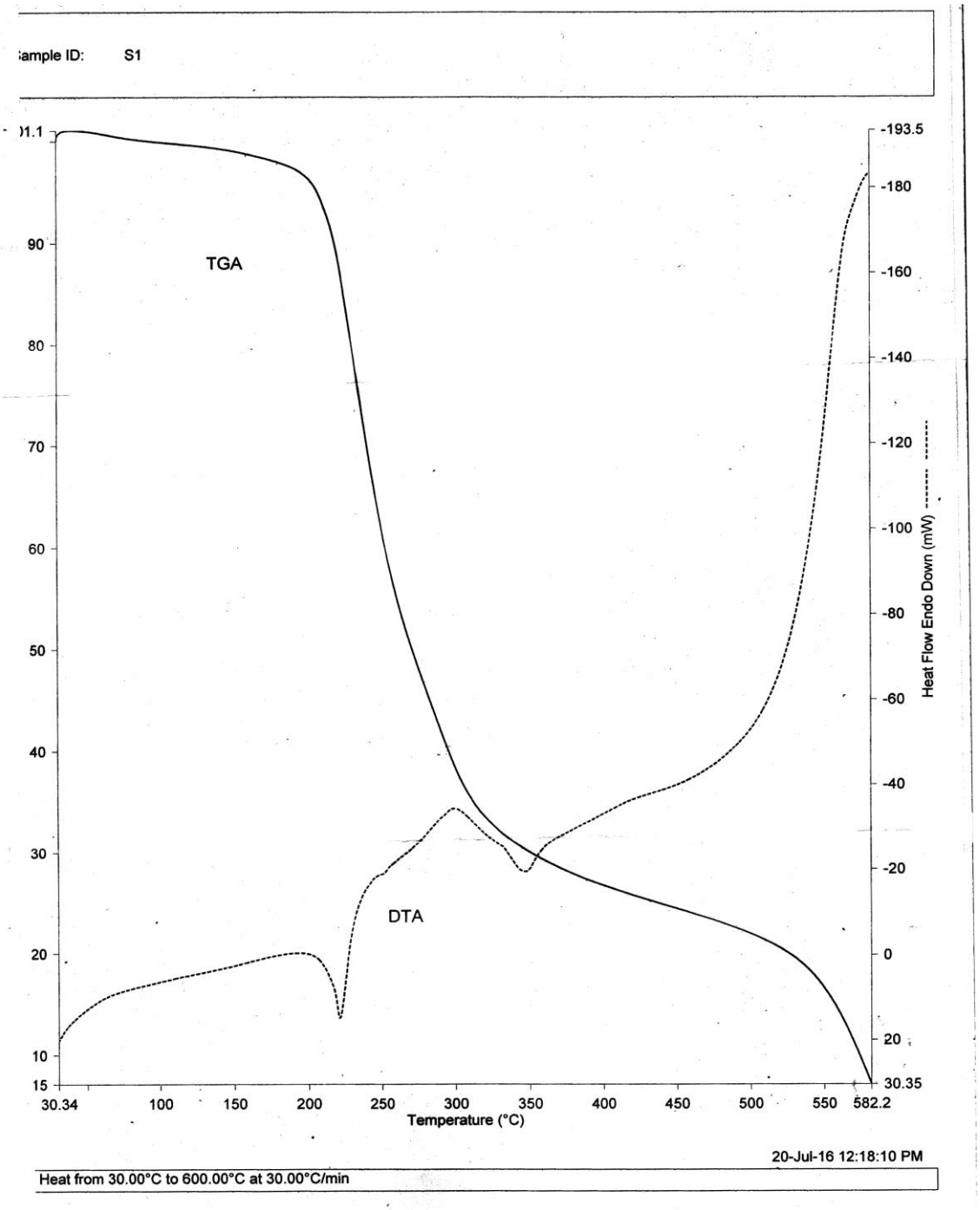



**Fig-4.3.13: TGA & DTA spectrum $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.**

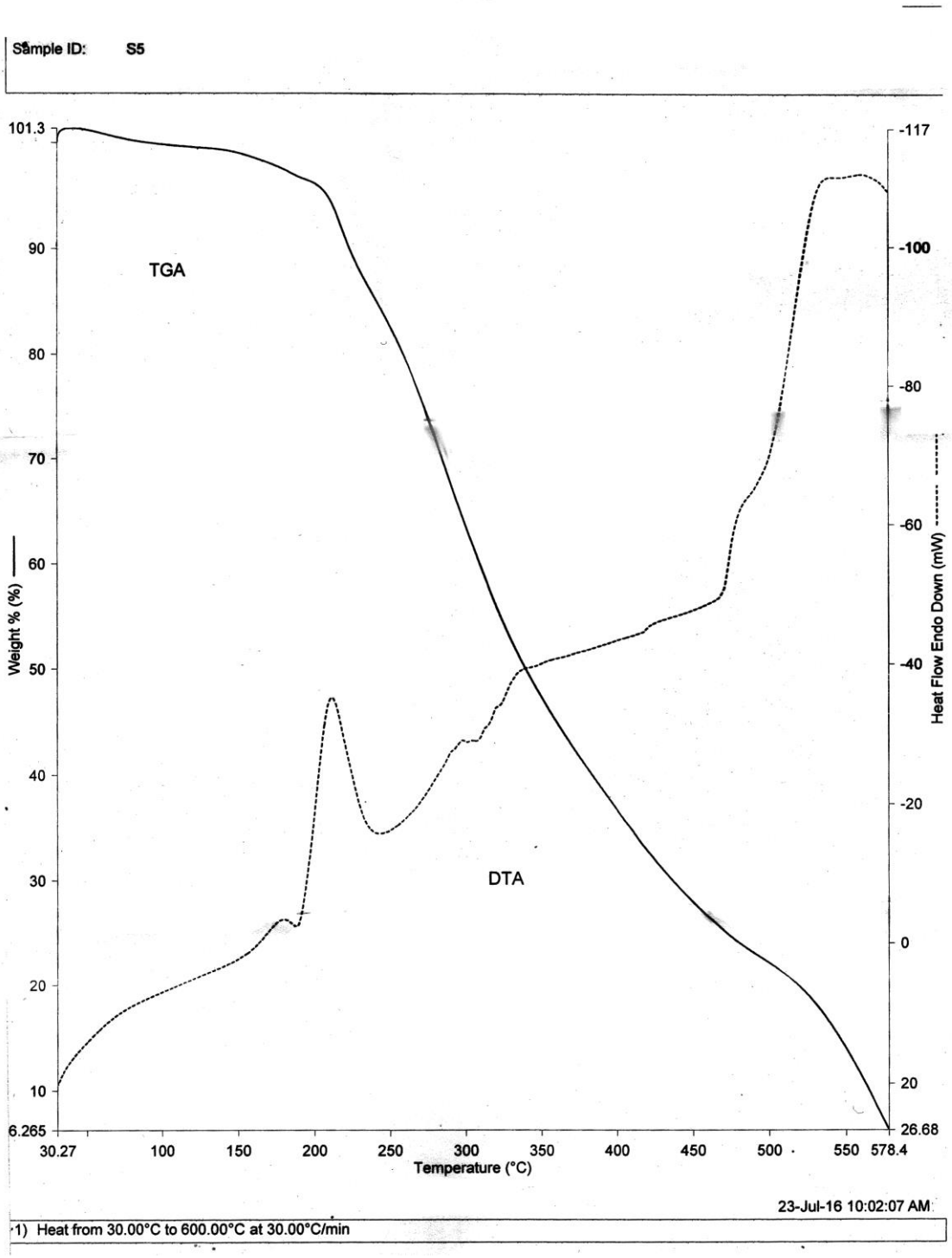



**Fig-4.3.14: TGA & DTA spectrum[$La(C_{10}H_{20}N_8O_4)(ClO_4)_2$]complex 2**

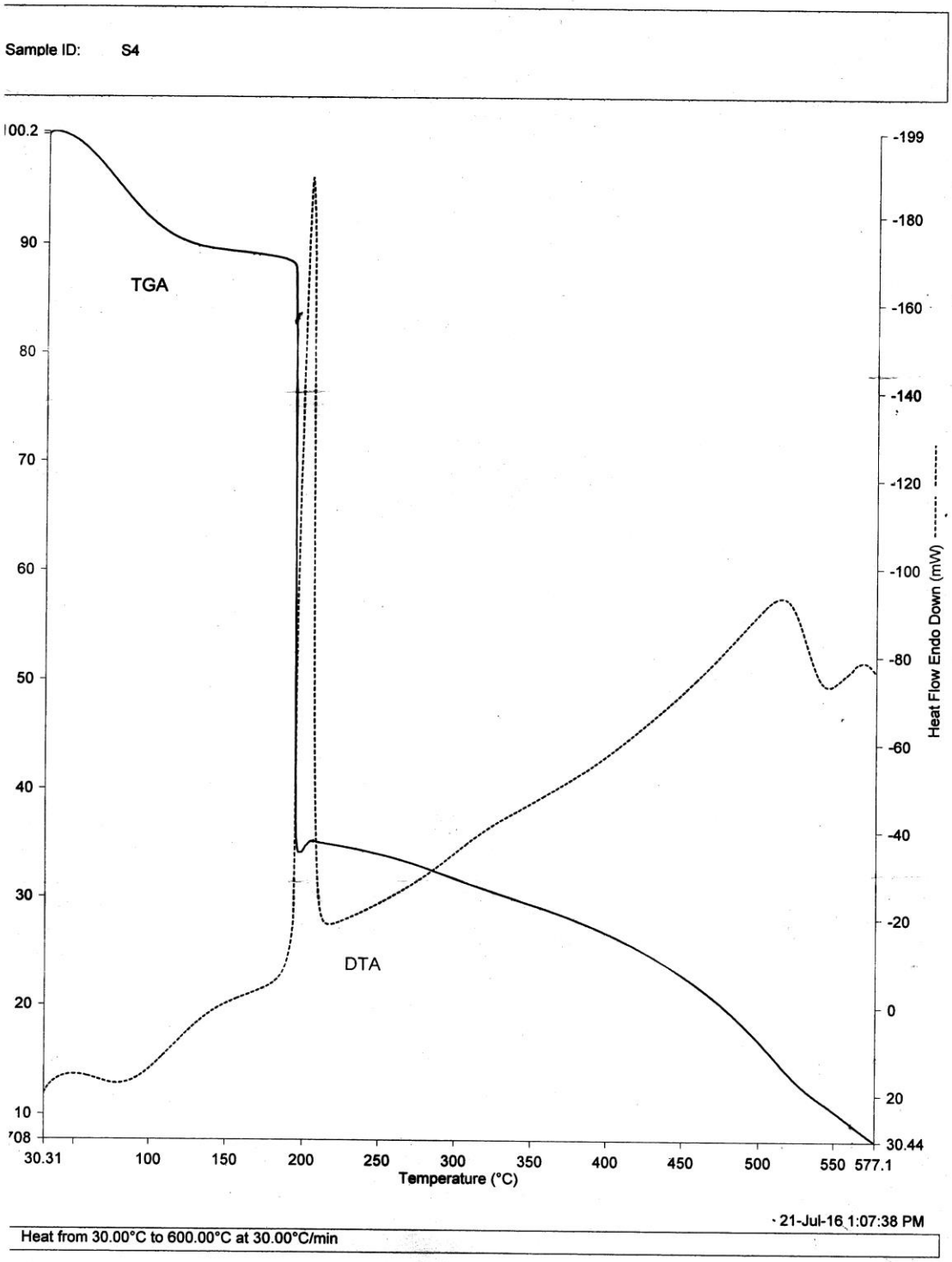



**Fig-4.3.15 : TGA & DTA spectrum $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3**

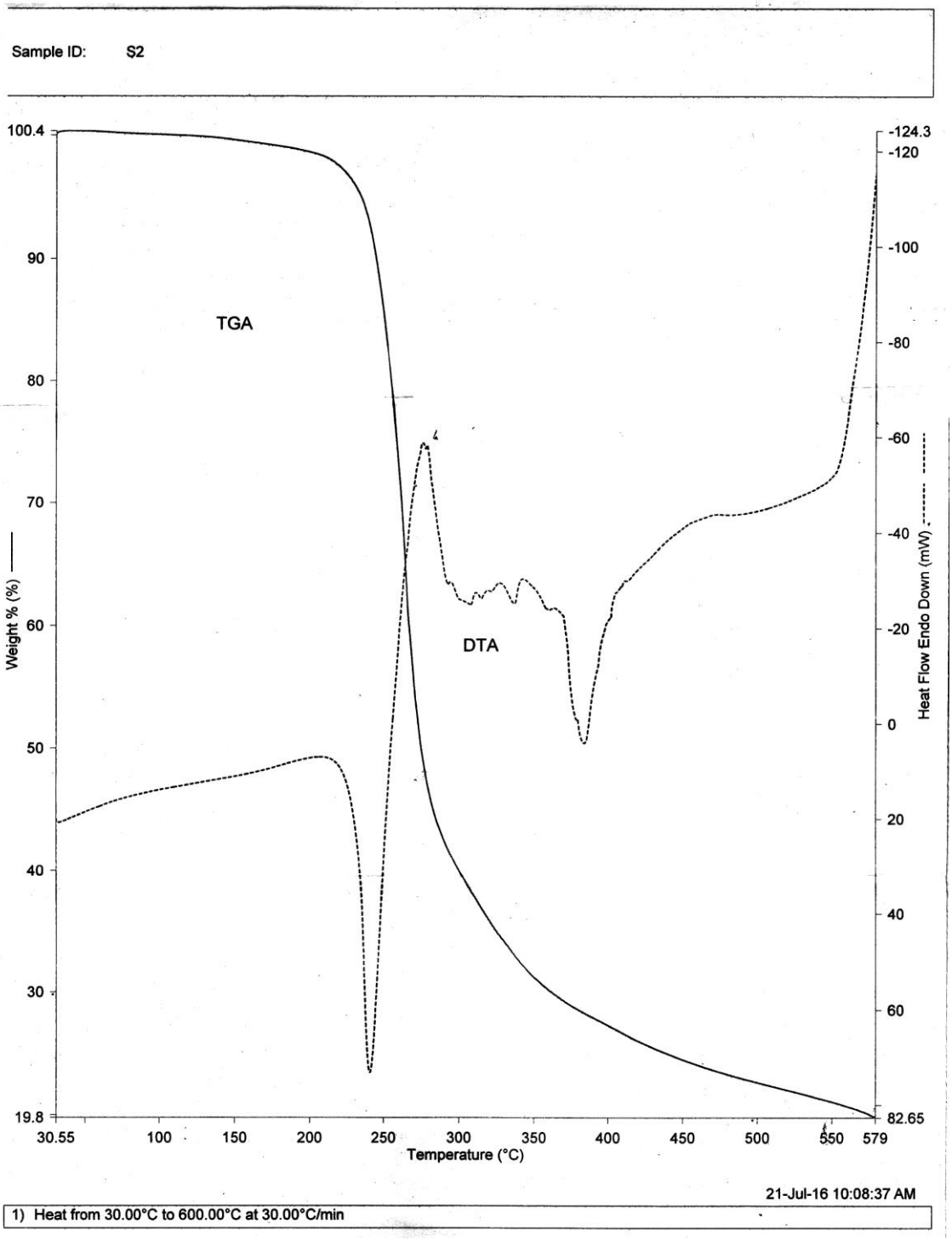



**Fig-4.3.16: TGA & DTA spectrum $[La(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4**

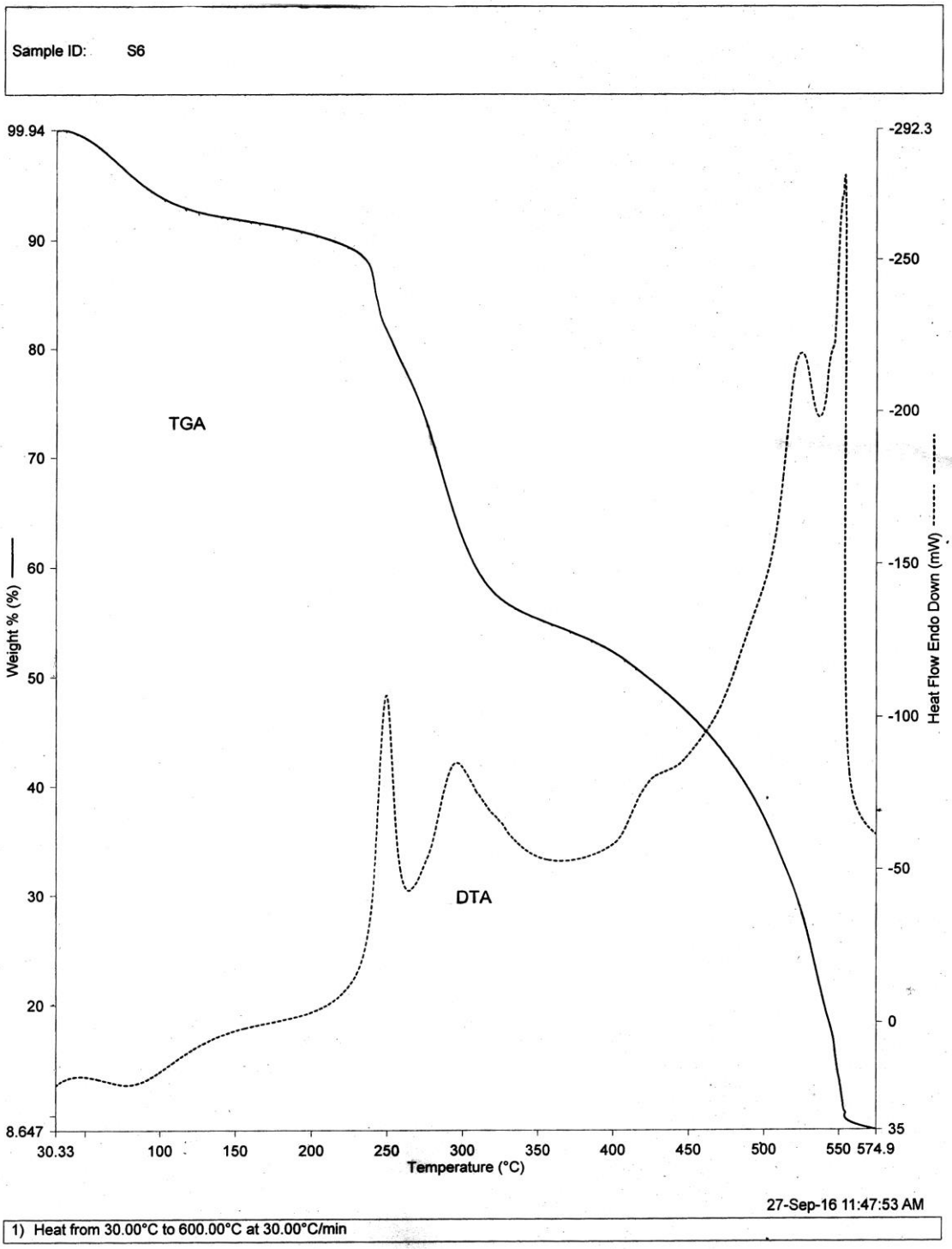



**Fig-4.3.17: TGA & DTA spectrum $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5.**

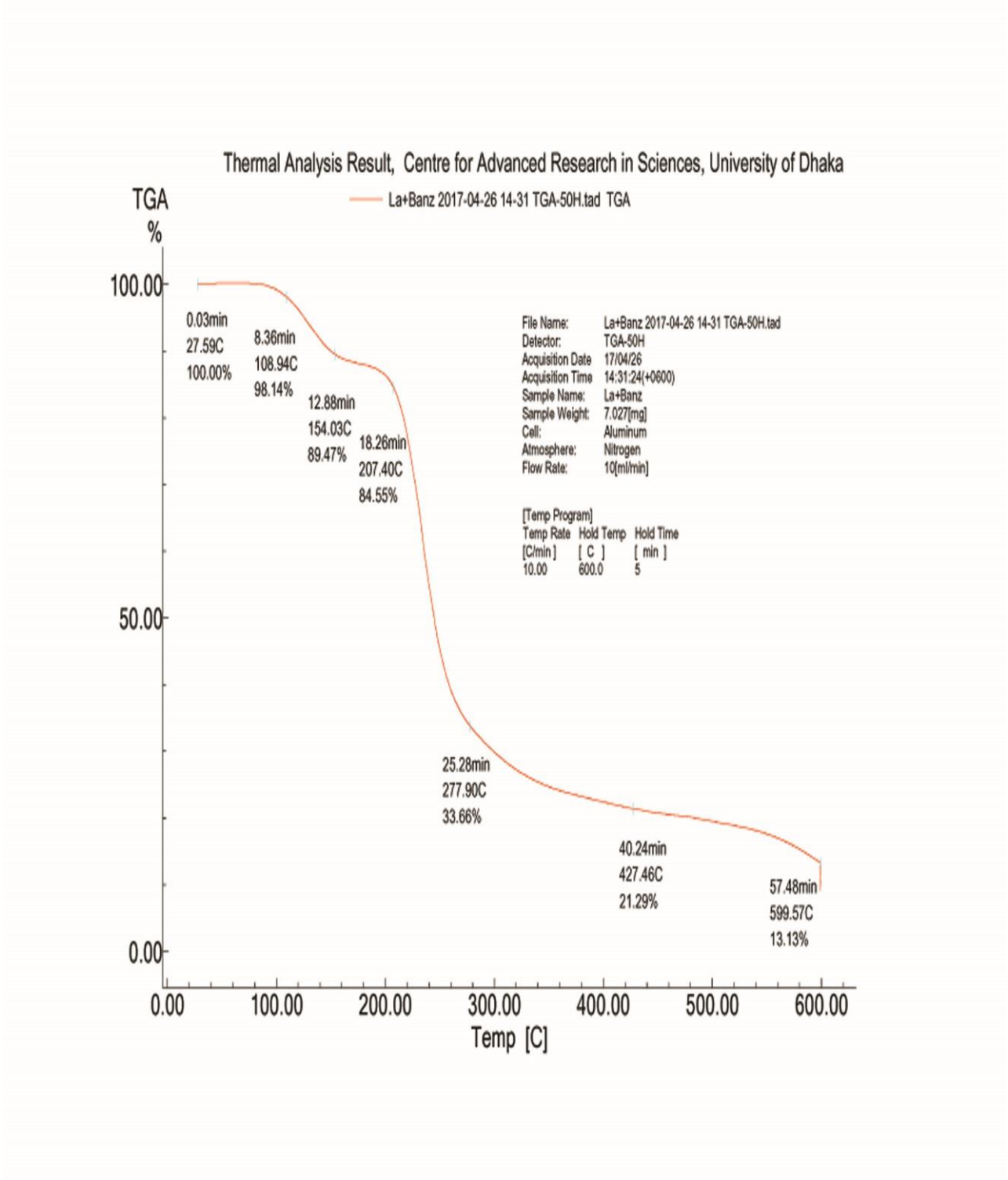



**Fig-4.3.18 : TGA & DTA spectrum $La(C_{20}H_{24}N_8O_4)(ClO4)_2]$ complex 6.**

## General information

| | | | |
|---|---|---|---|
| Analysis date | 2017/02/02 16:32:42 | | |
| Sample name | La+Ci+L | Measurement date | 2017/02/02 16:09:39 |
| File name | La+Ci+L.raw | Operator | User1 |
| Comment | | | |

## Measurement profile

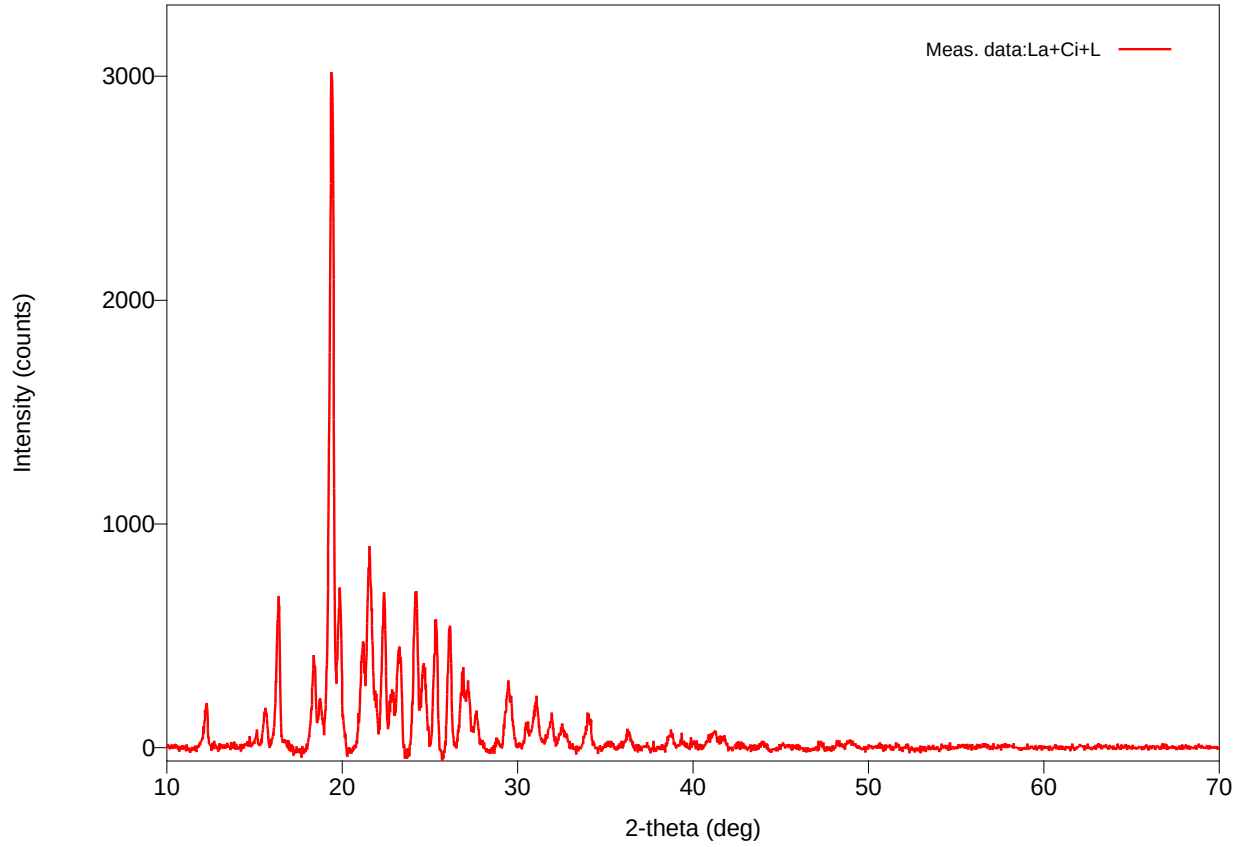


Table.4.3.7: X-ray Analysis data.

**Peak list**

| No. | 2-theta(deg) | d(ang.) | FWHM(deg) | Asym. factor | Rel. int. I(a.u.) |
|---|---|---|---|---|---|
| 1 | 12.302(4) | 7.189(2) | 0.231(12) | 5(3) | 6.45 |
| 2 | 15.156(11) | 5.841(4) | 0.23(3) | 4(4) | 1.91 |
| 3 | 15.661(13) | 5.654(4) | 0.227(13) | 2.5(6) | 6.49 |
| 4 | 16.397(7) | 5.401(2) | 0.212(5) | 2.7(5) | 23.79 |
| 5 | 18.412(15) | 4.815(4) | 0.254(14) | 1.8(5) | 13.74 |
| 6 | 18.730(14) | 4.734(3) | 0.29(5) | 0.7(5) | 7.89 |
| 7 | 19.438(4) | 4.5628(10) | 0.244(5) | 2.39(18) | 100.00 |
| 8 | 19.901(16) | 4.458(3) | 0.53(7) | 4.4(10) | 40.98 |
| 9 | 21.198(18) | 4.188(4) | 0.251(16) | 2.4(9) | 16.09 |
| 10 | 21.520(6) | 4.1259(11) | 0.393(16) | 0.57(14) | 44.06 |
| 11 | 22.401(13) | 3.966(2) | 0.248(12) | 2.0(4) | 23.32 |
| 12 | 22.86(3) | 3.887(6) | 0.28(4) | 2.3(16) | 9.35 |
| 13 | 23.28(2) | 3.817(3) | 0.29(2) | 1.9(6) | 19.03 |
| 14 | 24.187(12) | 3.6766(18) | 0.251(12) | 1.1(2) | 25.18 |
| 15 | 24.66(2) | 3.607(3) | 0.33(3) | 1.2(4) | 16.36 |
| 16 | 25.341(12) | 3.5117(17) | 0.222(10) | 1.5(3) | 17.94 |
| 17 | 26.14(6) | 3.406(8) | 0.20(6) | 2(3) | 18.00 |
| 18 | 27.51(4) | 3.240(4) | 1.08(11) | 3.2(18) | 22.49 |
| 19 | 29.476(16) | 3.0278(17) | 0.391(13) | 0.93(15) | 15.44 |
| 20 | 30.446(7) | 2.9335(7) | 0.22(4) | 0.20(13) | 3.07 |
| 21 | 31.043(18) | 2.8785(16) | 0.34(2) | 1.2(3) | 10.81 |
| 22 | 31.96(2) | 2.798(2) | 0.34(3) | 3.1(11) | 7.05 |
| 23 | 32.51(3) | 2.752(3) | 0.44(3) | 0.68(19) | 5.86 |
| 24 | 34.03(3) | 2.6320(19) | 0.336(19) | 1.2(3) | 6.40 |
| 25 | 36.31(6) | 2.472(4) | 0.30(5) | 1.0(8) | 3.09 |
| 26 | 38.742(19) | 2.3223(11) | 0.27(5) | 2(2) | 3.21 |
| 27 | 41.11(9) | 2.194(5) | 0.78(9) | 0.5(3) | 8.18 |

# RESULT AND DISCUSSION

## 4.4 MACROCYCLIC COMPLEXES OF Cd (II)

Reactions of malonodihydrazide with Cd(II) perchlorate hexahydrate in presence of formaldehyde, acetaldehyde, butanal dehyde, crotonaldehyde, cinnamaldehyde, and benzaldehyde give some 16 member macrocyclic complex as described in sec. 3.5

Complexes (1-6) are characterized on the basis of elemental analysis, magnetic moment & conductance measurements, UV-visible spectra & infrared studies, thermal studies and other physical properties, like melting point, solubility, colour etc.

Molar conductance data of the complexes (1-6) are shown in Table 4.4.1 The conductance values of the complexes suggested that they are non-electrolytic in nature[24].

The infrared spectra of the complexes (1-6) are shown as spectral data (Table 4.4.4) of the complexes showed a strong and broad band at (3250-3270) $cm^{-1}$ which is assigned for the ν(NH) stretching[25].

Due to coordination the ν(N-H) stretching of the amide group goes to the higher field at (3250, 3270) $cm^{-1}$ region as compared to the starting material malonodihydrazide[26]. In the complexes the terminal-$NH_2$ group of malonodihydrazide condensed with the aldehyde moiety form a new secondary[1]-NH group which may appear at the same region (or overlape) as to the amide-NH group as a result the ν(N-H) band appear as a strong and

broad band. [The starting material malonodihydrazide have three ν(N-H) bands at (3250, 3213, 3270) $cm^{-1}$. The bands at (3250, 3270) $cm^{-1}$ for the asymmetrie and symmetric ν(N-H) stretching of the terminal-$NH_2$ moiety and 3261 $cm^{-1}$ for amidic (N-H) group]. The complexes showed a broad band at (2920-3070) $cm^{-1}$ is suggested for the ν(C-H) stretching of aliphatic moiety[32]. The complexes showed a strong band at (1640-1680) $cm^{-1}$ which represent the ν(C=O) of NH-NH-CO-$CH_2$ moity[27]. Three or four band at (621-1180) $cm^{-1}$ region also indicated the $\nu_1,\nu_2,\nu_3,\nu_4$ bands of ($ClO^-_4$) moiety. These stretching frequency is suggested the coordination of perchlorate to the metal through the O atom[28]. A medium band at (406-430) $cm^{-1}$ region is tentatively attributed to the ν(M-N) mode[29,30.] indicating the coordination of the lignad to the metal through the nitrogen atom.

The magnetic moment measurement data (Table 4.4.3) of the Cd(II) complexes (1-6) showed (1.59-1.78) B.M. These values correspond to no unpaired electrons of Cd(II) $d^{10}$system suggest the octahedral environment of the complexes which are consistent with the literature value[1]. The elemental analyses (C, H and N) (Table 4.4.2) and metal estimation data (Table 4.4.3) of the complexes are consistent with the proposed formula.

The UV-visible spectra of the complexes (1-6) are shown (Table 4.4.5) band at 320,480 nm, (1-6) at represent the d-d transition of $^4A_{2g}$ (F) $\rightarrow$ $^4T_{1g}$(F), $^4A_{2g}$(F) $\rightarrow$ $^4T_{1g}$(P), which suggested the octahedral geometry of the Cd (II) complexes[31,32].

Thermal studies**:** The thermal properties of metal (II) complexes were investigated by thermo grams (TGA,DTA) and are shown in (Fig 4.4.13-4.4.18) and the corresponding thermal analysis is presented in (Table.4.4.6). In the case of complex (1-6) the decomposition occurs in the (230-325)$^0$C range. There is no mass loss up to 230$^0$C. The first stage of decomposition starts at 230$^0$C and end at 230$^0$C with a corresponding weight loss 25%. Which is accompanied by endothermic effect in the DTA curve in the range 225$^0$C which is accompanied by weight loss confirming the second stage of decomposition is observed at 225-350$^0$C (60% wt. loss).meanwhile the DTA curve exhibits endothermic effect in the range 325$^0$C which is accompanied by weight loss confirming.

On the basis of elemental analysis magnetic moment and conductance measurements, thermal studies UV Visible spectra, infrared spectra and other physical properties the suggested structure of the complexes are octahedral in nature as in Fig.4.419.

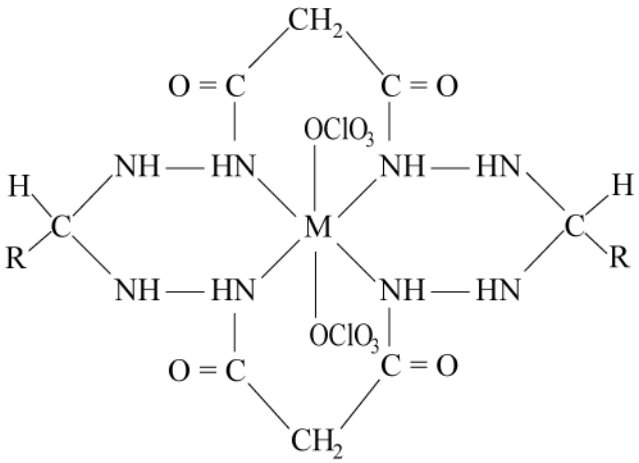



Fig. 4.4.19

M =Cd (II), where R=H(1), $CH_3$(2),$CH_3CH=CH$,(3) $C_6H_5CH=CH_2$-(4),$CH_3$-$CH_2$-$CH_2$-(5), $C_6H_6CH$(6)

Table- 4.4.1: Analytical data and other physical properties of compounds (1-6)

| No. | Compounds | %Yield | Colour | Milting point $^0C$ | % M | | Molar conductance $ohm^{-1}cm^2mol^{-1}$ |
|---|---|---|---|---|---|---|---|
| | | | | | Calculated | Found | |
| 1 | $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 60 | White | 217 | 18.69 | 18.60 | 10.48 |
| 2 | $[Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 70 | Yellow | 210 | 17.86 | 17.80 | 15.10 |
| 3 | $[Cd (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 70 | Red | 215 | 16.39 | 16.36 | 13.50 |
| 4 | $[Cd( C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 60 | Red | 220 | 16.49 | 16.40 | 16.08 |
| 5 | $[Cd (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 70 | Ashe | 215 | 13.94 | 13.90 | 14.00 |
| 6 | $[Cd (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 75 | Black | 220 | 14.91 | 14.90 | 12.49 |

Table- 4.4.2: Elemental analysis data of compounds (1-6)

| No. | Compounds | %C | | %H | | %N | |
|---|---|---|---|---|---|---|---|
| | | Calculated | Found | Calculated | Found | Calculated | Found |
| **1** | $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 16.02 | 16.20 | 2.67 | 2.86 | 18.69 | 19.00 |
| **2** | $[Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 19.13 | 19.18 | 3.18 | 3.51 | 17.86 | 17.80 |
| **3** | $[Cd (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 24.59 | 24.50 | 4.09 | 4.00 | 16.39 | 16.30 |
| **4** | $[Cd( C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 24.74 | 24.70 | 3.53 | 3.50 | 16.49 | 16.40 |
| **5** | $[Cd (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 35.86 | 35.80 | 3.48 | 3.40 | 13.94 | 13.90 |
| **6** | $[Cd (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 31.95 | 31.90 | 3.19 | 3.20 | 14.91 | 14.88 |

Table- 4.4.3: Magnetic moment data of compounds (1-6)

| No. | Compounds | Sample length, *l* in cm | Weight of the sample, m in gm | Susceptibility of the empty tube, Ro | Susceptibility of the sample with tube ,R | Mass Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | Molecular weight,M | Molar Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | μeff B.M |
|---|---|---|---|---|---|---|---|---|---|
| 1 | $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0695 | -48 | -22 | 1.71 | 599 | 0.712 | 1.30 |
| 2 | $[Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2.1 | 0.0692 | -47 | -23 | 1.51 | 627 | 0.712 | 1.30 |
| 3 | $[Cd\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0596 | -40 | -21 | 1.46 | 683 | 0.771 | 1.36 |
| 4 | $[Cd(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0559 | -46 | -24 | 1.47 | 679 | 0.896 | 1.46 |
| 5 | $[Cd\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0630 | -42 | -20 | 1.31 | 803 | 0.883 | 1.45 |
| 6 | $[Cd\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 1.7 | 0.0589 | -46 | -23 | 1.38 | 751 | 1.036 | 1.57 |

Table- 4.4.4: Important infrared spectral bands of compounds (1-6)

| No. | Compounds | $\nu$(C-H) $cm^{-1}$ | $\nu$(C=O ) $cm^{-1}$ | $\nu$(N-H) $cm^{-1}$ | $\nu$(M-N) $cm^{-1}$ | $\nu(ClO_4)$ $cm^{-1}$ |
|---|---|---|---|---|---|---|
| 1 | $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 3047 | 1664 | 3251 | 420 | 1180,1089,623 |
| 2 | $[Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2920 | 1649 | 3282 | 430 | 1105,979,623 |
| 3 | $[Cd\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 2960 | 1640 | 3250 | 416 | 1150,1060,620 |
| 4 | $[Cd(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 2967 | 1650 | 3261 | 408 | 1160,1040,623 |
| 5 | $[Cd\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 3040 | 1660 | 3254 | 409 | 1140,1080,621 |
| 6 | $[Cd\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 3070 | 1680 | 3270 | 406 | 1170,1090,625 |

Table- 4.4.5: U.V- Visible adsorption maxima of compounds (1-6)

| **No.** | **Compounds** | λ max (n,m) |
|---|---|---|
| **1** | $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 290,520 |
| **2** | $[Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 305,520 |
| **3** | $[Cd\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 300,490 |
| **4** | $[Cd(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 295,600 |
| **5** | $[Cd\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 290,500 |
| **6** | $[Cd\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 289,490 |

Table- 4.4.6: Thermal analysis data of compounds (1-6)

| No. | Compounds | %M Ligand | | | %M Metal oxide(MO) | | |
|---|---|---|---|---|---|---|---|
| | | $Tem^0C$ | Calculated | Found | $Tem^0C$ | Calculated | Found |
| 1 | $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 135.02 | 83.22 | 83.20 | 599.00 | 37.66 | 37.60 |
| 2 | $[Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 135.00 | 86.21 | 86.20 | 599.00 | 36.14 | 36.10 |
| 3 | $[Cd\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 130.90 | 87.14 | 87.10 | 550.60 | 33.72 | 33.60 |
| 4 | $[Cd(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 130.45 | 88.91 | 88.50 | 450.00 | 29.06 | 29.00 |
| 5 | $[Cd\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 140.50 | 88.23 | 88.20 | 530.00 | 30.86 | 30.40 |
| **6** | $[Cd\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | **137.45** | **87.07** | **87.00** | **540.00** | **33.89** | **33.30** |

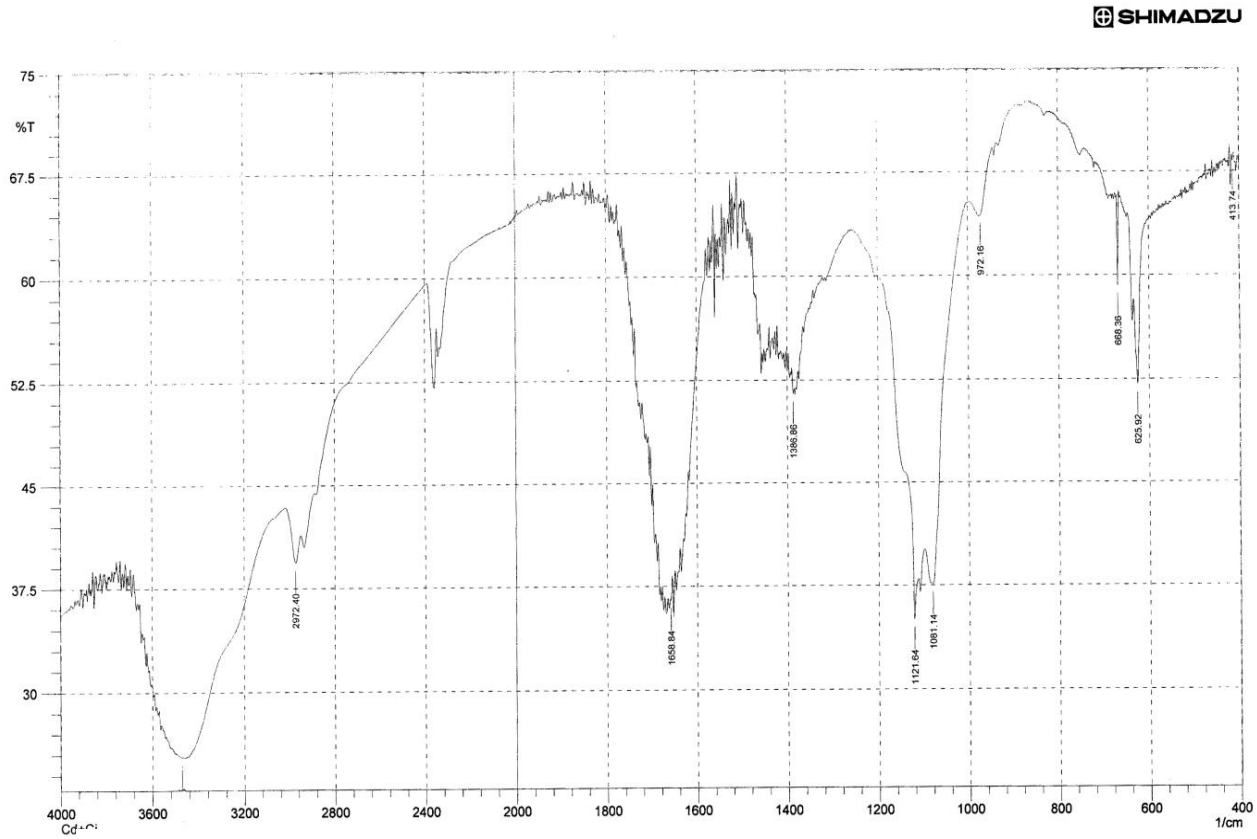



Fig-4.4.1: Infrared spectrum $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$complex 1

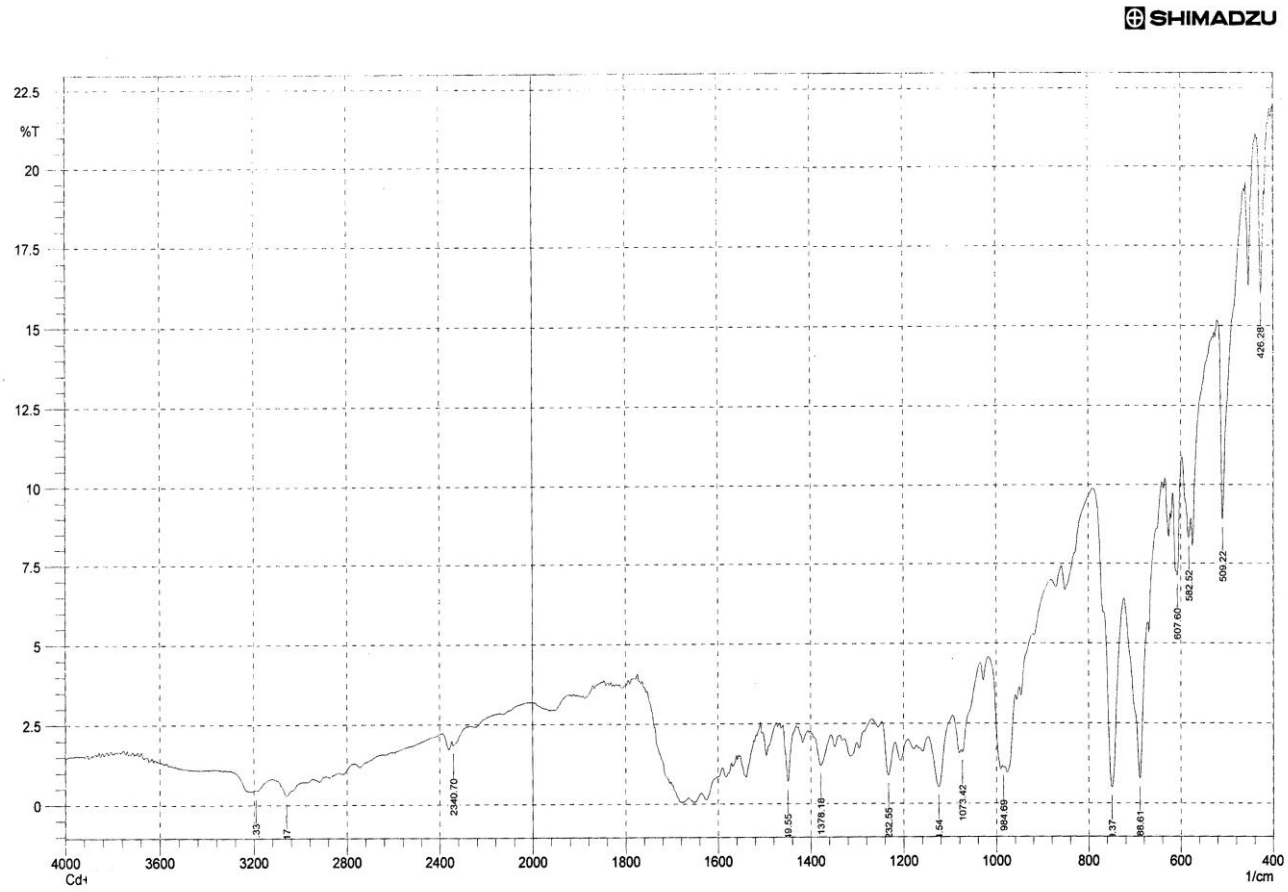
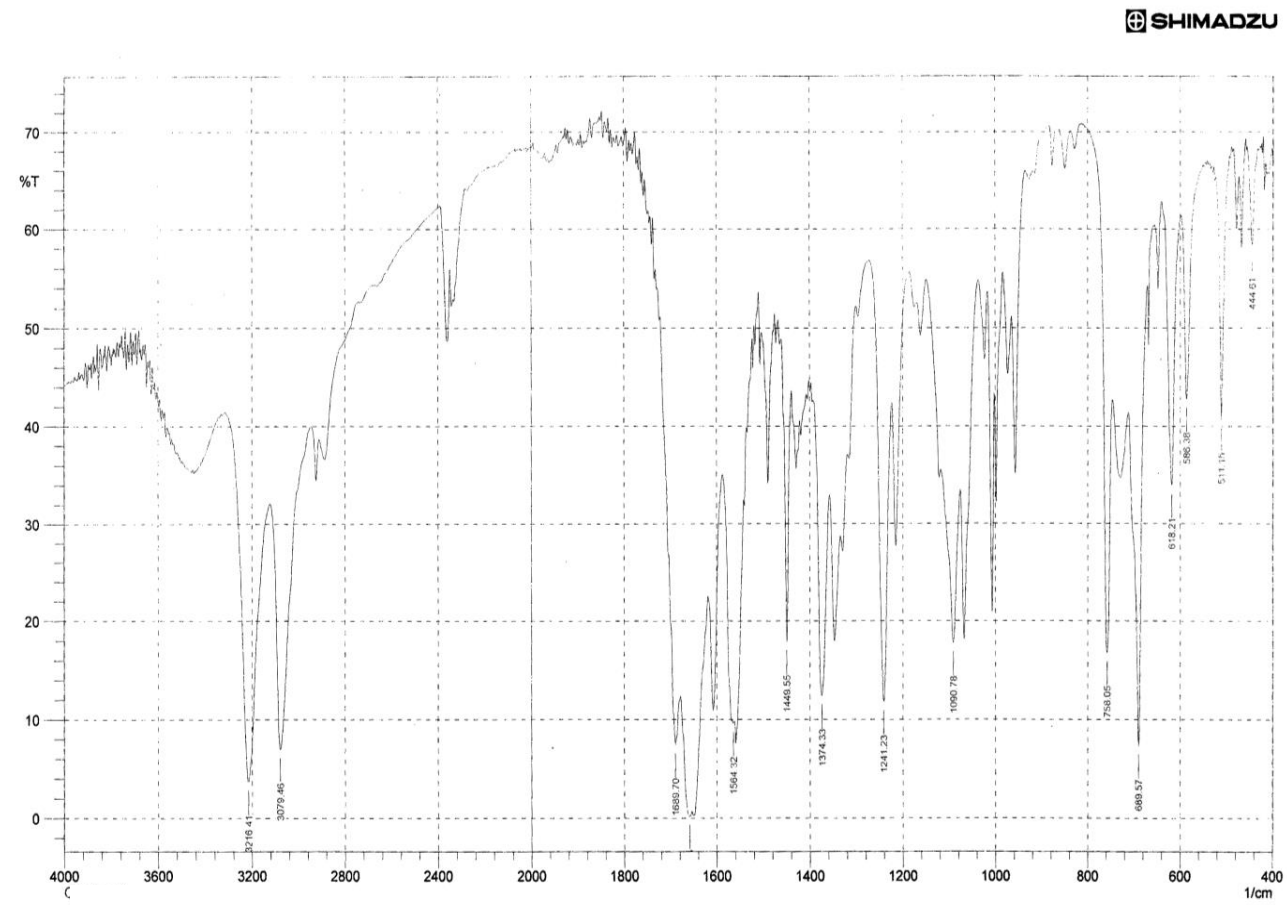
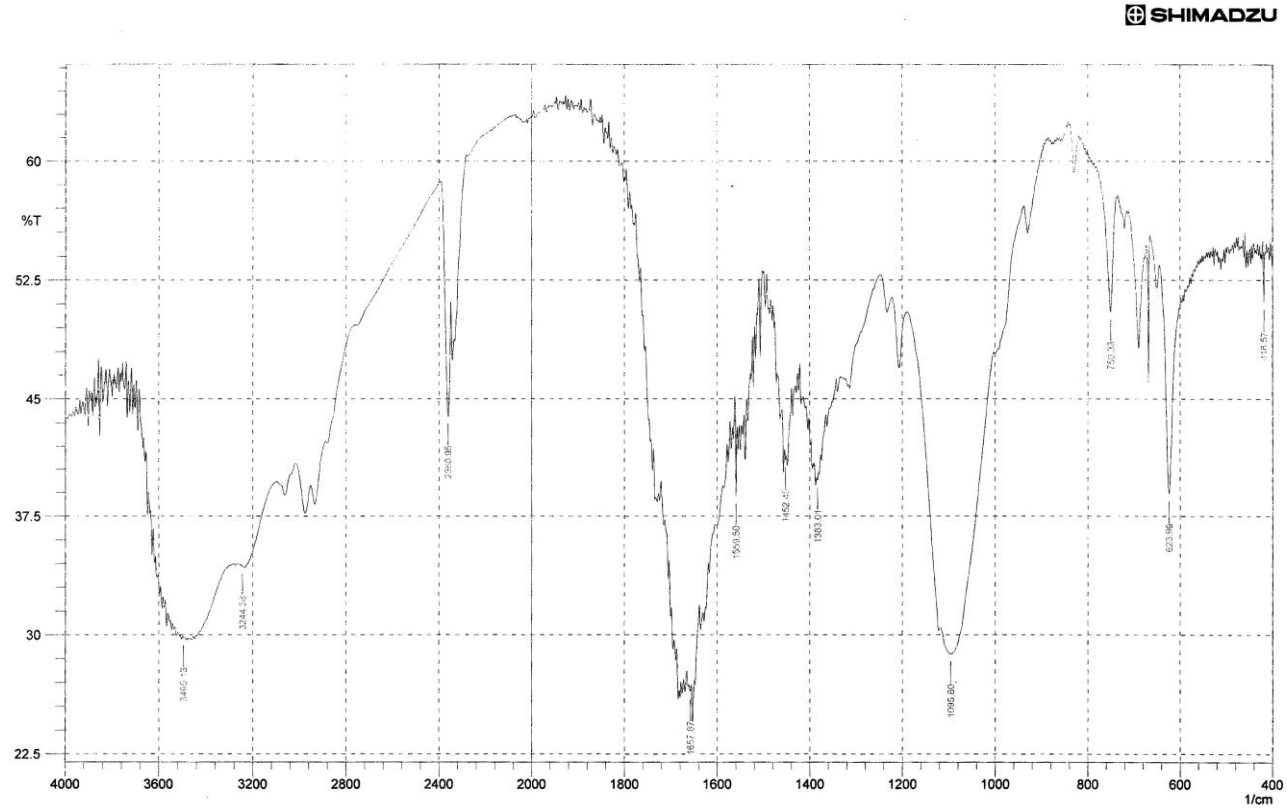



Fig-4.4.2: Infrared spectrum $[Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2




C

Fig-4.4.3:Infrared spectrum $[Cd(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3




F

Fig-4.4.4: Infrared spectrum $[Cd(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4

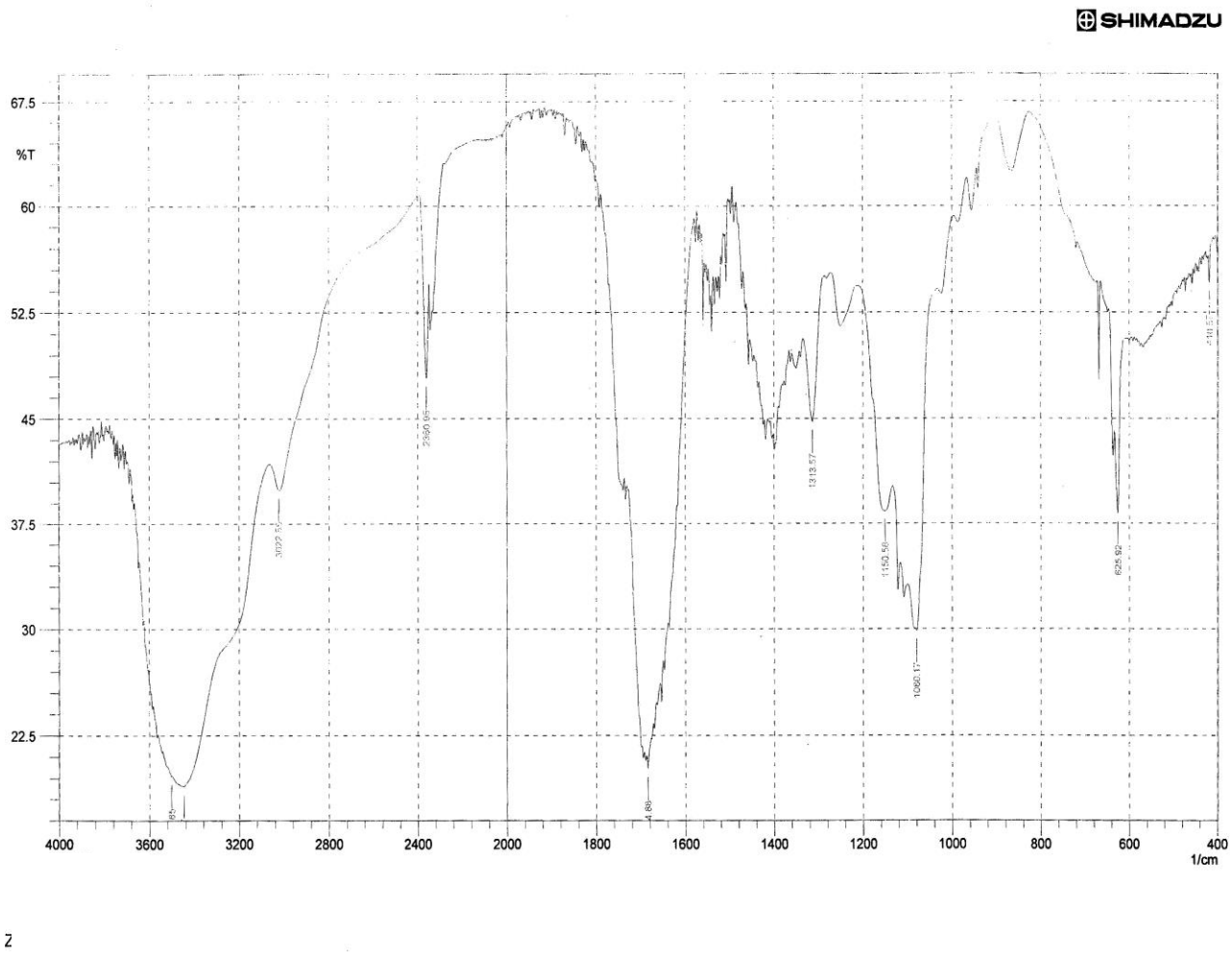



Fig-4.4.5: Infrared spectrum $[Cd(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5.

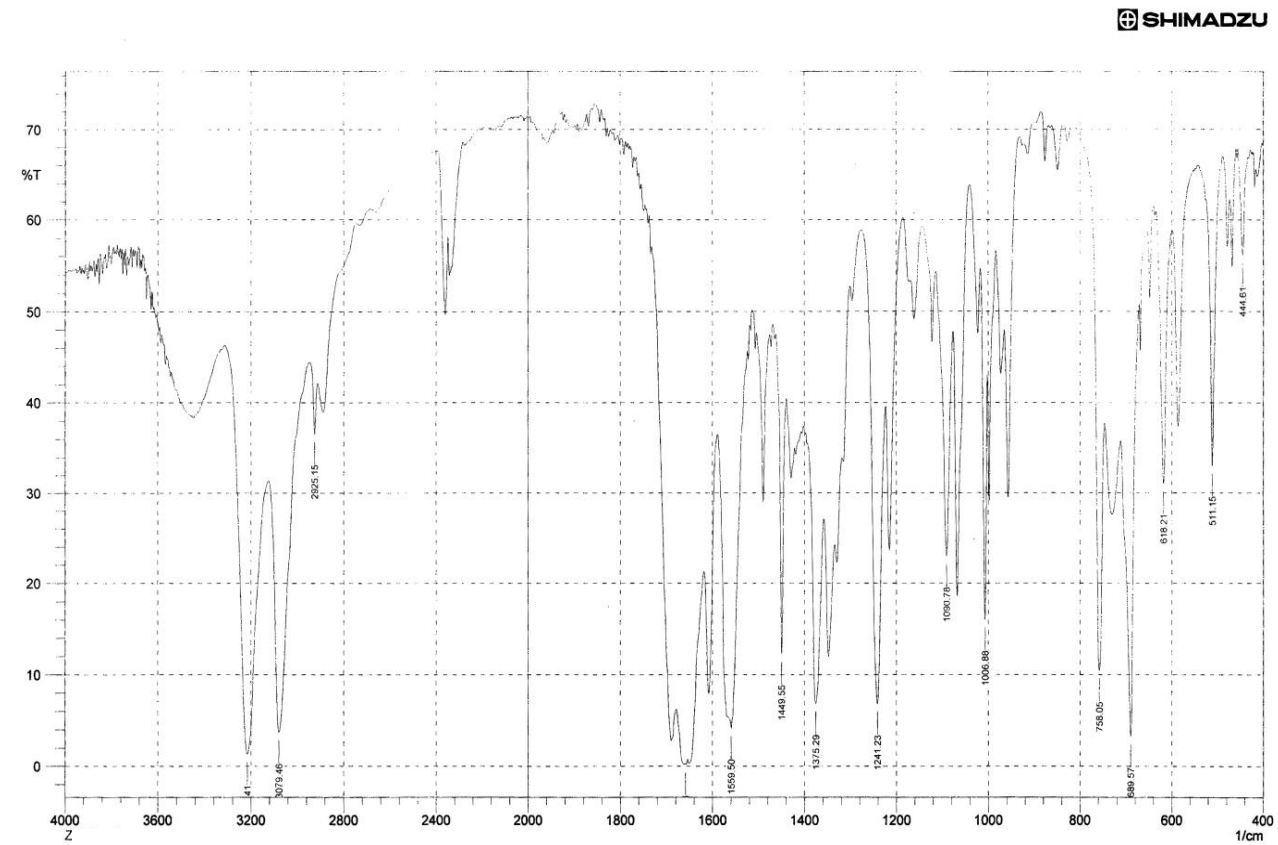



Fig-4.4.6: Infrared spectrum $[Cd(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex 6.

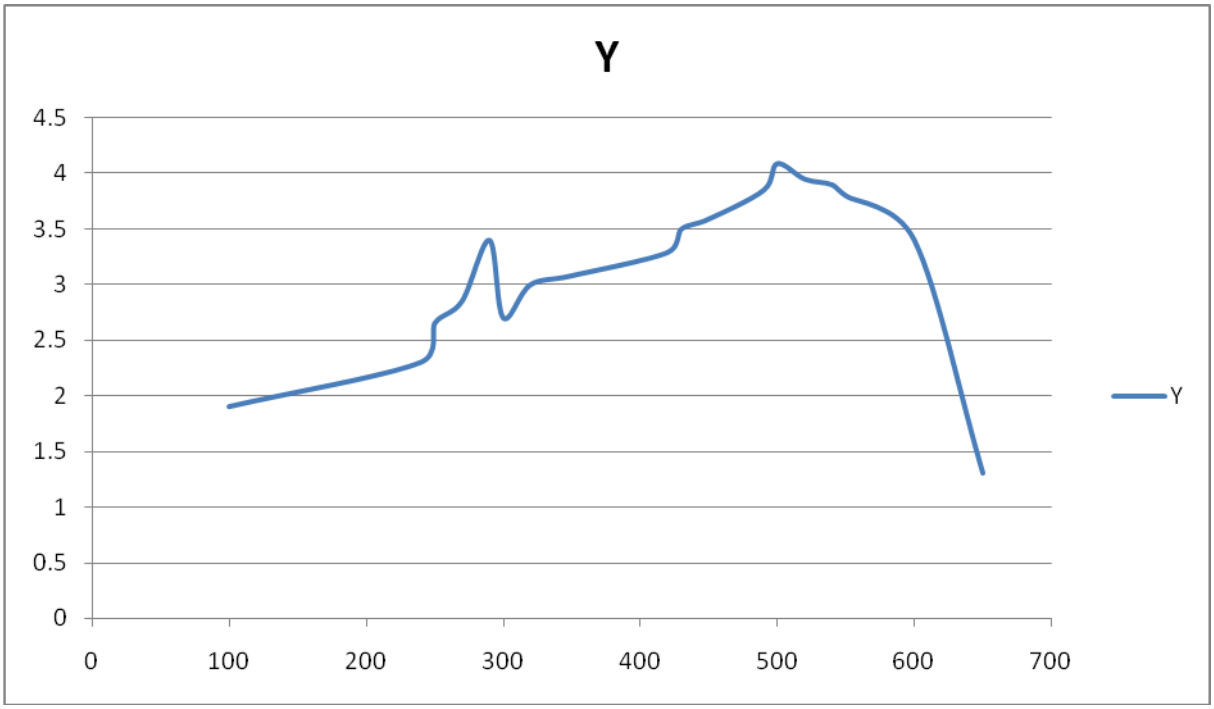



Fig-4.4.7: UV- Visible spectrum of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1

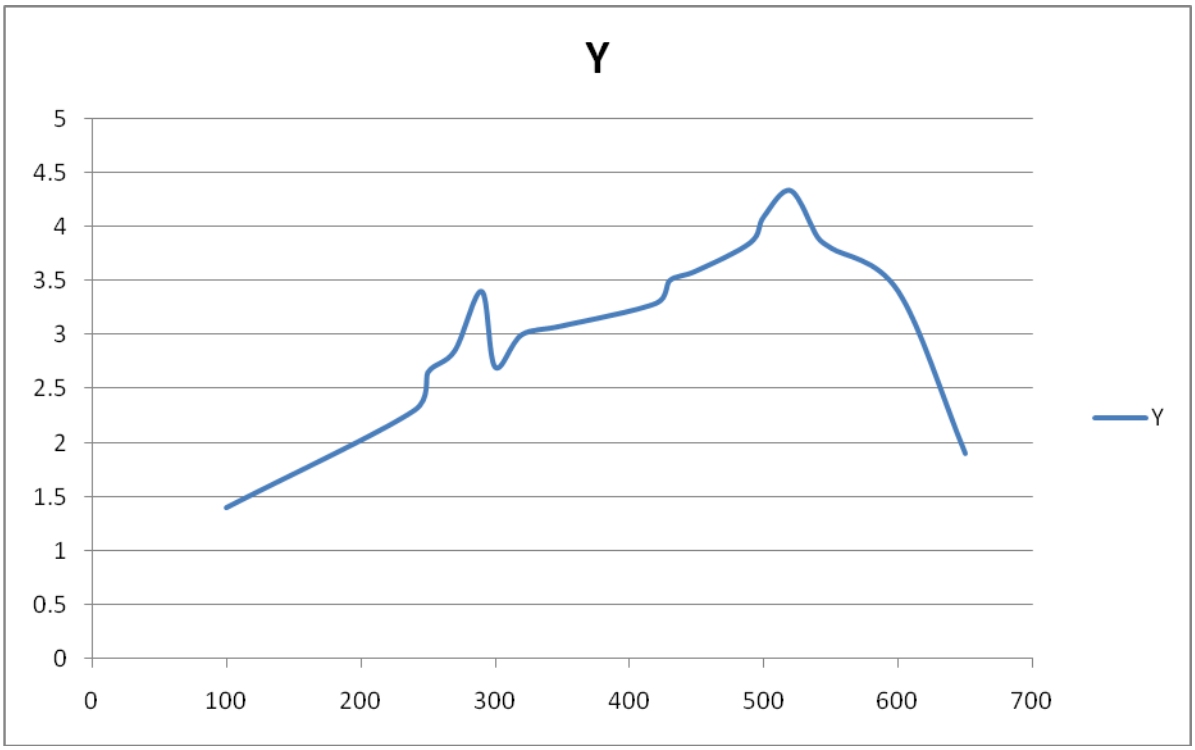



Fig-4.4.8 :UV- Visible spectrum $[Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

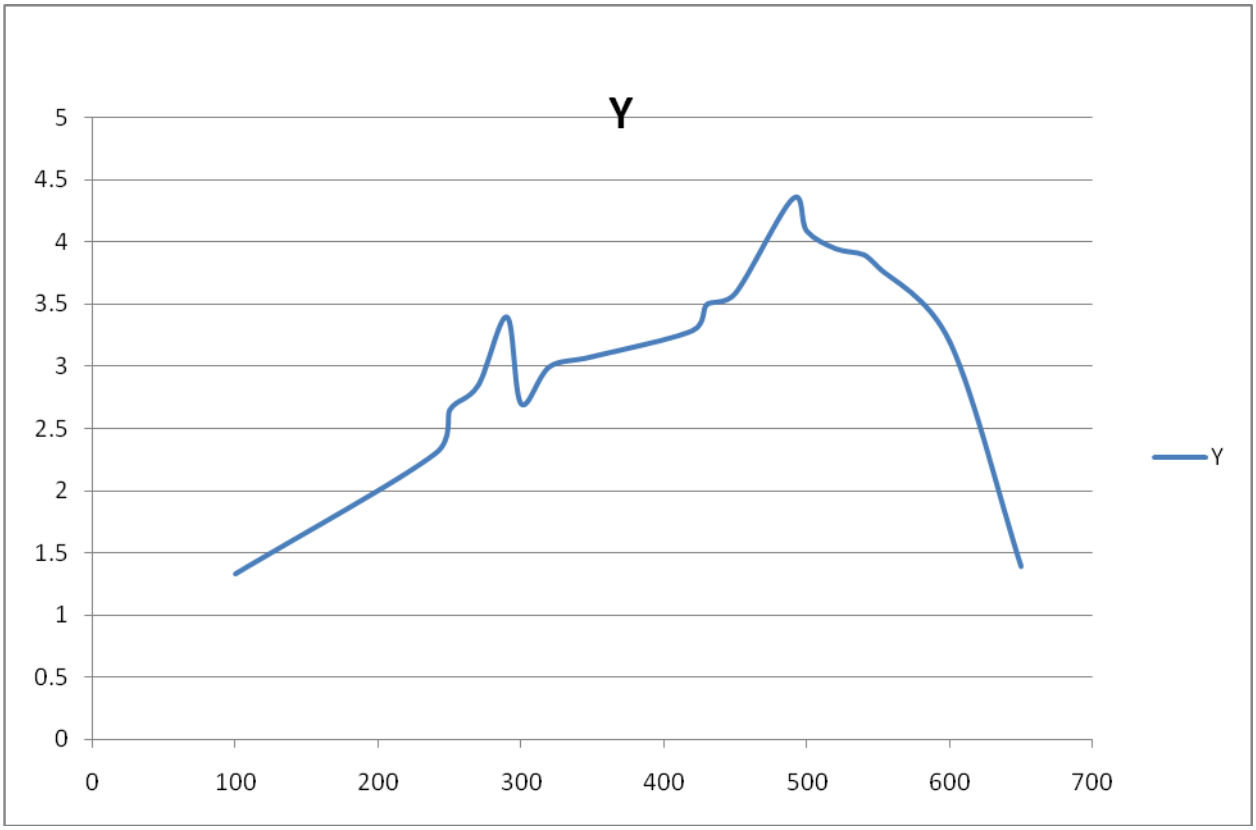



Fig-4.4.9 :UV- Visible spectrum of $[Cd(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

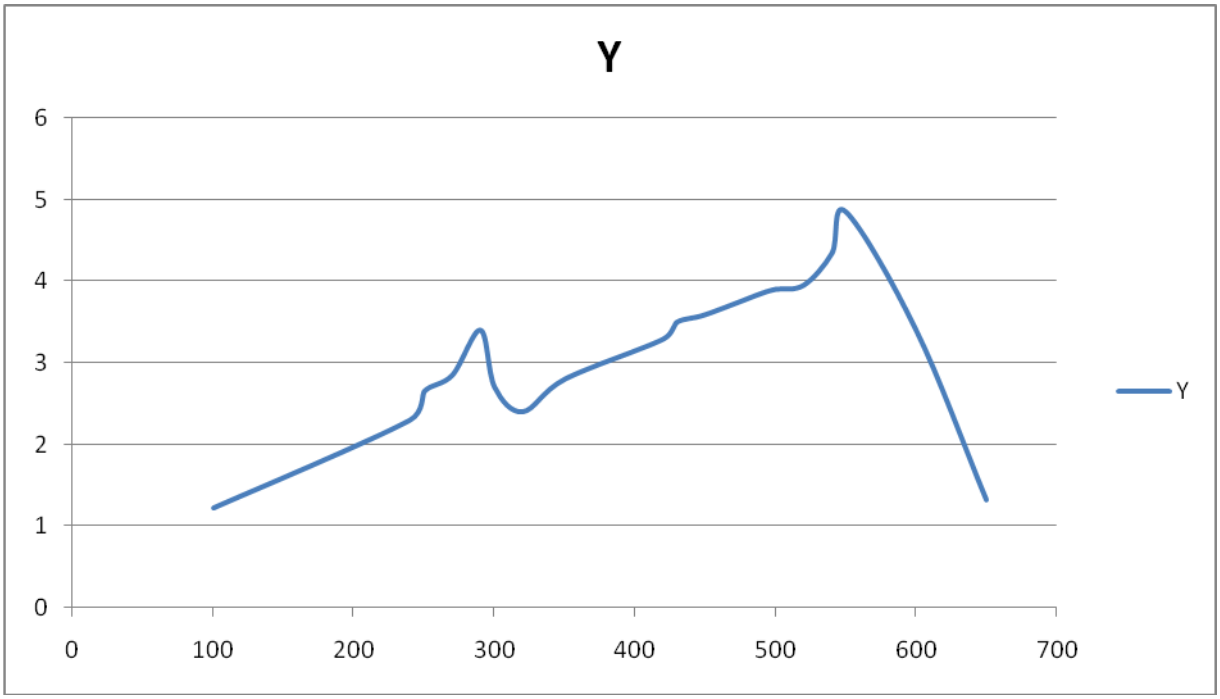



Fig-4.4.10 : UV- Visible spectrum of $[Cd(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4

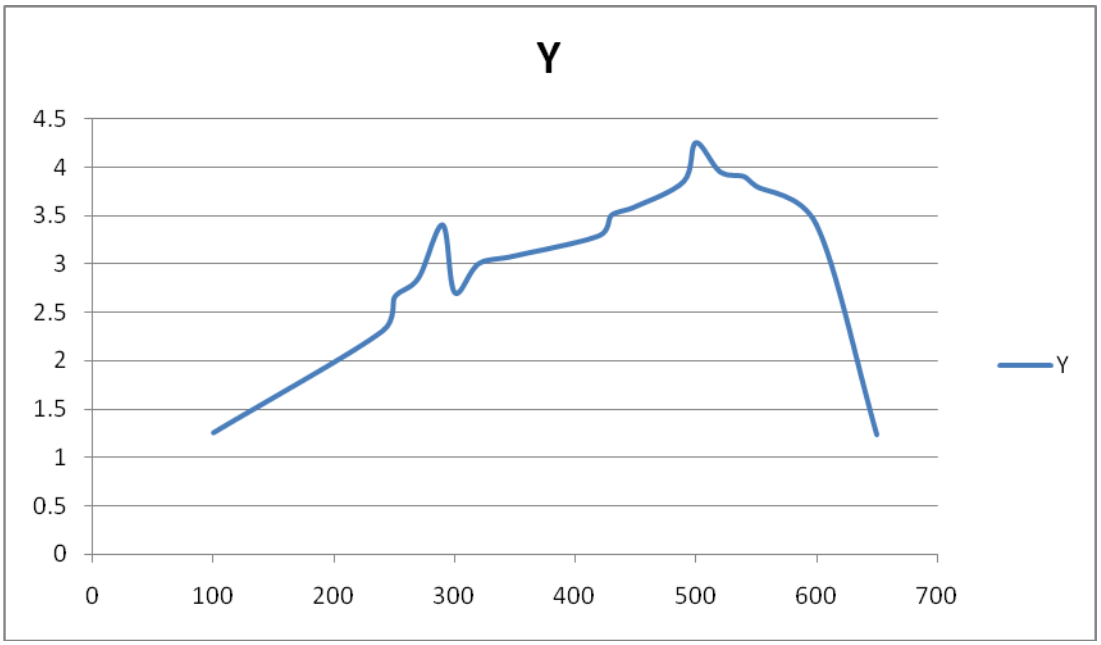



Fig-4.4.11: UV- Visible spectrum of $[Cd(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5

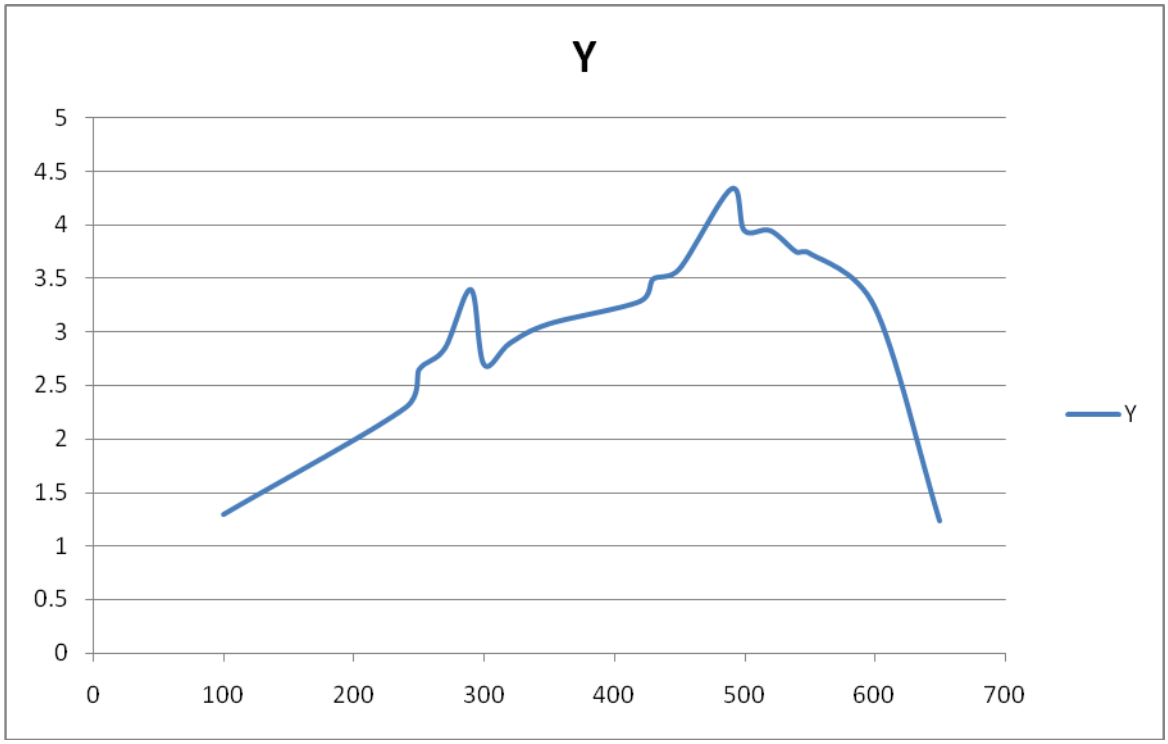



Fig-4.4.12: UV- Visible spectrum of $[Cd(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex 6.

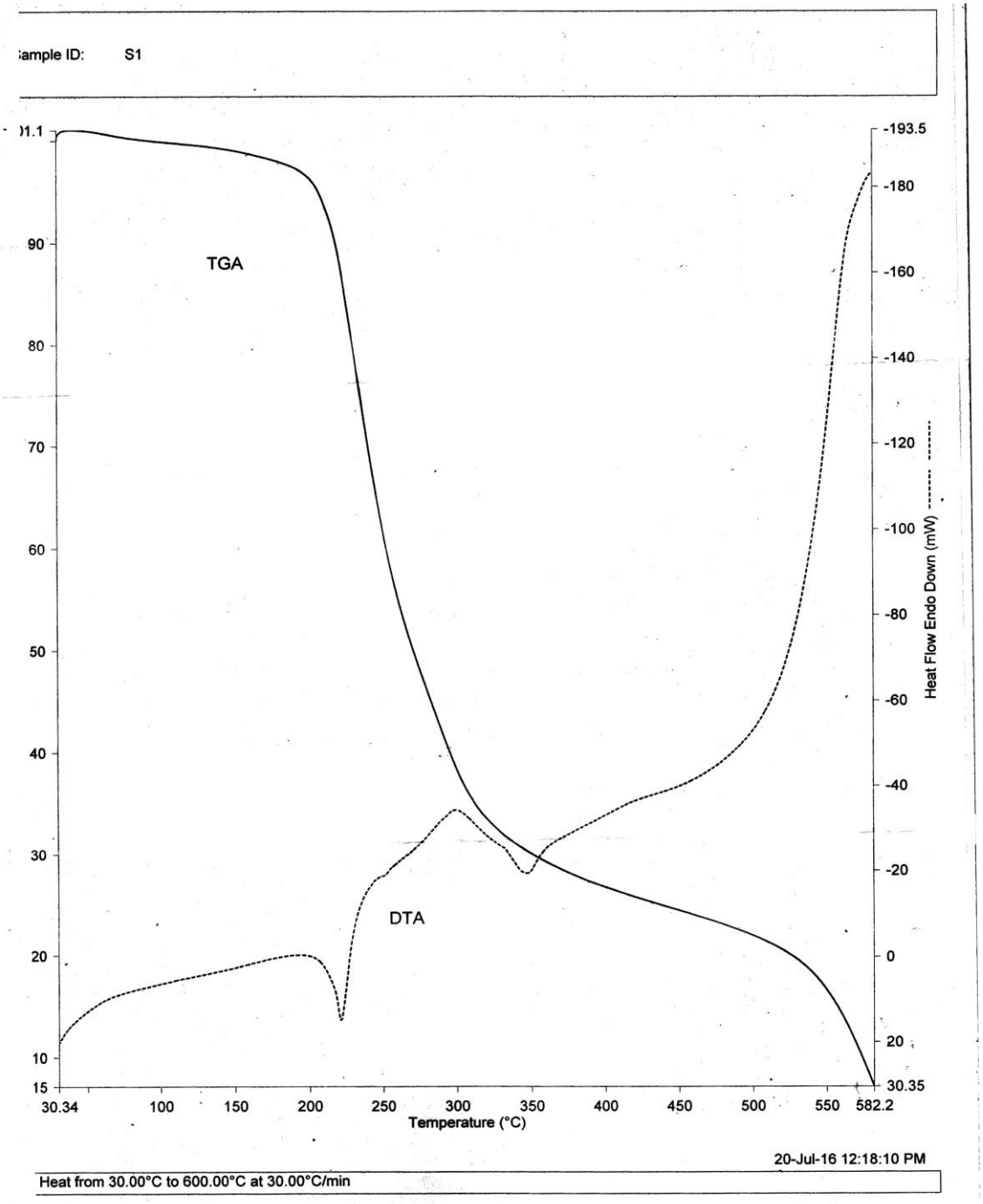



Fig-4.4.13 : TGA & DTA spectrum $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

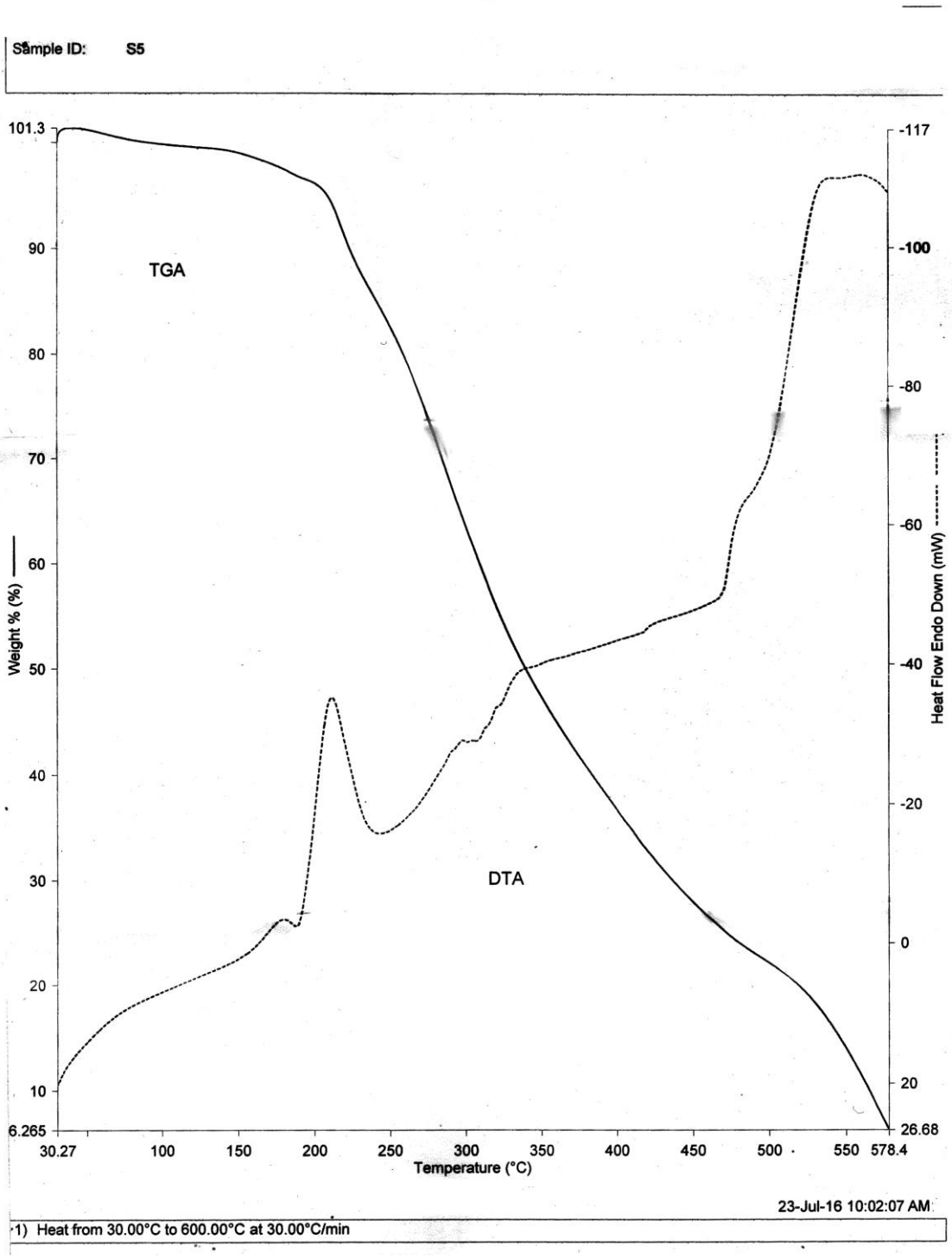



Fig-4.4.14 : TGA & DTA spectrum[$Cd(C_{10}H_{20}N_8O_4)(ClO_4)_2$] complex 2

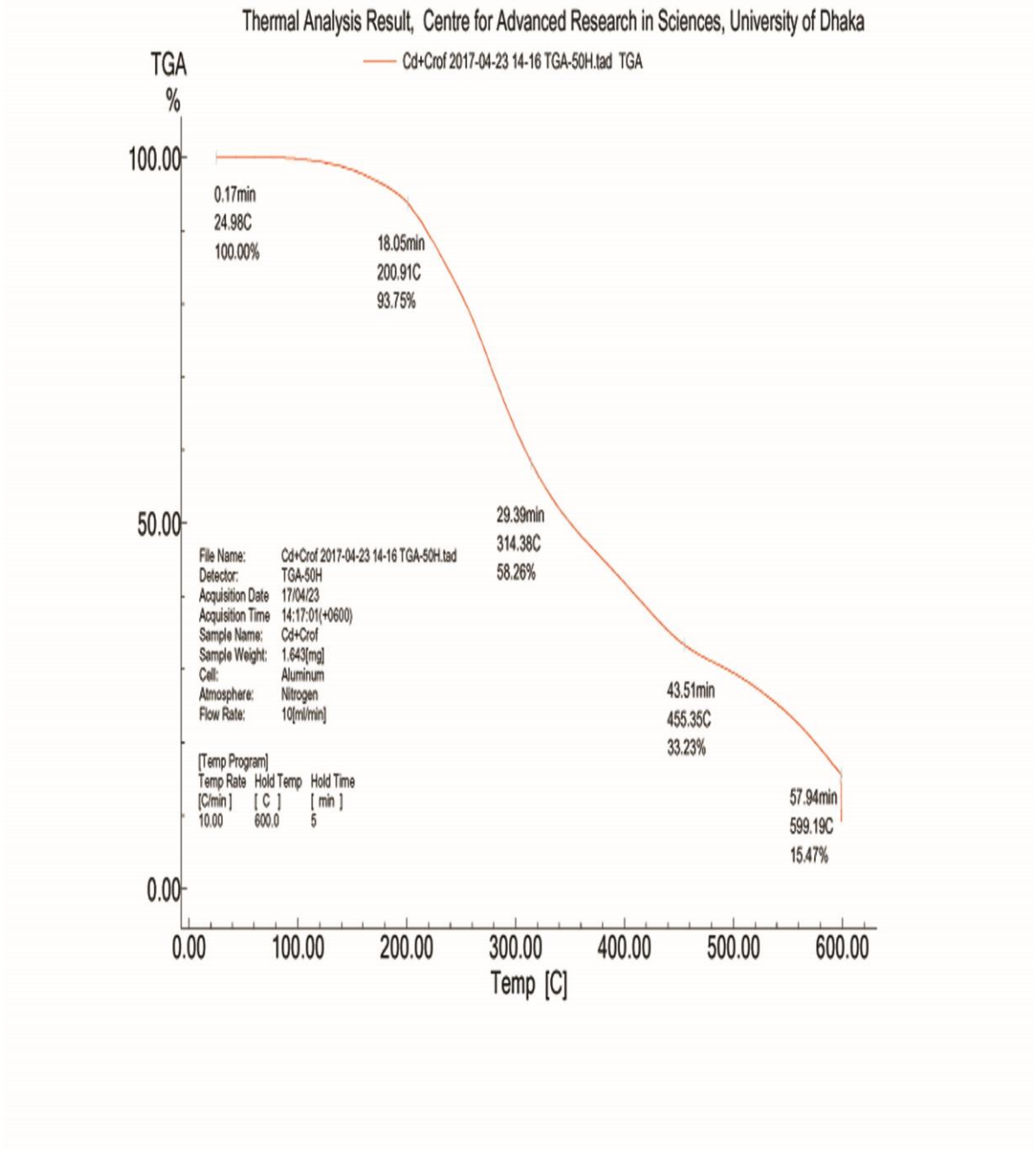



Fig-4.4.15: TGA & DTA spectrum $[Cd(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

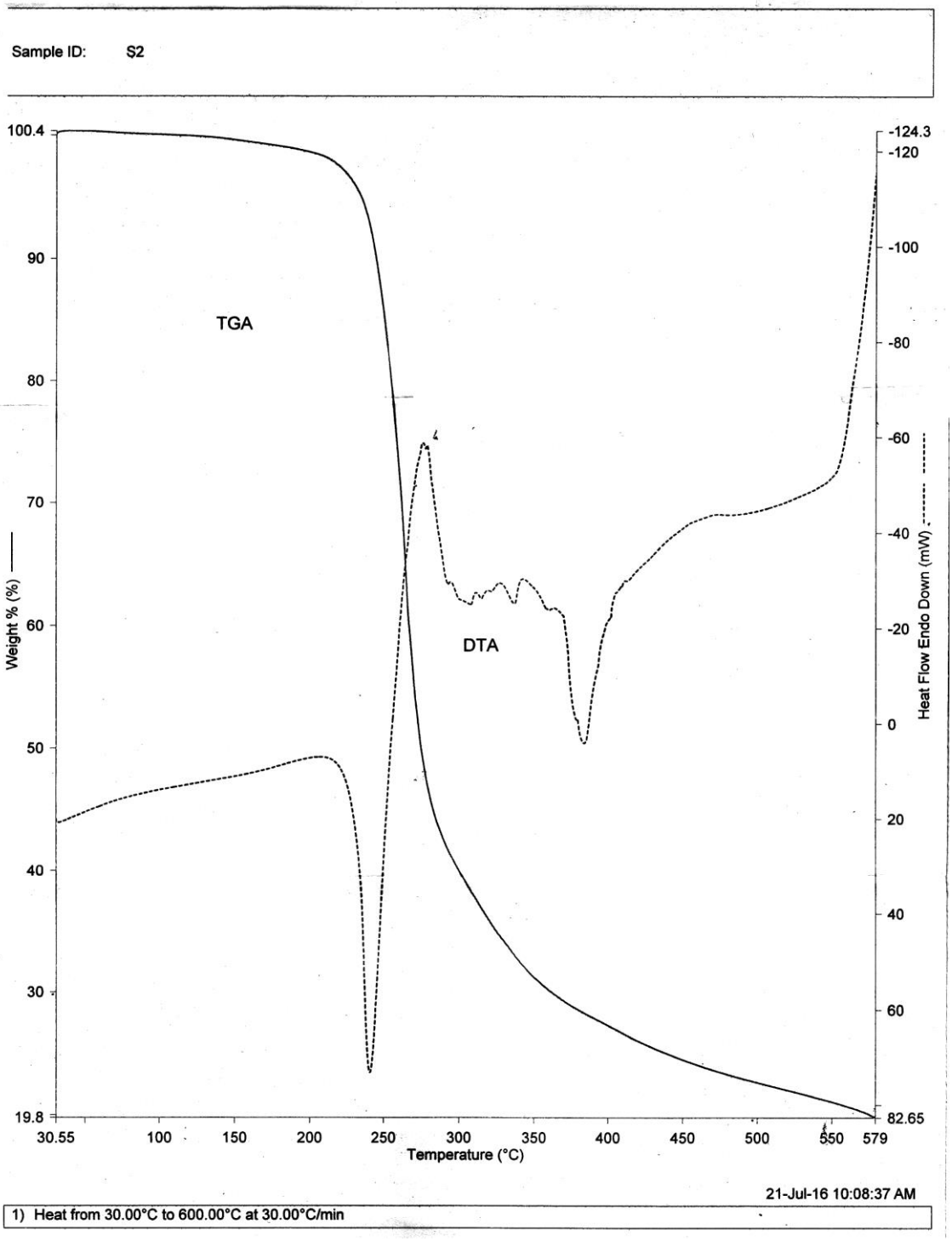



Fig-4.4.16 : TGA & DTA spectrum $[Cd(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4

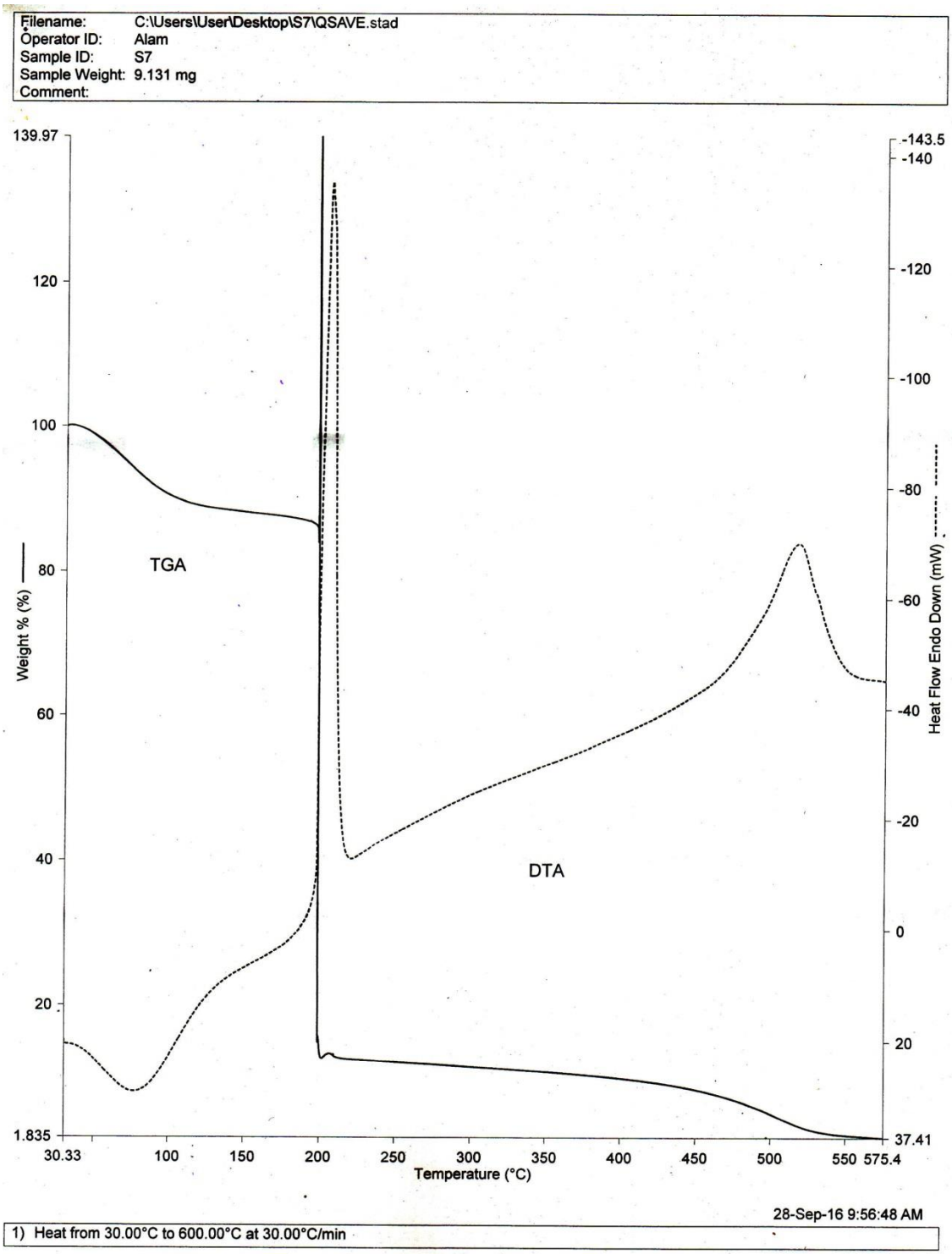



Fig-4.4.17 : TGA & DTA spectrum $[Cd(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5

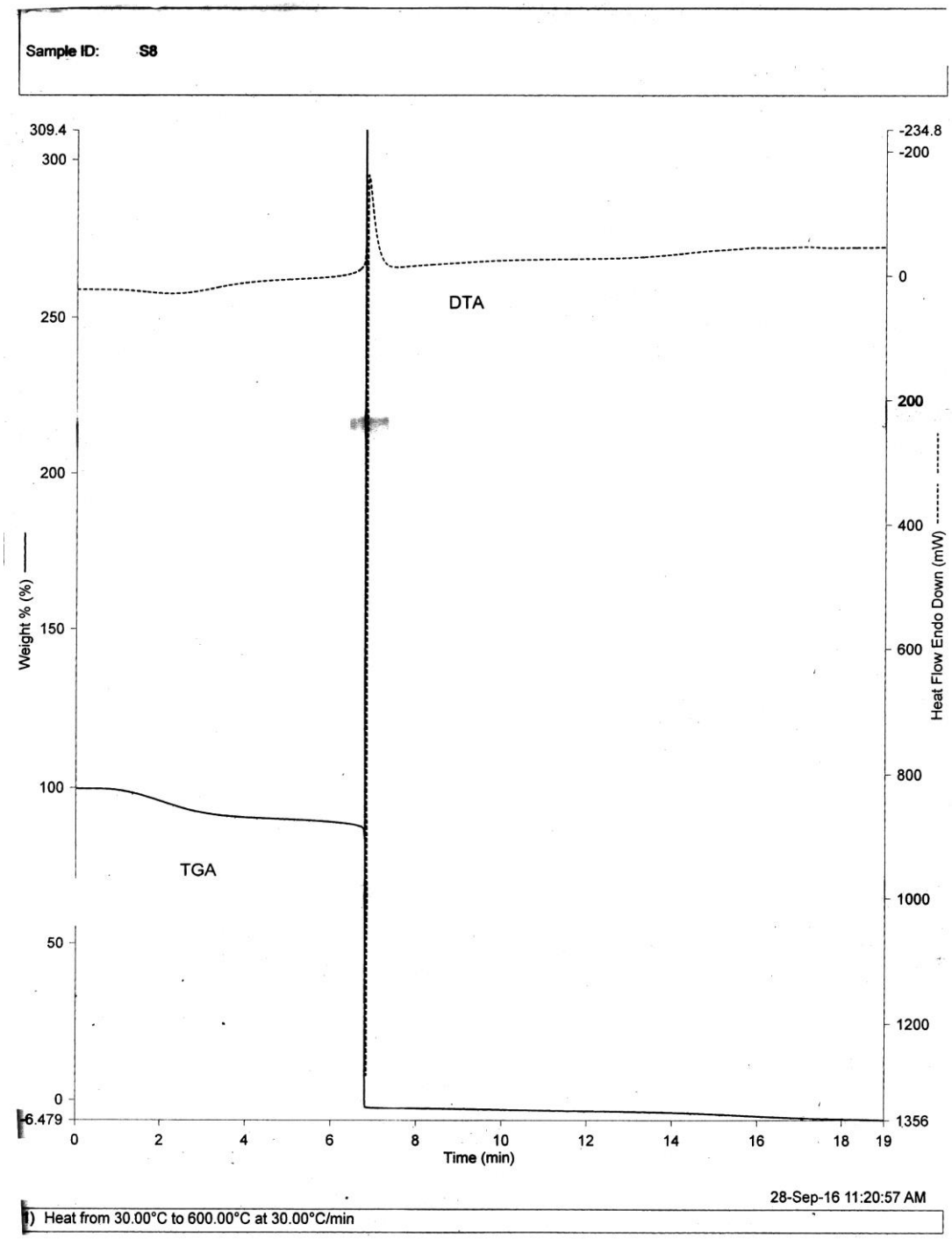


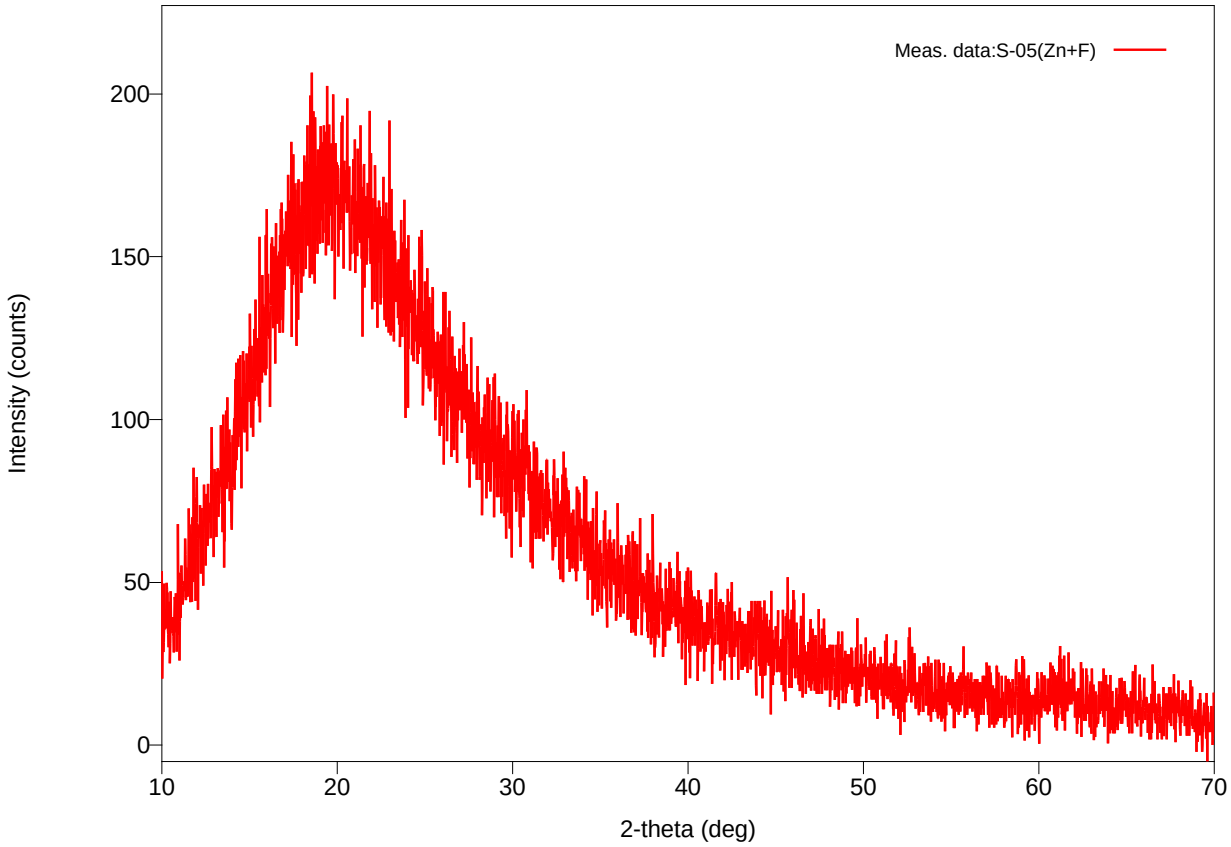

Fig-4.4.18: TGA & DTA spectrum $[Cd(C_{20}H_{24}N_8O_4)(ClO4)_2]$ complex 6.

# Peak List

## General information

| | | | |
|---|---|---|---|
| Analysis date | 2017/03/05 14:09:24 | | |
| Sample name | S-05(Cd+F) | Measurement date | 2017/03/05 13:30:31 |
| File name | S-05(Cd+F).raw | Operator | User1 |
| Comment | | | |

## Measurement profile



# RESULT AND DISCUSSION

## 4.5 MACROCYCLIC COMPLEXES OF Ag(II)

Reactions of malonodihydrazide with Ag (II) perchlorate hexahydrate in presence of formaldehyde, acetaldehyde, butanal dehyde crotonaldehyde, cinnamaldehyde, and benzaldehyde give some 16 member macrocyclic complex as described in sec.3.6

Complexes (1-6) are characterized on the basis of elemental analysis, magnetic moment & conductance measurements, UV-visible spectra & infrared studies, thermal studies and other physical properties, like melting point, solubility, colour etc.

Molar conductance data of the complexes (1-6) are shown in Table 4.5.1. The conductance values of the complexes suggested that they are electrolytic in nature[24].

The infrared spectra of the complexes (1-6) are shown as spectral data (Table 4.5.4) of the complexes showed a strong and broad band at (3238-3242) $cm^{-1}$ which is assigned for the ν(NH) stretching[25].

Due to coordination the ν(N-H) stretching of the amide group goes to the higher field at (3238, 3242) $cm^{-1}$ region as compared to the starting material malonodihydrazide[26]. In the complexes the terminal-$NH_2$ group of malonodihydrazide condensed with the aldehyde moiety form a new secondary[1]-NH group which may appear at the same region (or overlape) as

to the amide-NH group as a result the ν(N-H) band appear as a strong and broad band. [The starting material malonodihydrazide have three ν(N-H) bands at (3238, 3213, 3037) $cm^{-1}$. The bands at (3238, 3037) $cm^{-1}$ for the asymmetrie and symmetric ν(N-H) stretching of the terminal-$NH_2$ moiety and 3213 $cm^{-1}$ for amidic (N-H) group]. The complexes showed a broad band at (2920-2972) $cm^{-1}$ is suggested for the ν(C-H) stretching of aliphatic moiety[32]. The complexes showed a strong band at (1651-1680) $cm^{-1}$ which represent the ν(C=O) of NH-NH-CO-$CH_2$ moity[27]. Three or four weak band at (625-1145) $cm^{-1}$ region also indicated the $\nu_1,\nu_2,\nu_3,\nu_4$ bands of ($ClO^-_4$) moiety. These stretching frequency is suggested the not coordination of perchlorate to the metal through the O atom[28]. A medium band at (406-430) $cm^{-1}$ region is tentatively attributed to the ν(M-N) mode[29,30.] indicating the coordination of the lignad to the metal through the nitrogen atom.

The magnetic moment measurement data (Table 4.5.3) of the Ag(II) complexes (1-6) showed (1.59-1.78) B.M. These values correspond to no unpaired electrons of Ag(I) $d^{10}$system suggest the octahedral environment of the complexes which are consistent with the literature value[1]. The elemental analyses (C, H and N) (Table 4.5.2) and metal estimation data (Table 4.5.3) of the complexes are consistent with the proposed formula.

The UV-visible spectra of the complexes (1-6) are shown (Table 4.5.5) band at (330-380) nm, (1-6) at represent the d-d transition of $^4A_{2g}$ (F) $\rightarrow$ $^4T_{1g}$(F), $^4A_{2g}$(F) $\rightarrow$ $^4T_{1g}$(P), which suggested the octahedral geometry of the Ag (I) complexes[31,32].

Thermal studies: The thermal properties of metal (II) complexes were investigated by thermo grams (TGA, DTA) and are shown in (Fig 4.5.13-4.5.18) and the corresponding thermal analysis is presented in (Tabl.4.5.6). In the case of complex (1-6) the decomposition occurs in the $(230\text{-}325)^0$C range. There is no mass loss up to $230^0$C. The first stage of decomposition starts at $230^0$C and end at $230^0$C with a corresponding weight loss 25%. Which is accompanied by endothermic effect in the DTA curve in the range $225^0$C which is accompanied by weight loss confirming the second stage of decomposition is observed at $225\text{-}350^0$C (60% wt. loss).meanwhile the DTA curve exhibits endothermic effect in the range $325^0$C which is accompanied by weight loss confirming.

On the basis of elemental analysis magnetic moment and conductance measurements, thermal studies UV Visible spectra, infrared spectra and other physical properties the suggested structure of the complexes are square planer in nature as in Fig.4.5.19

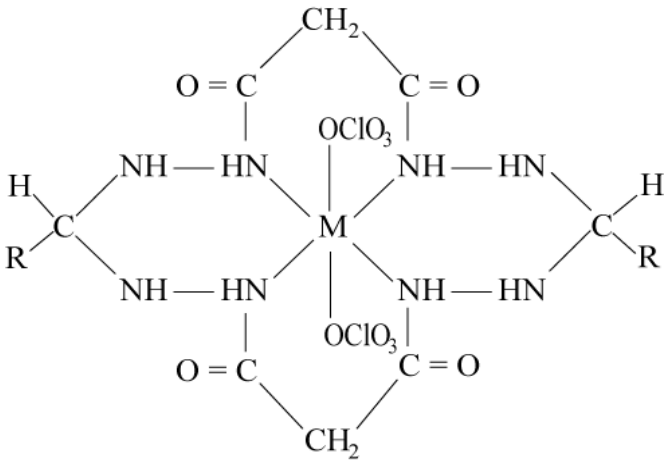



Fig. 4.5.19

M =Ag (II), where R=H(1), $CH_3$(2),$CH_3CH{=}CH$(3) $C_6H_5CH{=}CH_2$(4),$CH_3\text{-}CH_2\text{-}CH_2$(5), $C_6H_6CH$(6)

Table- 4.5.1: Analytical data and other physical properties of compounds (1-6)

| No. | Compounds | %Yield | Colour | Milting point $^0$C | % M | | Molar conductance ohm$^{-1}$cm$^2$mol$^{-1}$ |
|---|---|---|---|---|---|---|---|
| | | | | | Calculated | Found | |
| 1 | $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 80 | Ash colour | 200 | 21.80 | 21.85 | 56.30 |
| 2 | $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 75 | White | 190 | 20.63 | 20.64 | 56.03 |
| 3 | $[Ag\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 60 | Brown | 205 | 18.64 | 18.60 | 56.30 |
| 4 | $[Ag(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 70 | White | 198 | 18.77 | 18.70 | 55.90 |
| 5 | $[Ag\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 75 | Black | 195 | 15.44 | 15.40 | 56.04 |
| 6 | $[Ag\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 65 | Brown | 193 | 15.53 | 15.50 | 56.40 |

Table- 4.5.2: Elemental analysis data of compounds (1-6)

| No. | Compounds | %C | | %H | | %N | |
|---|---|---|---|---|---|---|---|
| | | Calculated | Found | Calculated | Found | Calculated | Found |
| 1 | $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 13.37 | 13.30 | 3.23 | 2.20 | 22.60 | 22.65 |
| 2 | $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 22.92 | 22.90 | 3.82 | 3.80 | 21.39 | 21.35 |
| 3 | $[Ag\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 28.99 | 128.97 | 4.83 | 4.80 | 19.32 | 19.30 |
| 4 | $[Ag(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 29.19 | 29.20 | 4.17 | 4.16 | 19.46 | 19.45 |
| 5 | $[Ag\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 41.17 | 41.15 | 4.00 | 4.01 | 16.01 | 16.02 |
| **6** | $[Ag\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | **41.40** | **41.35** | **3.45** | **3.40** | **16.10** | **16.00** |

Table- 4.5.3: Magnetic moment data of compounds (1-6)

| No. | Compounds | Sample length, *l* in cm | Weight of the sample, m in gm | Susceptibility of the empty tube, Ro | Susceptibility of the sample with tube ,R | Mass Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | Molecular weight,M | Molar Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | μeff B.M |
|---|---|---|---|---|---|---|---|---|---|
| 1 | $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0695 | -48 | -22 | 1.71 | 593 | 1.19 | 1.69 |
| 2 | $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2.1 | 0.0692 | -47 | -23 | 1.51 | 673 | 1.09 | 1.62 |
| 3 | $[Ag\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 2.2 | 0.0596 | -40 | -21 | 1.46 | 580 | 1.13 | 1.65 |
| 4 | $[Ag(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0559 | -46 | -24 | 1.47 | 756 | 1.32 | 1.78 |
| 5 | $[Ag\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 1.8 | 0.0630 | -42 | -20 | 1.31 | 494.5 | 1.10 | 1.63 |
| 6 | $[Ag\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | 1.7 | 0.0589 | -46 | -23 | 1.38 | 690.5 | 1.06 | 1.59 |

Table- 4.5.4: Important infrared spectral bands of compounds (1-6)

| No. | Compounds | $\nu$(C-H) $cm^{-1}$ | $\nu$(C=O $cm^{-1}$) | $\nu$(N-H) $cm^{-1}$ | $\nu$(M-N) $cm^{-1}$ |
|---|---|---|---|---|---|
| 1 | $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 3047 | 1664 | 3251 | 420 |
| 2 | $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 2920 | 1649 | 3282 | 430 |
| 3 | $[Ag\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 2960 | 1640 | 3250 | 416 |
| 4 | $[Ag(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 2967 | 1650 | 3261 | 408 |
| 5 | $[Ag\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 3040 | 1660 | 3254 | 409 |
| **6** | $[Ag\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ | **3070** | **1680** | **3270** | **406** |

Table- 4.5.5: U.V- Visible Adsorption Maxima Of Compounds (1-6)

| No. | Compounds | λ max (n,m) |
|---|---|---|
| **1** | $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 294,493 |
| **2** | $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 292,490 |
| **3** | $[Ag\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 290,490 |
| **4** | $[Ag(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 300,495 |
| **5** | $[Ag\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 290,490 |
| **6** | $[Ag\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 290,540 |

Table- 4.5.6: Thermal analysis data of compounds (1-6)

| No. | Compounds | %M (Ligand) | | | %M Metal oxide($Ag_2O$) | | |
|---|---|---|---|---|---|---|---|
| | | $Tem^0C$ | Calculated | Found | $Tem^0C$ | Calculated | Found |
| 1 | $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ | 225.00 | 83.22 | 83.20 | 550.00 | 37.66 | 37.60 |
| 2 | $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | 225.00 | 86.21 | 86.20 | 500.00 | 36.14 | 36.10 |
| 3 | $[Ag\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | 255.0 | 87.14 | 87.10 | 550.60 | 33.72 | 33.60 |
| 4 | $[Ag(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | 245.00 | 88.91 | 88.50 | 450.00 | 29.06 | 29.00 |
| 5 | $[Ag\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | 180.00 | 88.23 | 88.20 | 450.00 | 30.86 | 30.40 |
| 6 | $[Ag\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | 230.00 | 87.07 | 87.00 | 480.00 | 33.89 | 33.30 |

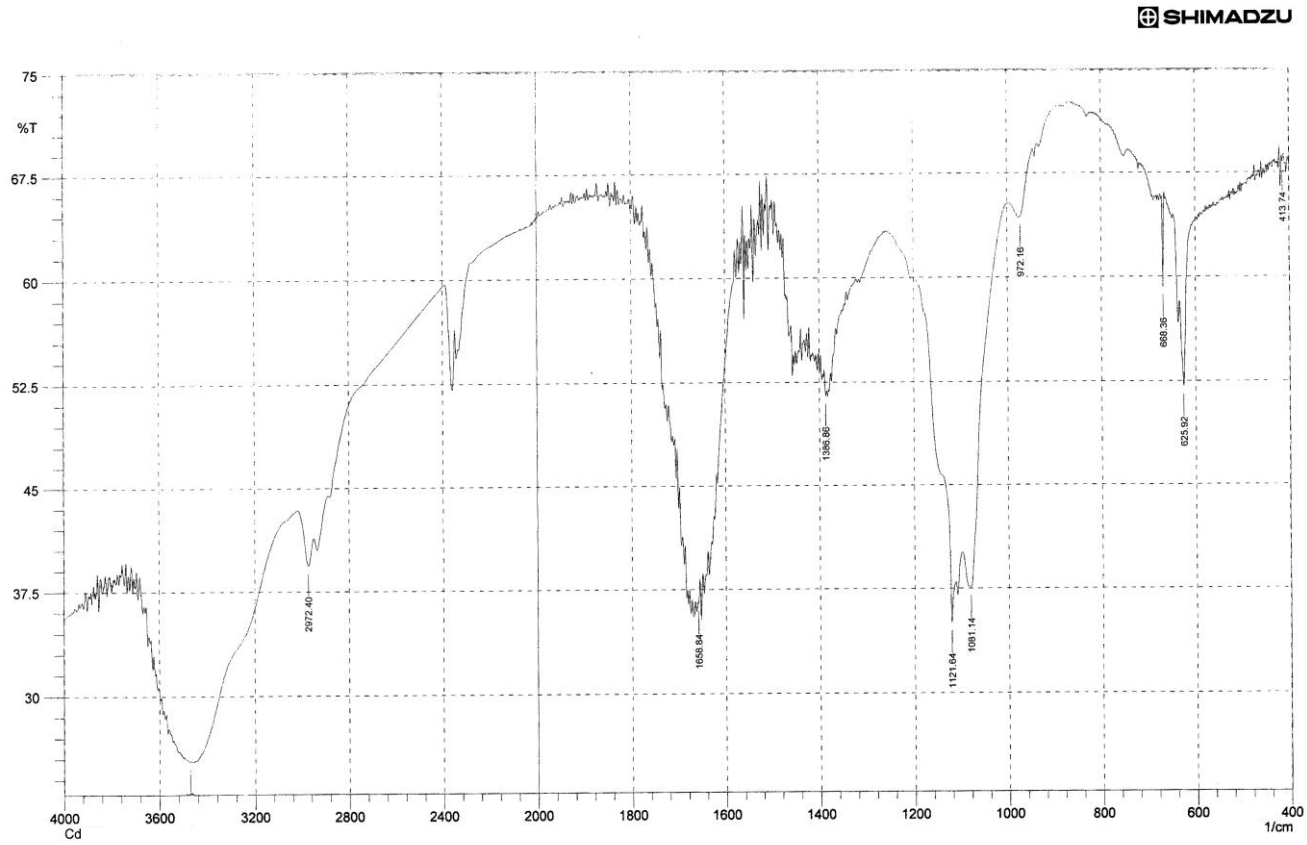
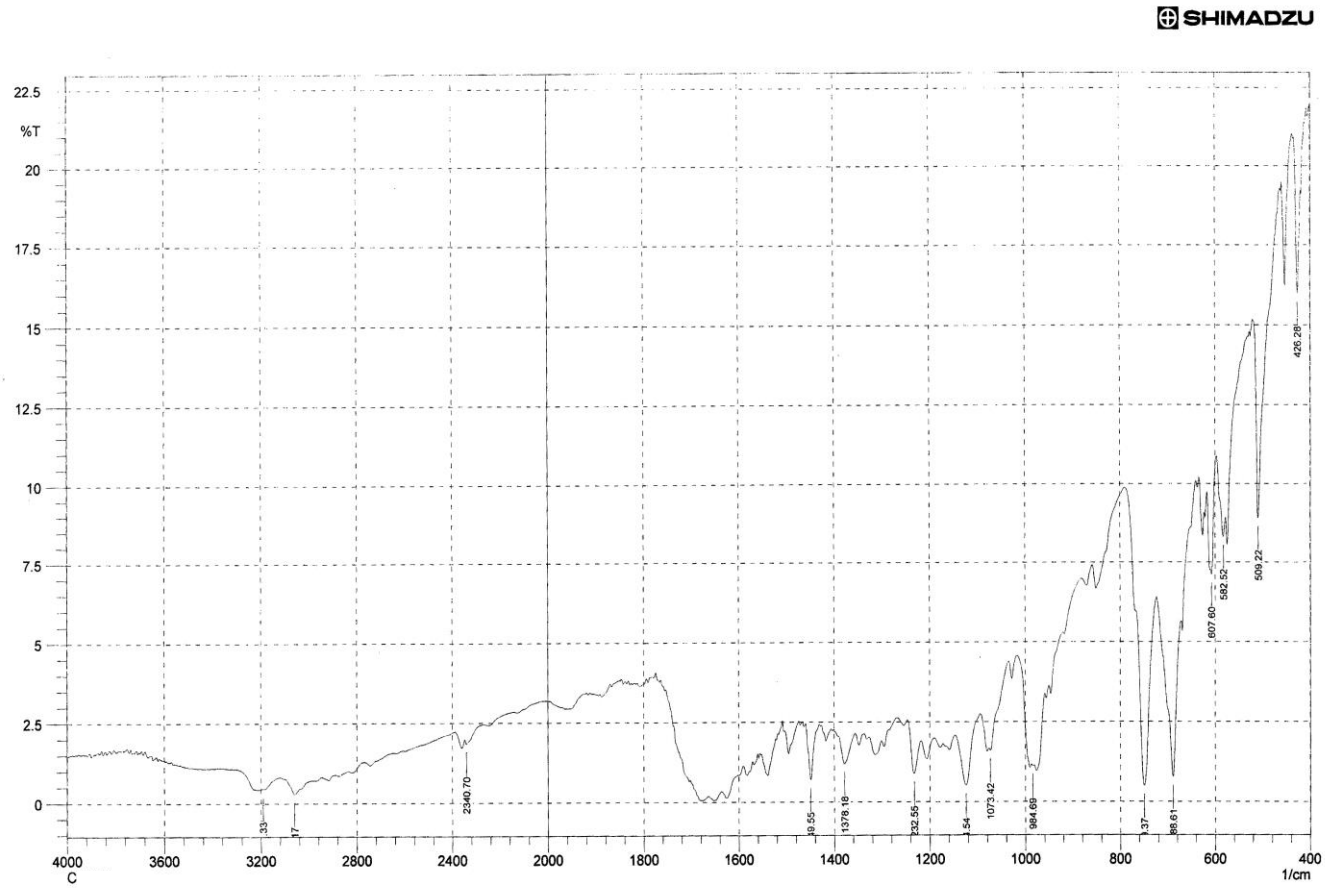



Fig-4.5.1: Infrared spectrum $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1




C

Fig-4.5.2: Infrared spectrum $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

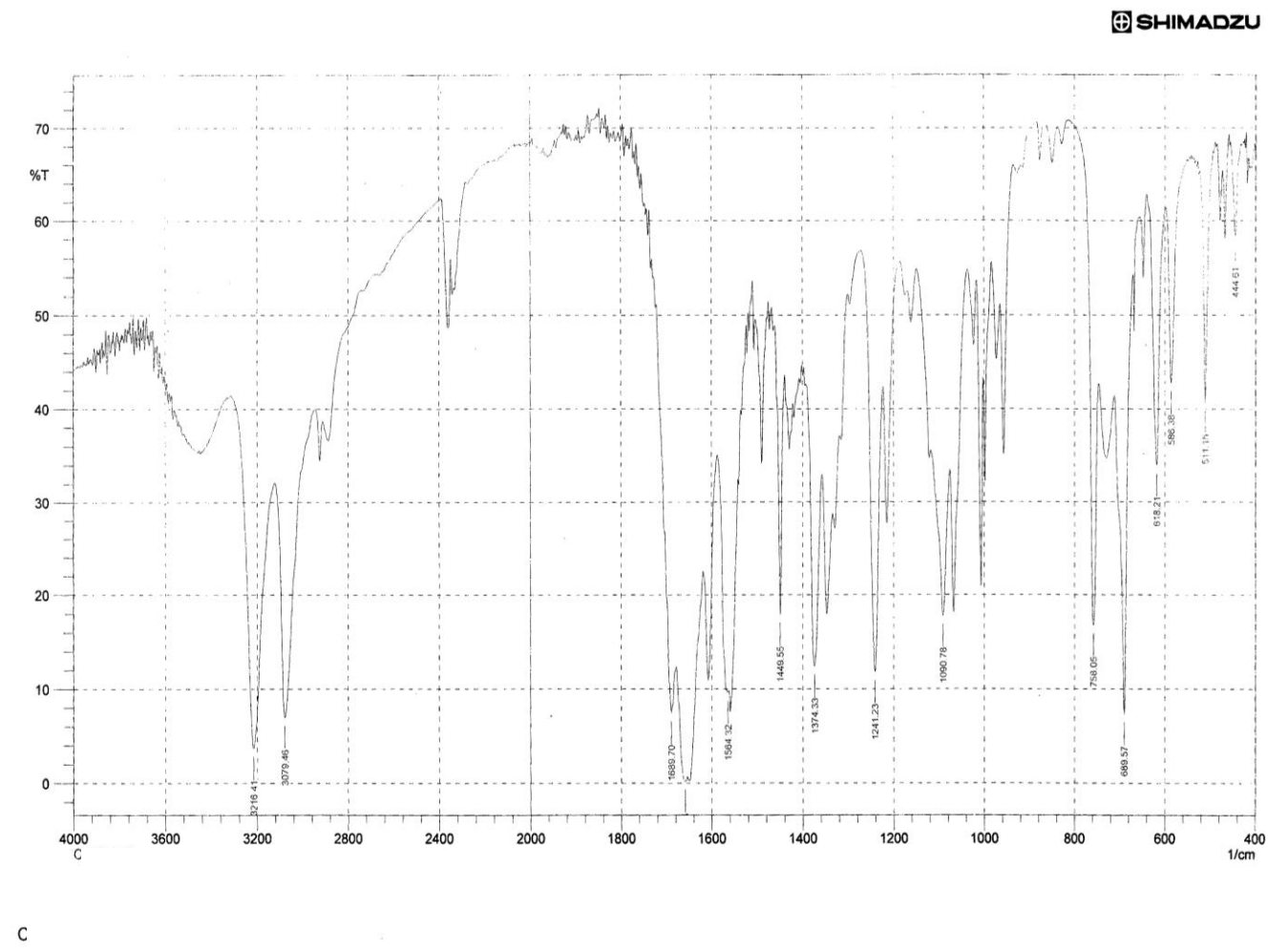



Fig-4.5.3: Infrared spectrum $[Ag(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

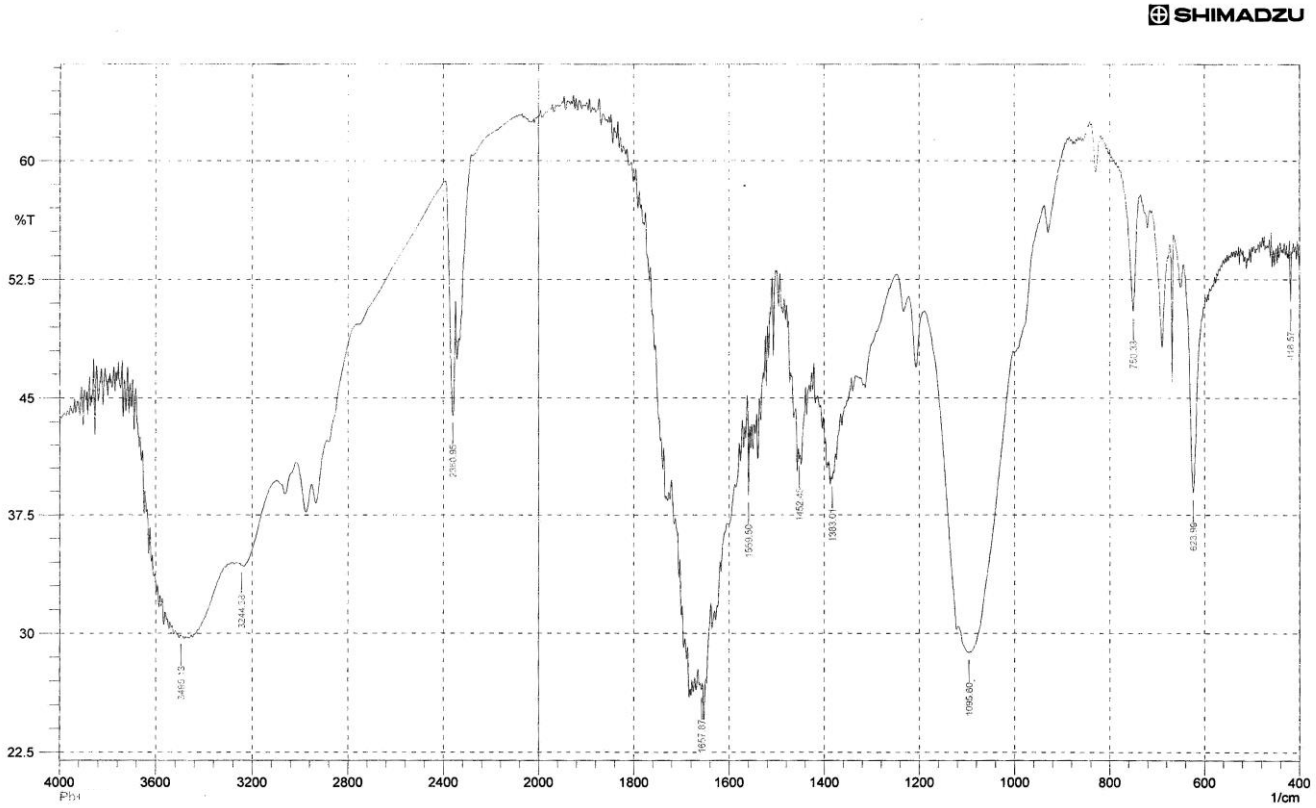



Fig-4.5.4: Infrared spectrum $[Ag(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4.

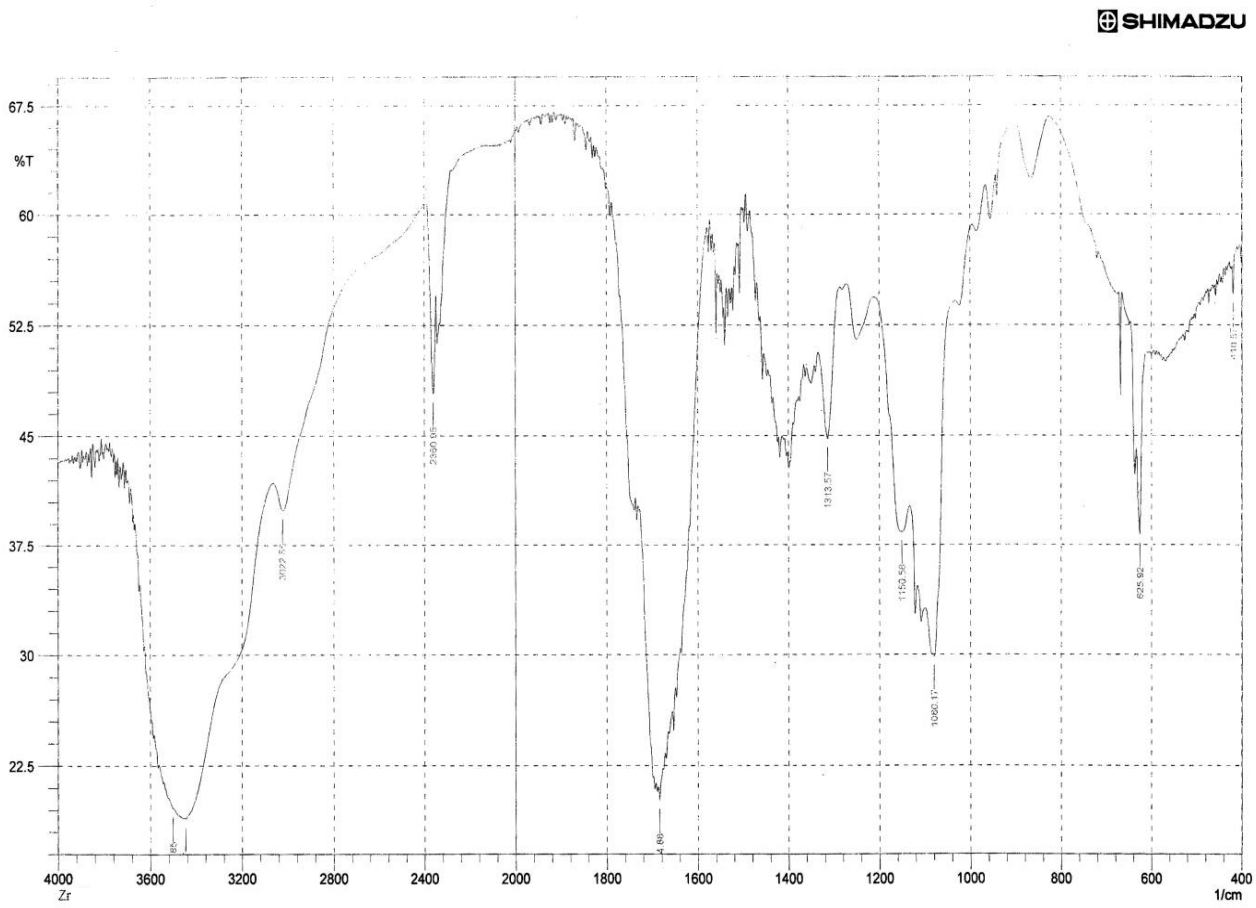



Fig-4.5.5: Infrared spectrum $[Ag(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5.

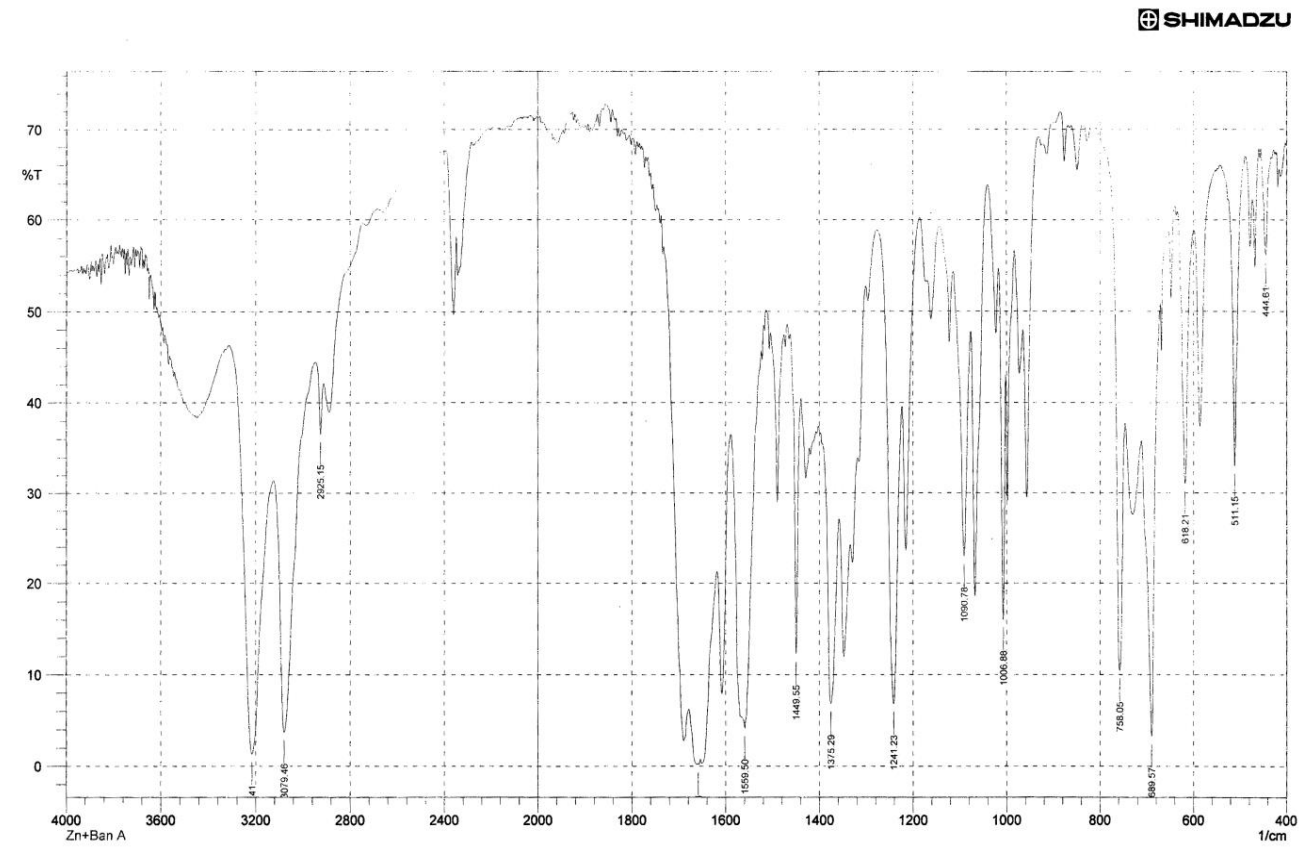



Fig-4.5.6: Infrared spectrum $[Ag(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex 6.

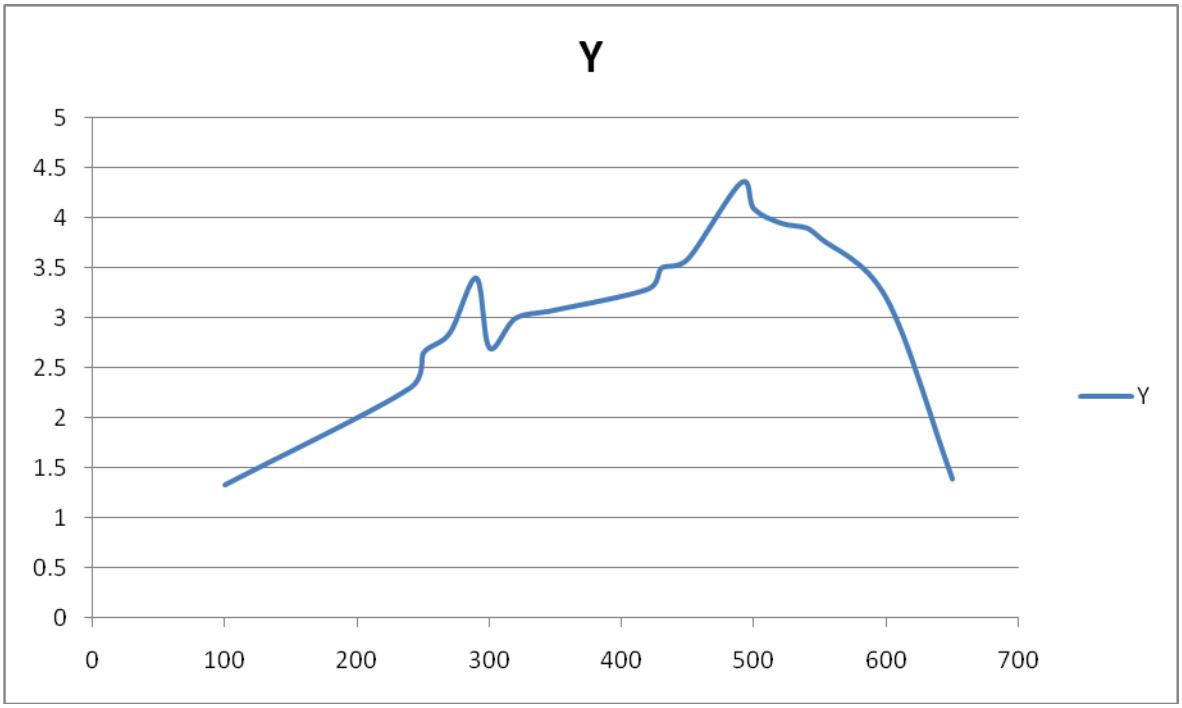



Fig-4.5.7: UV-Visible spectrum of [Ag $(C_8H_{16}N_8O_4)(ClO_4)_2$] complex 1.

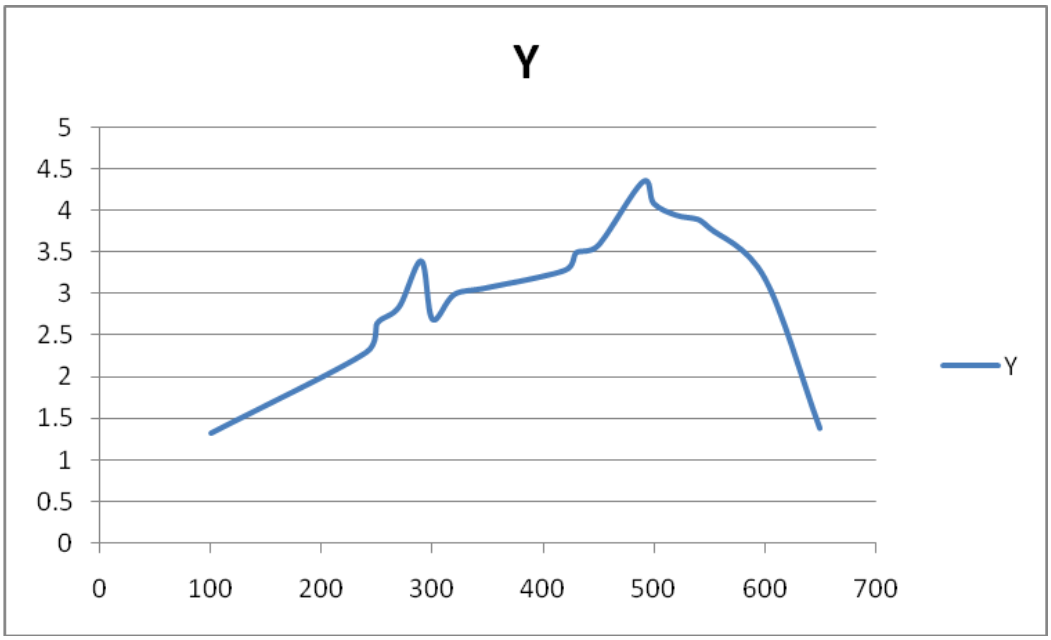



Fig-4.5.8: UV-Visible spectrum of $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

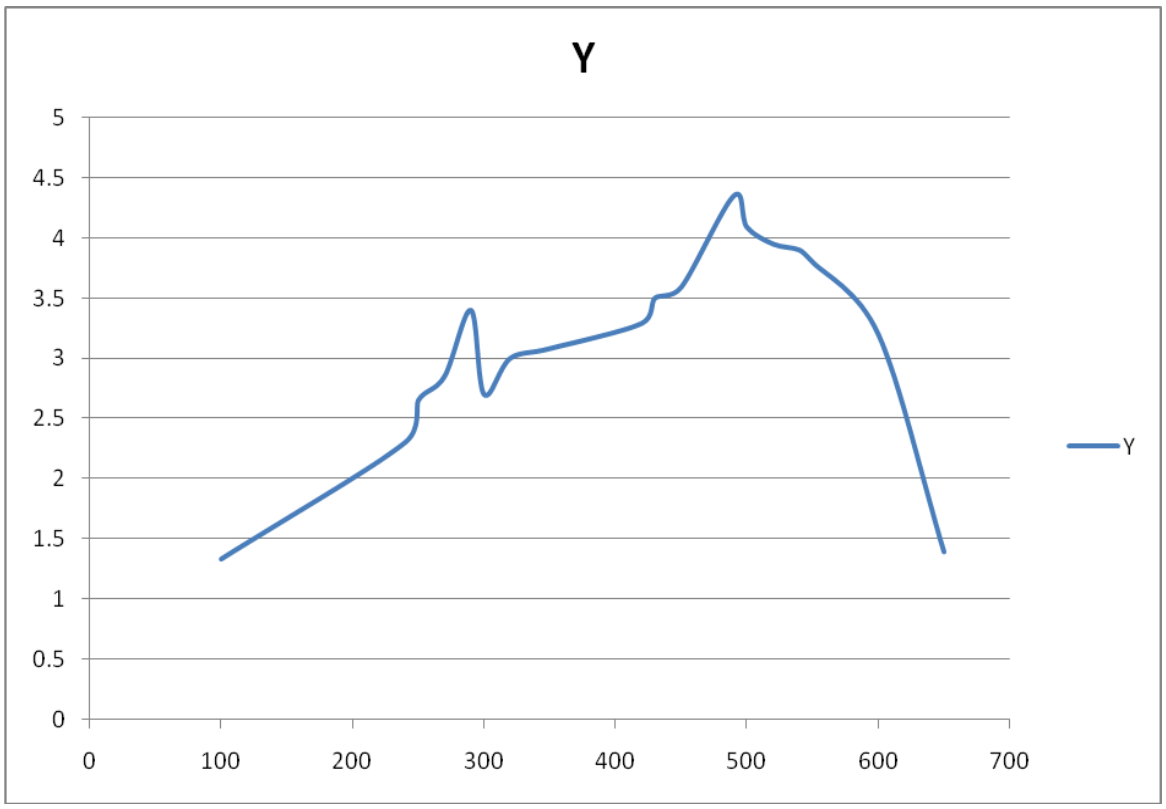



Fig-4.5.9: UV- Visible spectrum of $[Ag(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

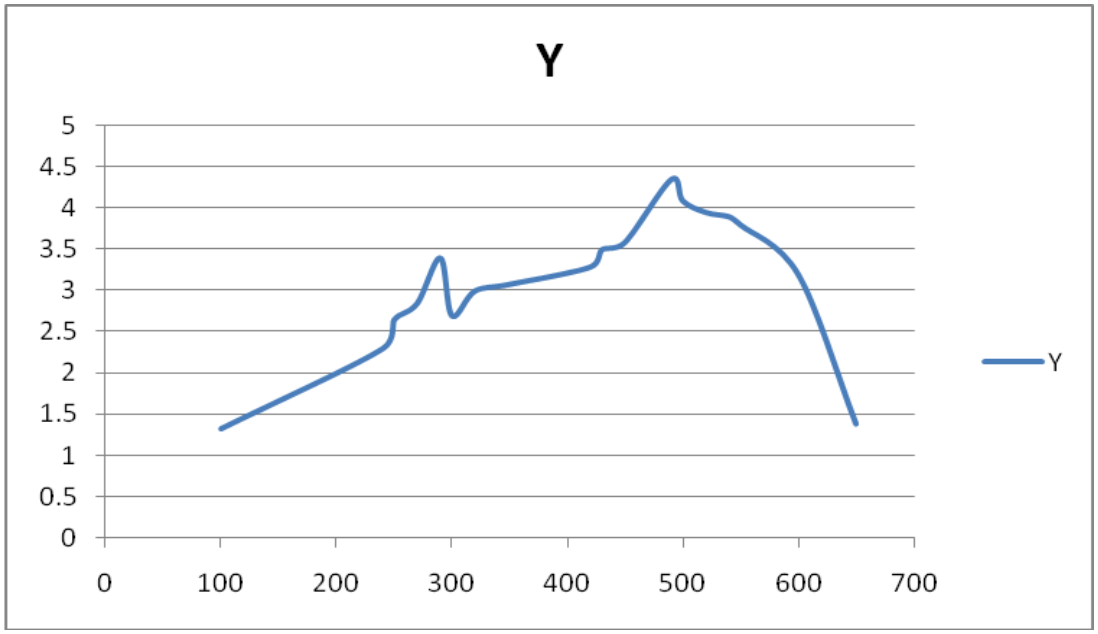



Fig-4.5.10: UV-Visible spectrum of $[Ag(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4.

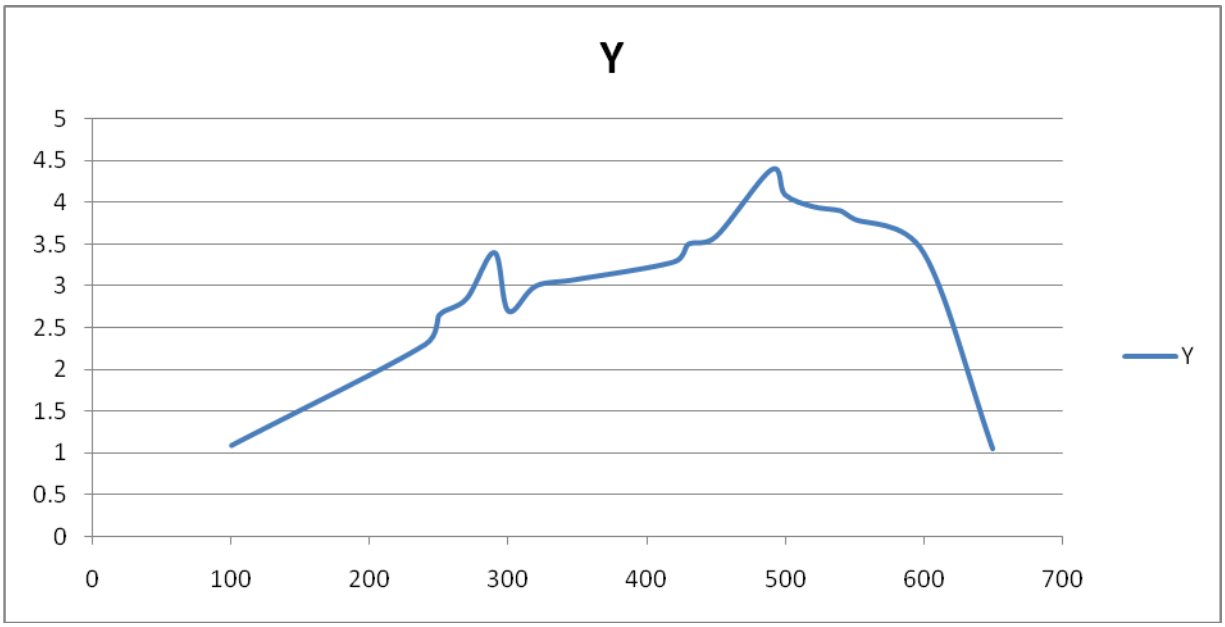



Fig-4.5.11: UV-Visible spectrum of $[Ag(C_{24}H_{28}N_8O_4)(ClO_4)_2]$complex 5.

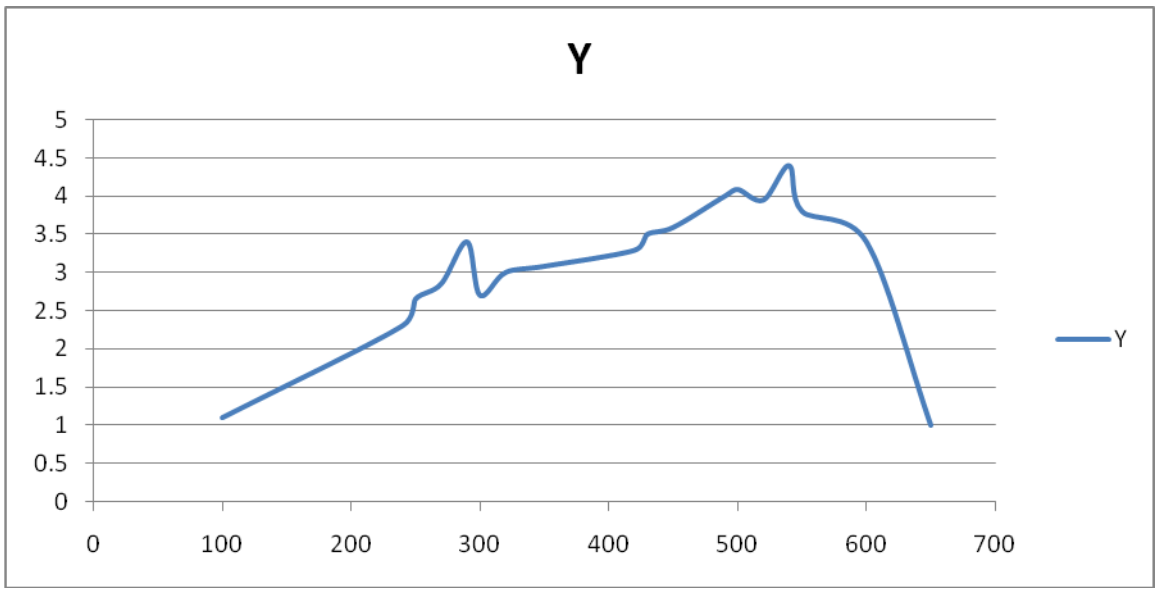



Fig-4.5.12: UV-Visible spectrum of $[Ag(C_{20}H_{24}N_8O_4)\ (ClO_4)_2]$ complex 6.

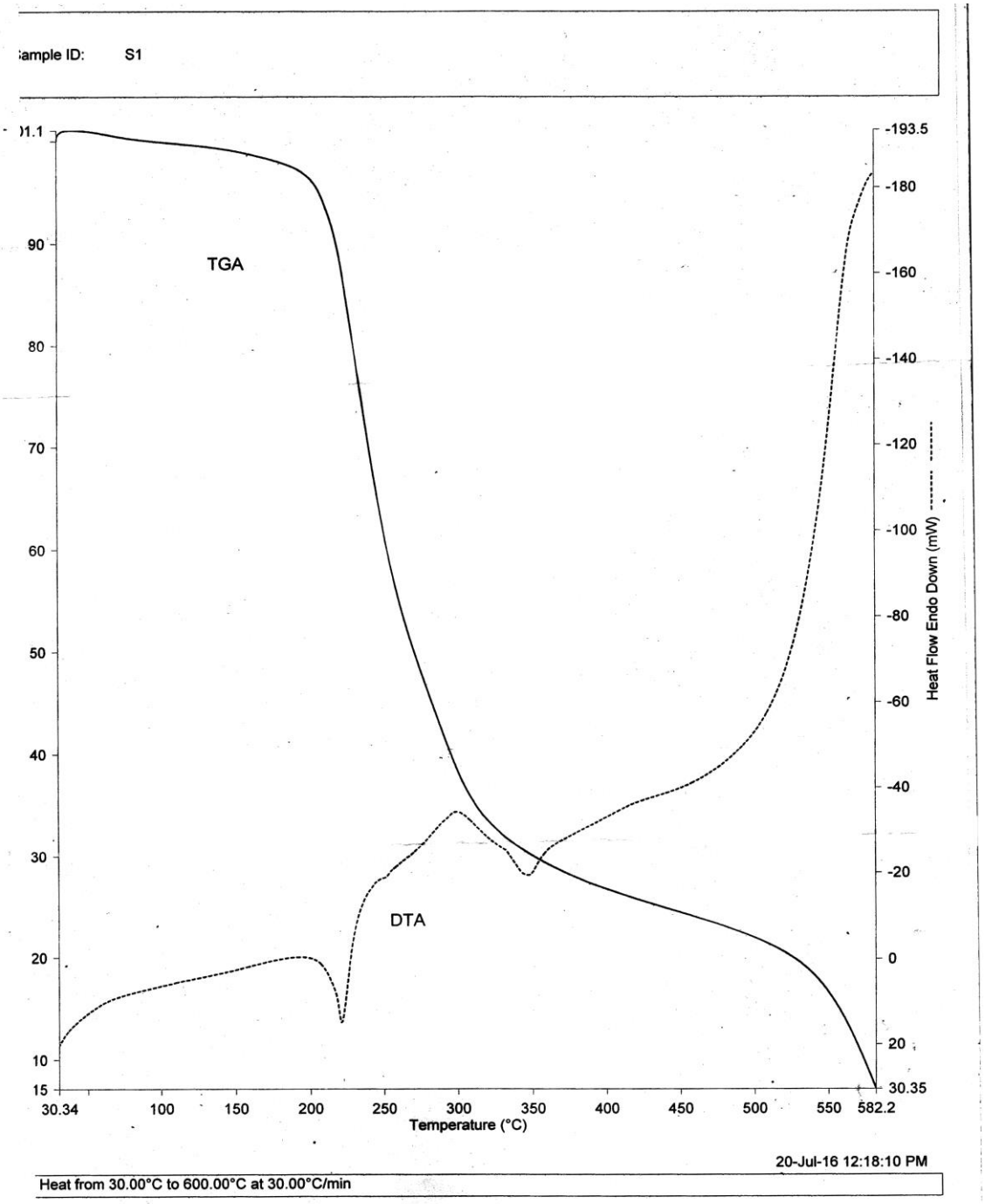



Fig-4.5.13: TGA & DTA spectrum $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex 1.

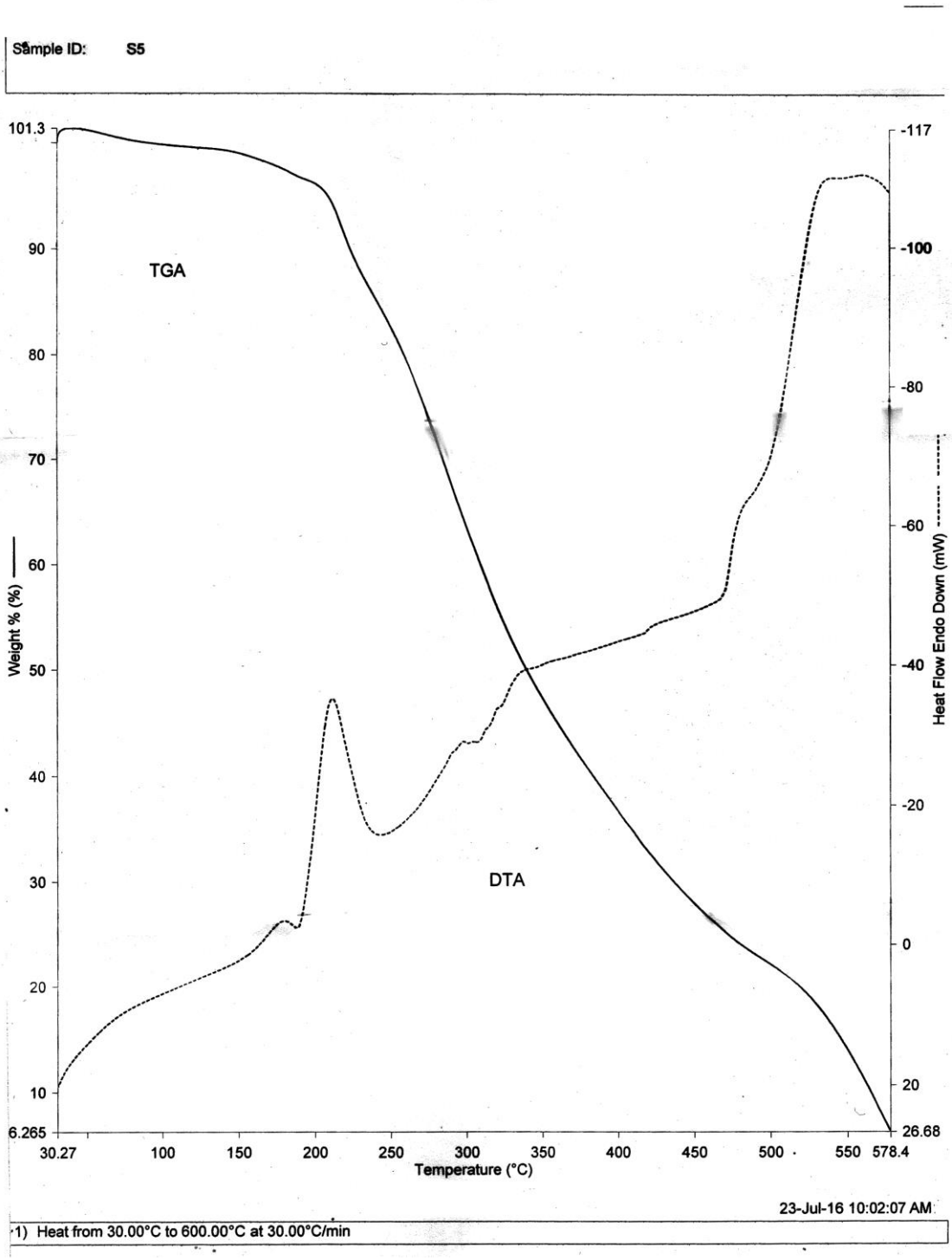



Fig-4.5.14: TGA & DTA spectrum $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ complex 2

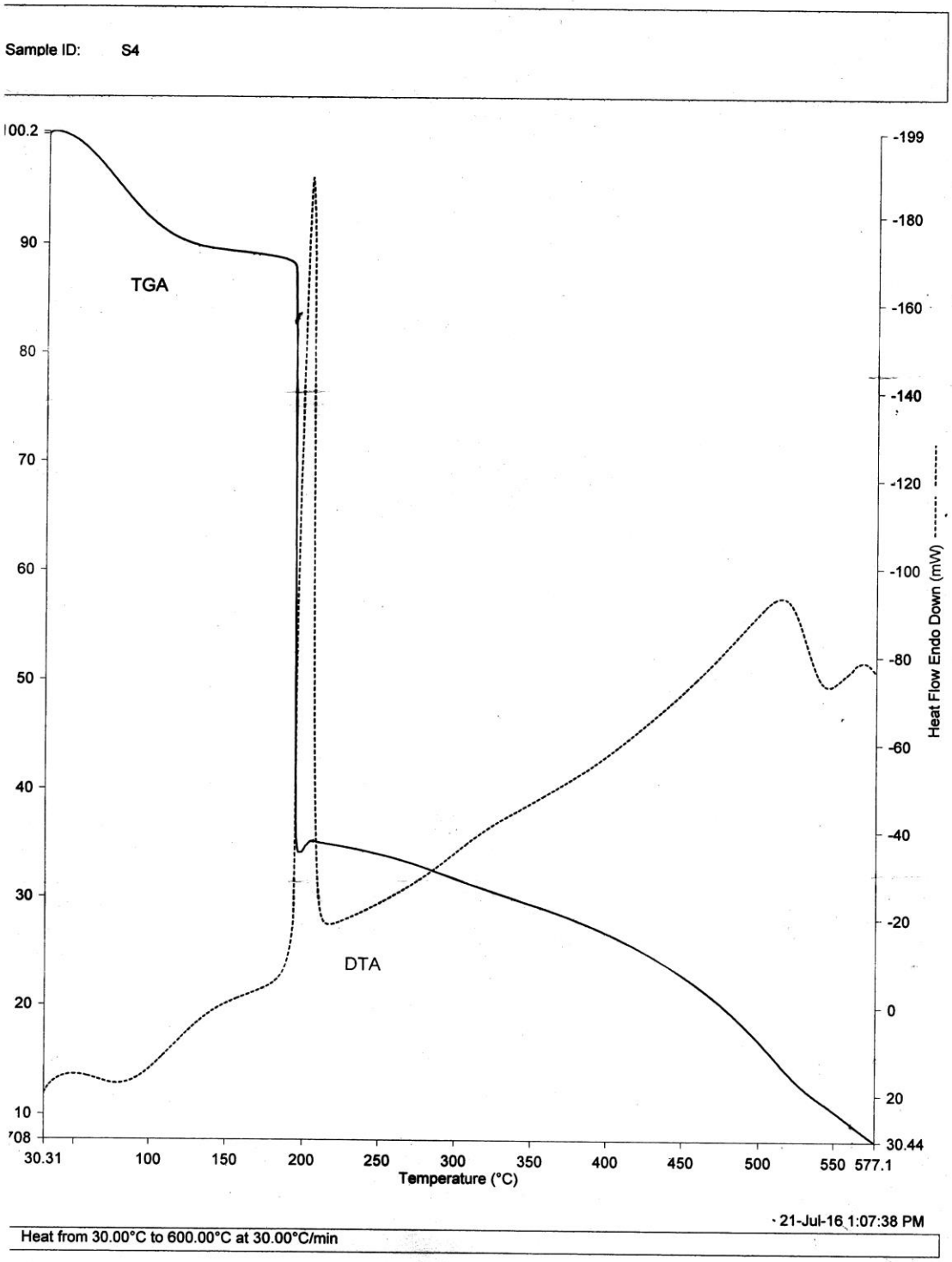



Fig-4.5.15 : TGA & DTA spectrum $[Ag(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3

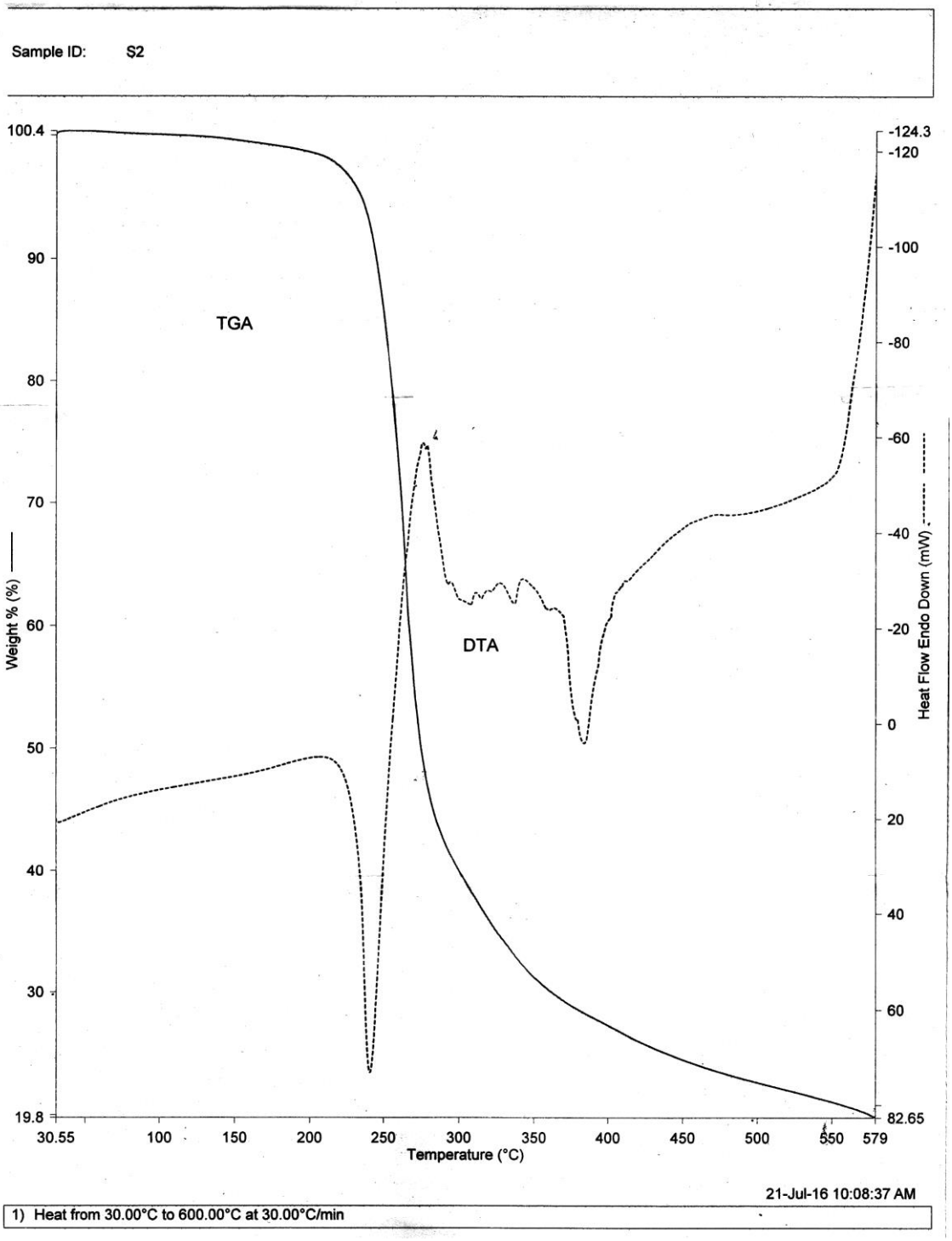



Fig-4.5.16: TGA & DTA spectrum $[Ag(C_{14}H_{24}N_8O_4)(ClO_4)_2]$ complex 4

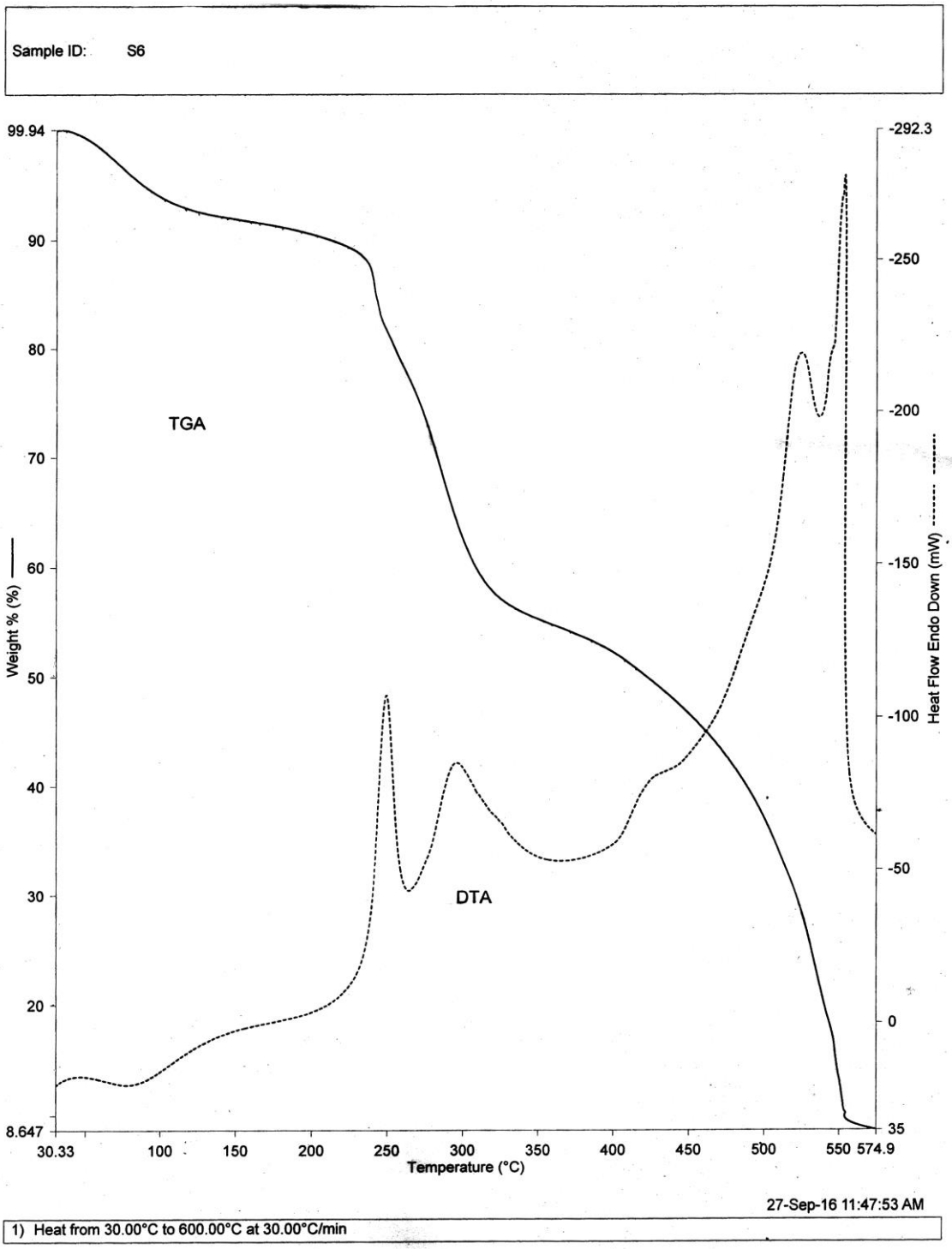



Fig-4.5.17: TGA & DTA spectrum $[Ag(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex 5.

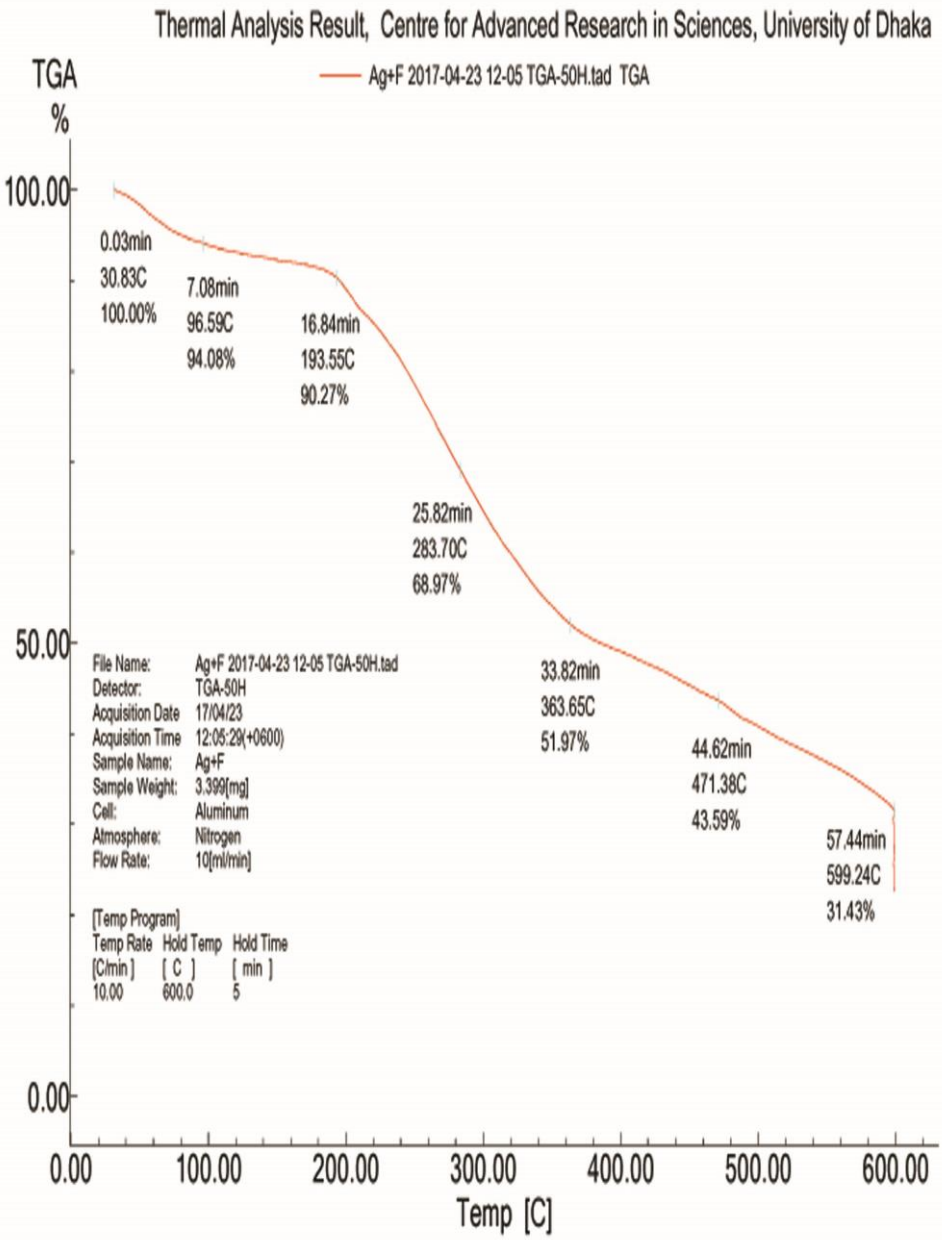


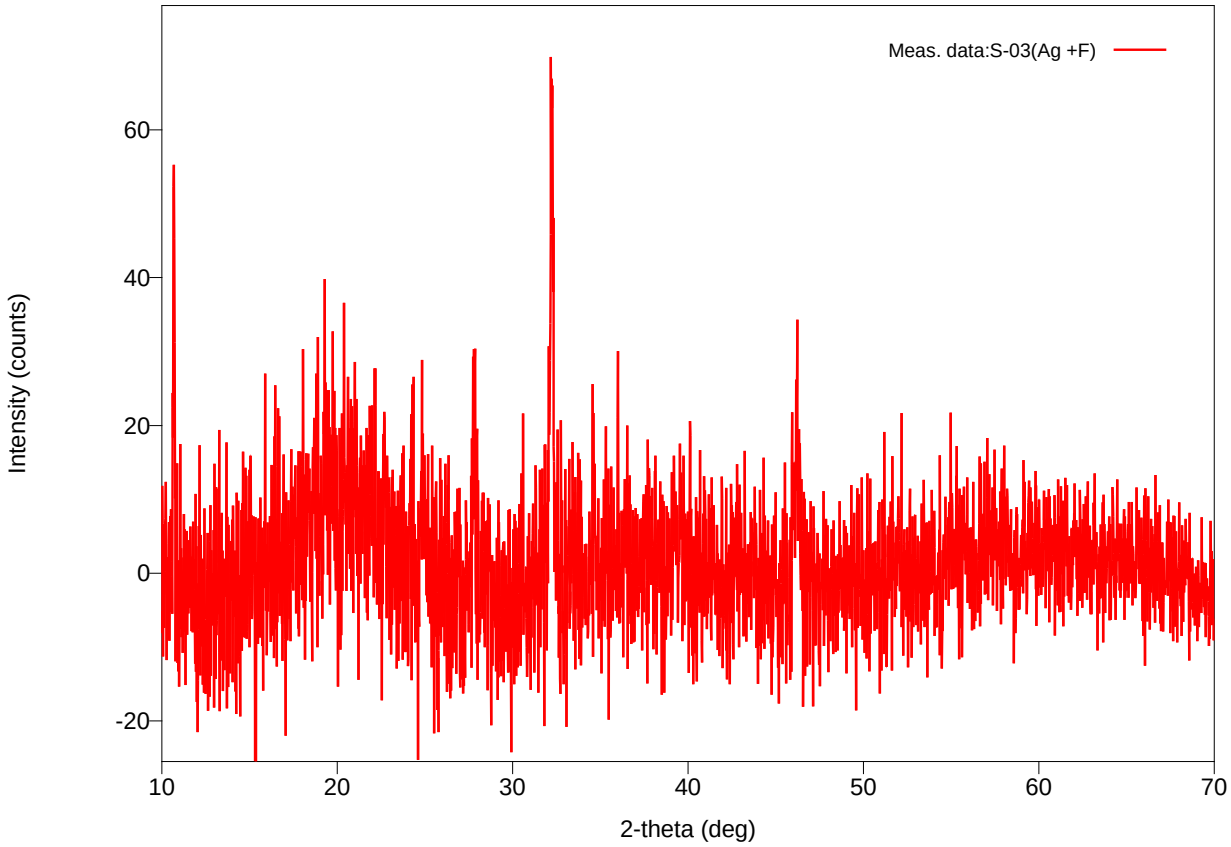

Fig-4.5.18: TGA & DTA spectrum $[Ag(C_{20}H_{24}N_8O_4)(ClO4)_2]$ complex 6.

# Peak List

## General information

| | | | |
|---|---|---|---|
| Analysis date | 2017/03/05 13:08:38 | | |
| Sample name | S-03(Ag +F) | Measurement date | 2017/03/05 12:41:47 |
| File name | S-03(Ag +F).raw | Operator | User1 |
| Comment | | | |

## Measurement profile



# RESULT AND DISCUSSION

## 4.6 A NEW MACROCYCLIC COMPLEXES OF METAL(II)ION

Reactions of ethylene diamine with Pb(II) perchlorate hexahydrate in presence of malonic acid give some 16 member macrocyclic complex as described in sec. 3.7

Complexes(1) are characterized on the basis of elemental analysis, magnetic moment & conductance measurements, UV-visible spectra & infrared studies, thermal studies and other physical properties, like melting point, solubility, colour etc.

Molar conductance data of the complexes (1) are shown in (Table 4.6.1). The conductance values of the complexes suggested that they are non-electrolytic in nature[24].

The infrared spectra of the complexes (1) are shown as spectral data (Table 4.6.4) of the complexes showed a strong and broad band at (3246-3265) $cm^{-1}$ which is assigned for the ν(NH) stretching[25].

Due to coordination the ν(N-H) stretching of the amide group goes to the higher field at (3246, 3265) $cm^{-1}$ region as compared to the starting material malonodihydrazide[26]. In the complexes the terminal-$NH_2$ group of malonodihydrazide condensed with the aldehyde moiety form a new secondary[1]-NH group which may appear at the same region (or overlape) as to the amide-NH group as a result the ν(N-H) band appear as a strong and broad band. [The starting material malonodihydrazide have three ν(N-H)

bands at (3248, 3213, 3050) $cm^{-1}$. The bands at (3248, 3050) $cm^{-1}$ for the asymmetrie and symmetric ν(N-H) stretching of the terminal-$NH_2$ moiety and 3213 $cm^{-1}$ for amidic (N-H) group]. The complexes showed a broad band at (2920-2972) $cm^{-1}$ is suggested for the ν(C-H) stretching of aliphatic moiety[32]. The complexes showed a strong band at (1649-1674) $cm^{-1}$ which represent the ν(C=O) of NH-NH-CO-$CH_2$ moity[27]. Three or four band at (625-1145) $cm^{-1}$ region also indicated the $\nu_1,\nu_2,\nu_3,\nu_4$ bands of ($ClO^-_4$) moiety. These stretching frequency is suggested the coordination of perchlorate to the metal through the O atom[28]. A medium band at (407-412) $cm^{-1}$ region is tentatively attributed to the ν(M-N) mode[29,30.] indicating the coordination of the ligand to the metal through the nitrogen atom.

The magnetic moment measurement data (Table 4.6.3) of the Pb(II) complexes (1-6) showed (1.56-1.78) B.M. These values correspond to no unpaired electrons of Pb(II) $d^{10}$system suggest the octahedral environment of the complexes which are consistent with the literature value[1]. The elemental analyses (C, H and N) (Table 4.6.2) and metal estimation data (Table 4.6.3) of the complexes are consistent with the proposed formula.

The UV-visible spectra of the complexes (1-6) are shown (Table 4.5) band at 320,480 nm, (1) at represent the d-d transition of $^4A_{2g}$ (F) $\rightarrow$ $^4T_{1g}$(F), $^4A_{2g}$(F) $\rightarrow$ $^4T_{1g}$(P), which suggested the octahedral geometry of the Pb (II) complexes[31,32].

Thermal studies: The thermal properties of metal (II) complexes were investigated by thermograms (TGA, DTA) and are shown in (Fig 4.6.13) and the corresponding thermal analysis is presented in (Table.4.6.6). In the

case of complex (1) the decomposition occurs in the (230-325)$^0$C range. There is no mass loss up to 230$^0$C. The first stage of decomposition starts at 230$^0$C and end at 230$^0$C with a corresponding weight loss 25%. Which is accompanied by endothermic effect in the DTA curve in the range 225$^0$C which is accompanied by weight loss confirming the second stage of decomposition is observed at 225-350$^0$C (60% wt. loss).meanwhile the DTA curve exhibits endothermic effect in the range 325$^0$C which is accompanied by weight loss confirming.

X-Ray diffraction: The possible geometry of the product [Pb($C_{10}H_{12}N_4O_4$)($ClO_4$)$_2$] has been deduced on the basis of x-ray power diffraction studies. The result show that the compound belong to the orthorhombic crystal. The 2θ angles are reported in table 4.6.7.

On the basis of elemental analysis magnetic moment and conductance measurements, thermal studies UV Visible spectra, infrared spectra and other physical properties the suggested structure of the complexes are octahedral in nature as in Fig.4.6.20

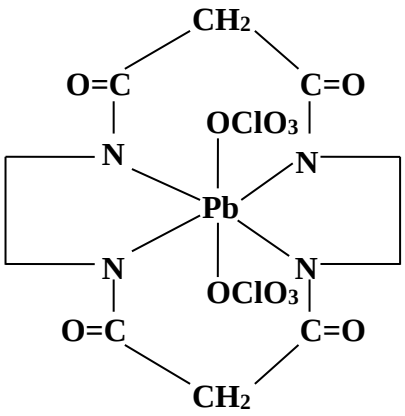



Fig. 4.5.20

Table- 4.6.1: Analytical Data and Other Physical Properties of Compounds (1-6)

| No. | Compounds | %Yield | Colour | Milting point $^0C$ | % M | | Molar conductance ohm$^{-1}$cm$^2$mol$^{-1}$ |
|---|---|---|---|---|---|---|---|
| | | | | | Calculated | Found | |
| 1 | $[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ | 80 | Ash colour | 200 | 21.80 | 21.85 | 56.30 |

Table- 4.6.2: Elemental analysis data of compounds (1-6)

| No. | Compounds | %C | | %H | | %N | |
|---|---|---|---|---|---|---|---|
| | | Calculated | Found | Calculated | Found | Calculated | Found |
| 1 | $[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ | 13.37 | 13.30 | 3.23 | 2.20 | 22.60 | 22.65 |

Table- 4.6.3: Magnetic moment data of compounds (1-6)

| No. | Compounds | Sample length, *l* in cm | Weight of the sample, m in gm | Susceptibility of the empty tube, Ro | Susceptibility of the sample with tube ,R | Mass Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | Molecular weight ,M | Molar Susceptibility $x_g \times 10^{-6}$ C.G.S.unit | μeff B.M |
|---|---|---|---|---|---|---|---|---|---|
| 1 | $[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ | 2.2 | 0.0695 | -48 | -22 | 1.71 | 593 | 1.19 | 1.69 |

Table- 4.6.4: Important infrared spectral bands of compounds (1-6)

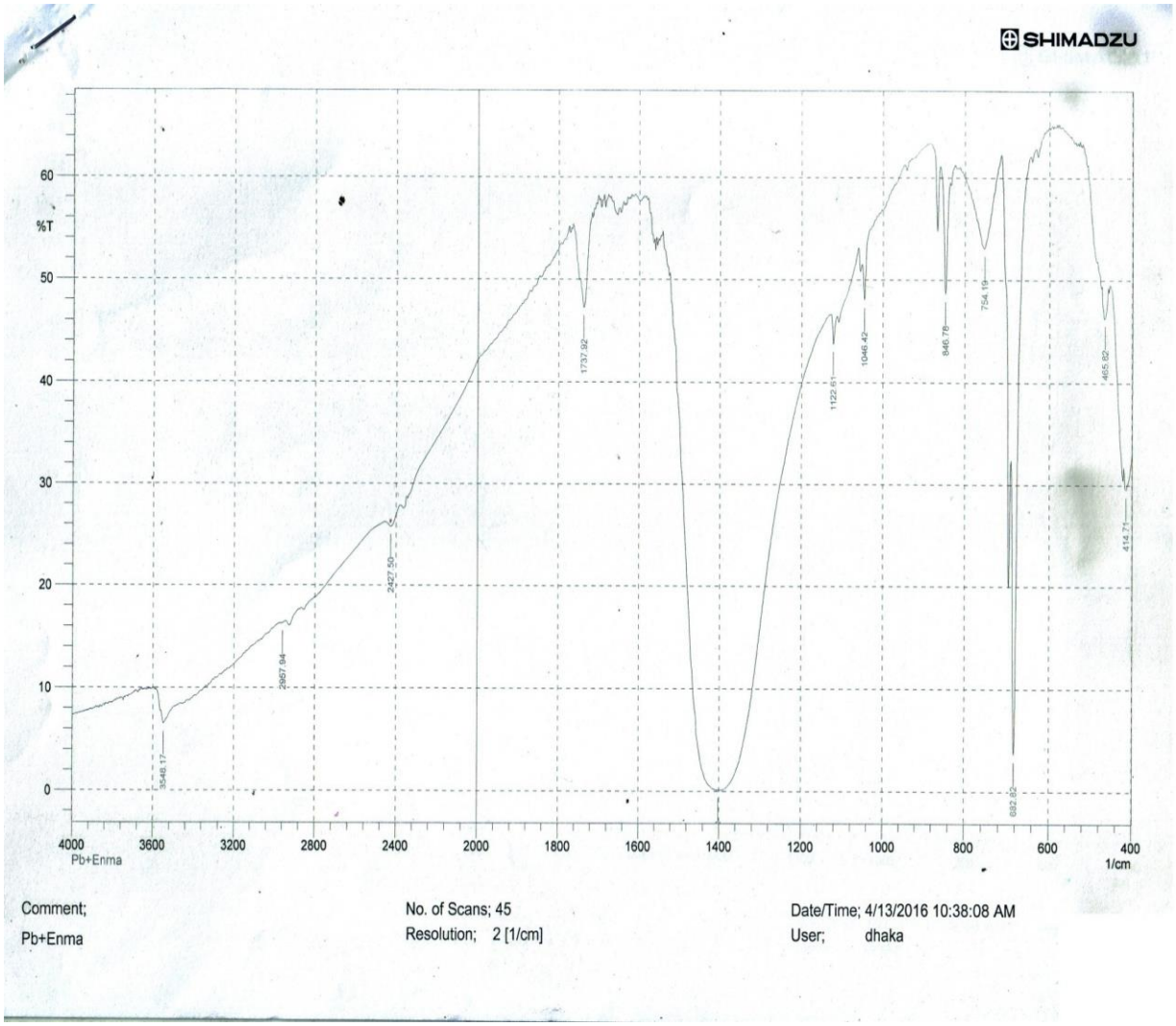
| No. | Compounds | $\nu$(C-H) $cm^{-1}$ | $\nu$(C=O $cm^{-1}$) | $\nu$(N-H) $cm^{-1}$ | $\nu$(M-N) $cm^{-1}$ |
|---|---|---|---|---|---|
| 1 | $[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ | 3047 | 1664 | 3251 | 420 |

Table- 4.6.5: U.V- Visible Adsorption Maxima Of Compounds (1-6)

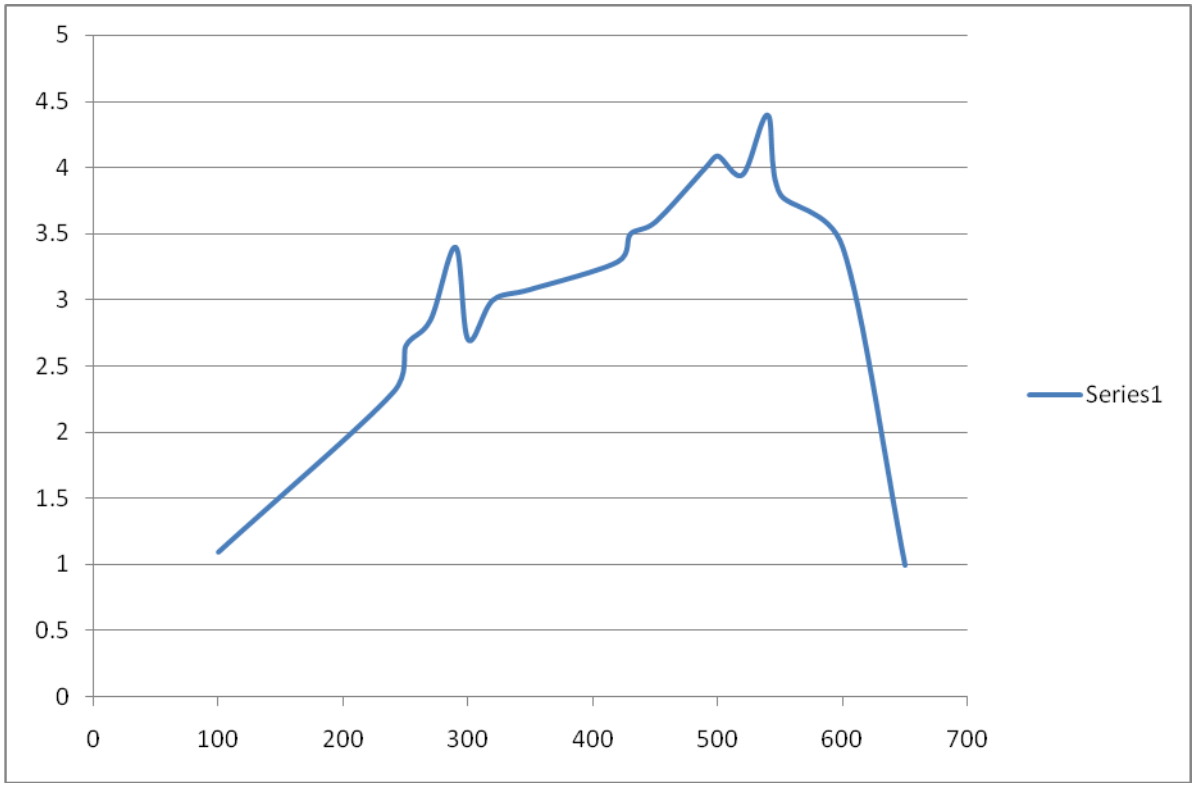
| No. | Compounds | λ max (n,m) |
|---|---|---|
| 1 | $[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ | 294,493 |

Table- 4.6.6: Thermal Analysis Data of Compounds (1-6)

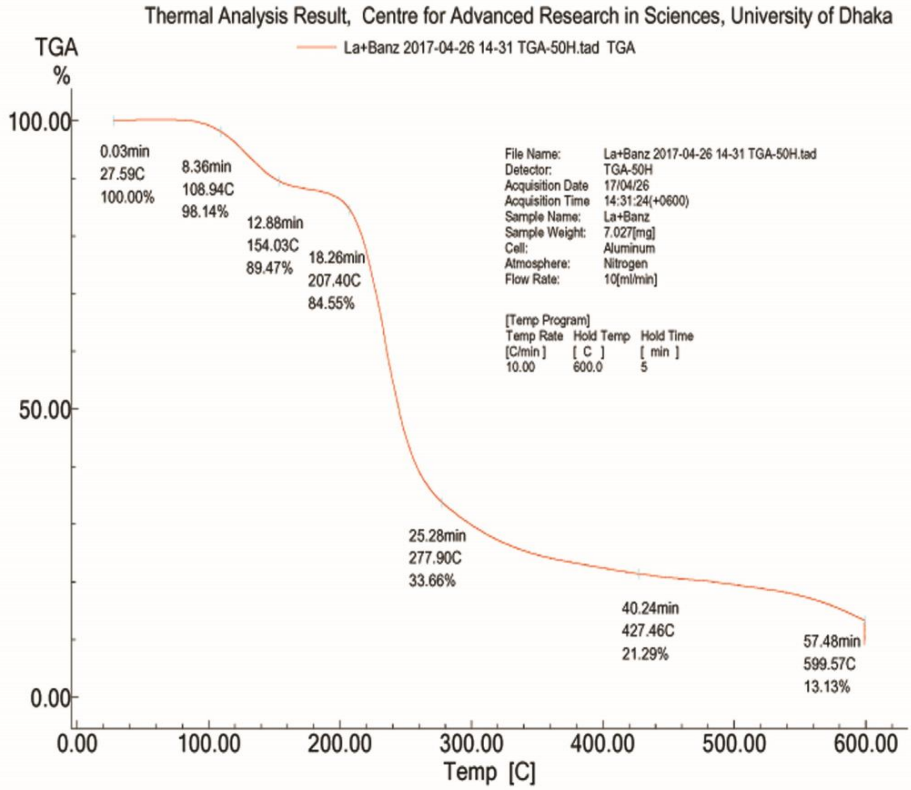
| No. | Compounds | %M (Ligand) | | | %M Metal oxide($Ag_2O$) | | |
|---|---|---|---|---|---|---|---|
| | | Tem$^0$C | Calculated | Found | Tem$^0$C | Calculated | Found |
| 1 | $[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ | 225.00 | 83.22 | 83.20 | 550.00 | 37.66 | 37.60 |




$[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ complex 1**.**




$[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ complex 1.




$[Pb(C_{10}H_{12}N_4O_4)(ClO_4)_2]$ complex 1.

# Peak List

## General information

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| Comment | | | |

## Measurement profile

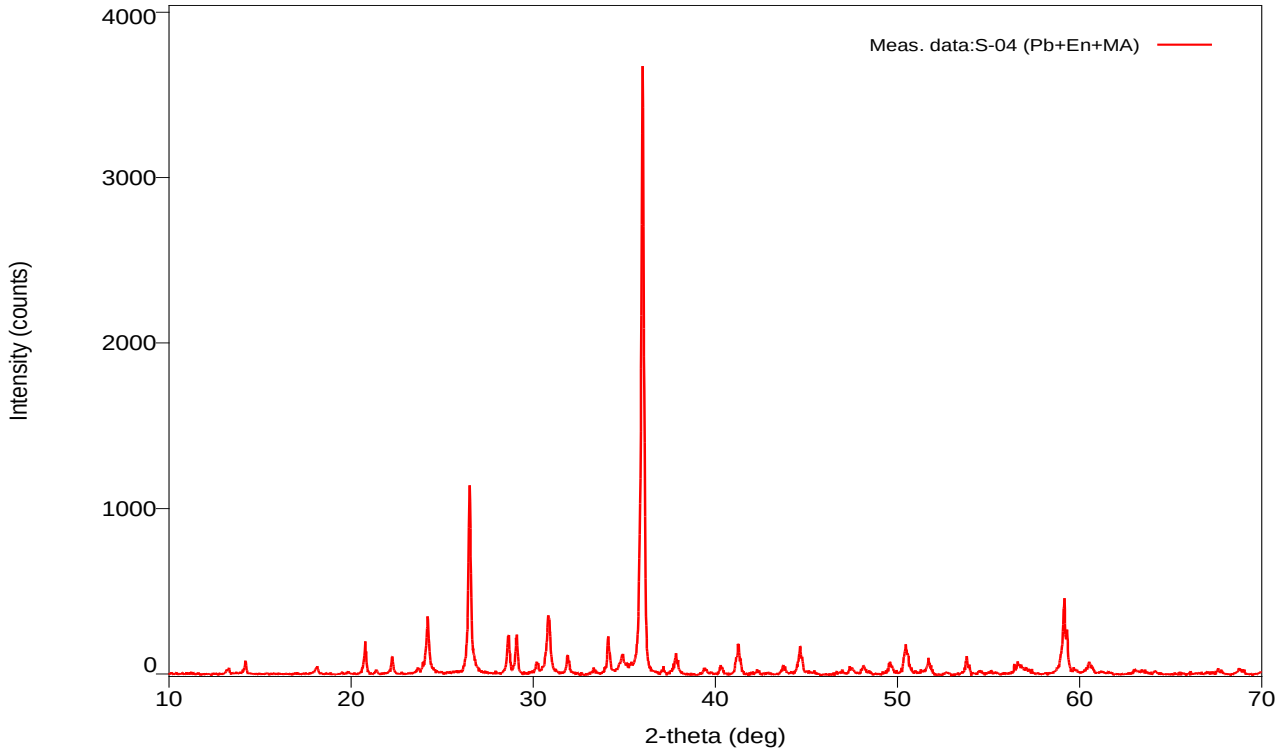

Table. 4.6.7: X-Ray diffraction data.

| No. | 2-theta(deg) | d(ang.) | FWHM(deg) | Asym. factor | Rel. int. I(a.u.) |
|---|---|---|---|---|---|
| 1 | 14.207(4) | 6.2291(18) | 0.085(8) | 0.99(15) | 1.55 |
| 2 | 20.796(4) | 4.2679(9) | 0.115(10) | 4.0(16) | 3.85 |
| 3 | 22.274(8) | 3.9879(14) | 0.089(16) | 2.5(6) | 2.10 |
| 4 | 24.211(10) | 3.6731(15) | 0.16(2) | 1.4(5) | 11.91 |
| 5 | 26.494(5) | 3.3614(6) | 0.118(10) | 0.74(17) | 31.40 |
| 6 | 28.662(8) | 3.1120(8) | 0.139(8) | 2.9(8) | 5.67 |
| 7 | 29.115(6) | 3.0645(7) | 0.134(7) | 3.4(10) | 5.17 |
| 8 | 30.209(9) | 2.9560(9) | 0.08(2) | 1.5(7) | 1.69 |
| 9 | 30.858(11) | 2.8953(10) | 0.200(11) | 1.9(5) | 13.53 |
| 10 | 31.89(2) | 2.804(2) | 0.149(17) | 1.1(6) | 2.18 |
| 11 | 34.130(8) | 2.6248(6) | 0.100(11) | 1.3(6) | 5.37 |
| 12 | 34.89(2) | 2.5691(14) | 0.28(2) | 1.6(4) | 4.35 |
| 13 | 36.037(3) | 2.49023(19) | 0.153(3) | 3.8(5) | 100.00 |
| 14 | 37.819(9) | 2.3769(6) | 0.23(3) | 0.8(6) | 3.49 |
| 15 | 40.272(5) | 2.2376(3) | 0.103(16) | 0.3(3) | 1.17 |
| 16 | 41.265(18) | 2.1860(9) | 0.232(12) | 1.5(4) | 5.15 |
| 17 | 43.740(6) | 2.0678(3) | 0.101(19) | 1.0(9) | 1.44 |
| 18 | 44.680(11) | 2.0265(5) | 0.177(11) | 3.2(12) | 5.30 |
| 19 | 47.40(3) | 1.9164(11) | 0.18(4) | 0.4(4) | 1.28 |
| 20 | 48.14(2) | 1.8885(8) | 0.15(3) | 1.5(10) | 1.43 |
| 21 | 49.630(16) | 1.8354(6) | 0.17(2) | 4(2) | 2.06 |
| 22 | 50.471(9) | 1.8067(3) | 0.184(11) | 2.1(5) | 6.96 |
| 23 | 51.716(11) | 1.7661(4) | 0.149(16) | 1.1(2) | 2.89 |
| 24 | 53.822(11) | 1.7019(3) | 0.136(11) | 2.7(12) | 2.34 |
| 25 | 56.68(3) | 1.6226(8) | 0.40(4) | 1.5(3) | 4.71 |
| 26 | 59.160(5) | 1.56040(12) | 0.131(8) | 0.96(17) | 17.35 |
| 27 | 60.54(2) | 1.5282(5) | 0.18(3) | 1.1(7) | 3.27 |
| 28 | 63.20(13) | 1.470(3) | 0.64(10) | 0.7(6) | 1.40 |
| 29 | 67.60(3) | 1.3846(5) | 0.13(3) | 0.8(8) | 0.77 |
| 30 | 68.76(3) | 1.3641(4) | 0.15(3) | 0.4(4) | 1.15 |

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# ANTIBACTERIAL ACTIVITY TESTING

## 5.1 INTRODUCTION AND PRINCIPLE

Any chemical or biologic agent that either kills or inhibits the growth of microorganism is called antimicrobial agent.

The susceptibility of microorganism to antimicrobial agent can be determined in vitro by a number of methods. The disc diffusion technique[1,2] is widely acceptable for preliminary investigation of materials, which are suspected to posses antimicrobial properties. Diffusion procedure is normally used for inessential qualitative test, which allocates organism of the susceptible intermediate (moderately susceptible) or resistant categories.

The dried filter paper discs containing the materials are usually applied to the test plate containing the culture of microorganisms. These are kept at low temperature ($4^0$C) for 24 hours.

Initially the dried discs absorb water from the surrounding test medium and the drug is dissolved. The drug migrates through the adjacent test medium by concentration gradient of the drug according to physical law that governs diffusion of molecules through and agar gel[3]. As a result there is a gradual change of drug concentration in the agar surrounding each disc. Then the plates are incubated in an incubator at $37^0$C for 6 hours. Activities of test sample are expressed by measuring the zone of inhibition observed around the area of the disc.

As the antibiotic diffusion progresses microbial multiplication also proceeds. After an initial lag phase a logarithmic growth phase in initiated at that moment bacterial multiplication proceeds more rapidly than the drug can diffuse and the bacterial cell which are not inhibited by the antimicrobial agents will continue to multiply until a lawn of grown can be visualised. No growth will appear in the area where drug is present in inhibitory concentration.

Generally more susceptible the test organism the larger is the circular zone of inhibition. Antimicrobial activities of the test sample are expressed by measuring the zone of inhibition observed around the area of the disc. The diameter of the inhibition is usually measured to understand the extent of inhibition in different concentration.

**The size of the inhibitory zones depends on the following principle factors.**

i. Intrinsic antimicrobial sensitivity of the test sample.
ii. Growth rate of the test microorganism.
iii. Diffusion rate of the drug which is rated to its water solubility.
iv. Number of concentration of the freshly seeded test organism.
v. Amount of the test sample on disc.
vi. Thickness of the test medium in the petridishes.
vii. Thickness of the filter paper disc.

## 5.2 APPARATUS AND REAGENTS

i. Micropipette.
ii. Autoclave.
iii. Incubator
iv. Refrigerator.
v. Filter paper disc.
vi. Petri dishes.
vii. Inoculation loop.
viii. Sterile cotton.
ix. Sterile forceps.
x. Spirit lamp.
xi. Laminar air flow unit.
xii. Nutrient agar.

## 5.3 METHOD

The test organisms are pathogenic for human beings. For this reason, all steps of the work were done with high precaution and aseptic condition which are mentioned below. All steps of the work were carried out at microbiology laboratory at Microbiology Department in Dhaka University.

## 5.4 TEST OF ORGANISMS USED FOR THE STUDY

Fourteen pathogenic bacteria were selected for the test, ten of which were gram negative and the rests of them were gram positive.

List of the Fourteen Pathogenic Bacteria

| *Gram Negative* | *Gram Positive* |
|---|---|
| ***1. Salmonella-17*** | ***11. Bacillus megaterium*** |
| ***2. Klebsilla*** | ***12. Sarcina lutea*** |
| ***3. Shigella dysenteriae*** | ***13. Staphylococcus aureus*** |
| ***4. Shigella shiga*** | ***14. Bacillus cereus*** |
| ***5. Shigella boydii*** | |
| ***6. Shigella sonnei*** | |
| ***7. Shigella flexneri*** | |
| ***8. Escherichia coli*** | |
| ***9. Pseudomonas aeruginosa*** | |
| ***10. Salmonella*** | |

## 5.5 CULTURE MEDIA

Nutrient agar medium was used as culture media. The instant nutrient agar (DIFCO) medium was weight and then reconstituted with distilled water in a conical flask according to pacification (2.3% w/v). The formulation of nutrient agar media (DIFCO) is as follows:

**Nutrient agar (mast diagnostics)**

| Formulation | Grams/litre |
|---|---|
| Peptone A | 6.0 |
| Yeast extract | 2.0 |
| Beef extract | 1.0 |
| Sodium chloride | 5.0 |
| Agar | 14.0 |
| Distilled water sq. to 1000 mL | |

Total 28 grams of power was weighed and dispersed in one litre of distilled water allowed to shake for 10 minutes, scoirled to mixed and then sterilised by autoclaving for 15 minutes at $121^0$C. Then medium was cooled to (40-45)$^0$c and mixed well, then poured in to plates.

## 5.6 PREPARATION OF FRESH CULTURE

The liquid culture is called broth culture. The culture media without agar powder per litre.

| Formulation | Grams/Litre |
|---|---|
| Bacto tryptone | 10.0 g |
| Bacto yeast extract | 5.0 g |
| NaCl | 10.0 g |

Adjusted pH to 7.5 with sodium hydroxide.

Trpytone NaCl and yeast extract of calculate amount were taken in a conical flask and distilled water was added (volume should be less then 1 litre) the contents were heated in water bath to make a clear solution. The pH of the solution was then adjusted 7.5 using NaOH or HCl as necessary. Distilled water was added sufficiently to make the final volume (1 liter). Again the total volume was heated on a water bath to obtain a clear solution. The conical flask was plugged with cotton and then autoclaved at 1 atm pressure for 15 minutes at $120^0$C.

50 mL of broth medium was transferred in a conical flask. The test microorganisms of pure culture were streaked on the nutrient broth media with the help of sterile loop in an aseptic condition and incubated at $37^0$C for 24 hours. The broth culture thus obtained was considered as fresh culture. Fresh culture of this type was always used throughout the sensitivity testing.

## 5.7 PREPARATION OF THE CULTURE PLATE

A small bottle containing 10 mL sterile nutrient broth was taken and the test organism (Bacillus megaterium) from the pure culture transferred to this bottle with the help of an inoculation loop in an aseptic condition. After inoculation the bottle was subjected to incubation at $37^0$C for 24 hours to provide sufficient time and temperature for the growth of the test organism.

To 100 mL of the nutrient agar 1 mL of the prepared culture was added and was mixed thoroughly with shaking. A 25 mL portion of this culture was poured in to a petridish and in order to faeifitate homogeneous distribution of the test organism, the petidish was rotated several times first in clockwise

direction and then in anticlockwise direction. The media were pour in to petridish on a level horizontal surface so as to give a uniform depth of approximately 4 mm. The petridish was kept undisturbed for about 15 minutes during which it was solidified. After complete solidified of the media 4-5 holes were made inside it with the help of a brother.

Just before using plates with lids agar were placed in an incubator (250C) for about 10-15 minutes until the execs of surface moisture was lost by evaporation. There should be no droplets of on observing their antibacterial activities the species *Bacillus megatrium*, was taken as test organism.

**5.8 Preparation of discs**

A. Sample discs.

i. Solution of the compounds were prepared in respective solvents so that 20 μL contained 200 μg of the compounds
ii. Filter paper disc were taken in petridish and sterilised by oven at $110^0$C for 1 hours.
iii. 20 μL of the solutions were placed on the discs with the help of a micropipette thus discs containing 200 μg compounds were prepared.
iv. These discs were than air-dried.

**B. Standard Disc**

Ready mad ekanamycin K-30 μg/disc containin 30 μg/disc of antibiotic kanamycin were used as standard disc.

## 5.9 PLACEMENT OF THE DISC AND INCUBATION

The solidified agar plate were seeded with the 200 μL of fresh culture with the help of a micropipette and spread the microorganisms with the help of a sterile spreader in an aseptic condition.

The prepared disc of samples were placed gently on the freshly seeded solidified agar plates with a sterile forceps. Standard disc were also placed on the test plate to compare the effect of the test sample and to nullity the effect of solvent respectively.

The plates were then kept in a refrigerator at $4^0$C for 24 hours in order that the materials had sufficient time to diffuse to a considered area of the plates. After this the plates were incubated at $37^0$C for 6 hours.

## 5.10 Determination of the zone of inhibition

The antimicrobial potency of the test agents are measured by their activity to prevent the growth of the microorganisms surrounding the discs which gives clear zone of inhibition.

After incubation, the antibacterial activity of the test materials was determined by measuring the diameter of the zones of inhibition in millimeter with transparent scale, result, obtained from these are listed in the Table (5.1-5.14)

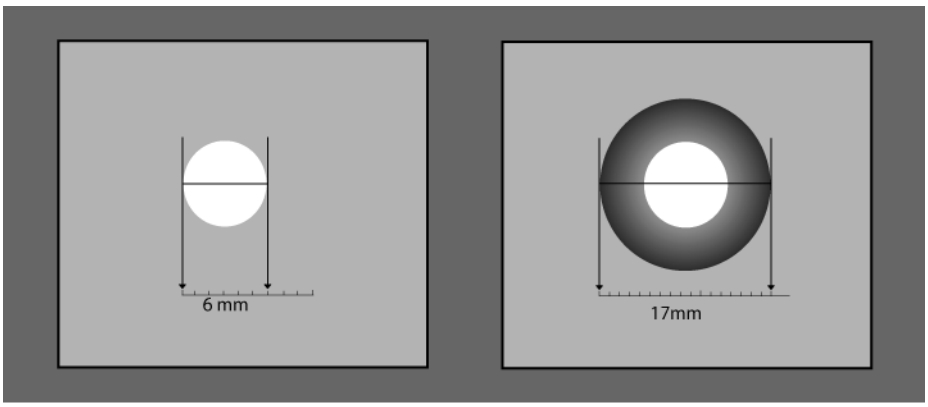


Fig 5: Determination of the zone of inhibition

## 5.11 RESULT Pb (II) CONTAING COMPLEX.

RESULT OF THE ANTIBACTERIAL ACTIVITY OF THE COMPLEXES (1-6) AGAINIST THE FOURTEEN PATHOGENIC BACTERIA VIZ. *SALMONELLA-17, KLEBSILLA, SHIGELLA DYSENTERIAE, SHIGELLA SHIGA, SHIGELLA BOYDII, SHIGELLA SONNEL, SHIGELLA FLEXNERI, ESCHERICHIA COLI, PSEUDOMONAS AERUGINOSA, SALMONELLA, BACILLUS MEGATERIUM, SARCINA LUTEA, STAPHYLOCOCCUS AUREUS, BACILLUS CEREUS.*

Table 5.0 Complexes abbreviation for antibacterial activity

| Complexes No. | Compound | Symbol |
|---|---|---|
| 1. | $[Pb(C_8H_{16}N_8O_4)(ClO_4)_2]$ | Pb+F+L |
| 2. | $[Pb(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | Pb+A+L |
| 3. | $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | Pb+B+L |
| 4. | $[Pb(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | Pb+Cro+L |
| 5. | $[Pb\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | Pb+Ci+L |
| 6. | $[Pb\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | Pb+Banzal+L |
| Ligand | $C_3N_4H_8O_2$ | L |

Table 5.1: Antibacterial activity of the complexes 2.3,4 against *Salmonella-17.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | Pb+A+L | 20 | 10 | 6 | - |
| 3 | Pb+B+L | 17 | 8 | 3 | - |
| 4 | Pb+Cro+L | 18 | 10 | 5 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 16 |

Table 5.2: Antibacterial activity of the complexes 3.4,5 against *Klebsilla*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Pb+B+L | 18 | 8 | 4 | - |
| 4 | Pb+Cro+L | 15 | 7 | 3 | - |
| 5 | Pb+Ci+L | 17 | 9 | 4 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 19 |

Table 5.3: Antibacterial activity of the complexes 4, 5, 6 against *Shigella dysenteriae.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 4 | Pb+Cro+L | 12 | 6 | 3 | - |
| 5 | Pb+Ci+L | 10 | 5 | 2 | - |

| | | | | | |
|---|---|---|---|---|---|
| 6 | Pb+Banzal+L | 6 | 3 | 2 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 21 |

Table 5.4: Antibacterial activity of the complexes 3,4,5 against *Shigella shiga*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Pb+B+L | 18 | 10 | 4 | - |
| 4 | Pb+Cro+L | 20 | 10 | 5 | - |
| 5 | Pb+Ci+L | 15 | 8 | 4 | - |
| | Control disc | Nil | | | - |
| | Standard disc | | | | 21 |

Table 5.5: Antibacterial activity of the complexes 3, 4, 5 against *Shigella boydii.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Pb+B+L | 18 | 10 | 5 | - |
| 4 | Pb+Cro+L | 15 | 8 | 4 | - |
| 5 | Pb+Ci+L | 14 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 5.6: Antibacterial activity of the complexes 3, 4,5 against *Shigella sonnei.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) |
|---|---|---|

| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
|---|---|---|---|---|---|
| 3 | Pb+B+L | 18 | 10 | 5 | - |
| 4 | Pb+Cro+L | 12 | 6 | 3 | - |
| 5 | Pb+Ci+L | 16 | 8 | 4 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 5.7: Antibacterial activity of the complexes 1, 2, 3 against *Escherichia coli.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1. | Pb+F+L | 26 | 12 | 5 | - |
| 2. | Pb+A+L | 20 | 10 | 4 | - |
| 3 | Pb+B+L | 15 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 5.8: Antibacterial activity of the complexes 2, 3, 4 against *Salmonella.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | Pb+A+L | 10 | 8 | 2 | - |
| 3 | Pb+B+L | 18 | 14 | 4 | - |
| 4 | Pb+Cro+L | 18 | 9 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 5.9: Antibacterial activity of the complexes 2, 3, 4 against *Bacillus megaterium.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | Pb+A+L | 20 | 10 | 5 | - |
| 3 | Pb+B+L | 18 | 9 | 4 | - |
| 4 | Pb+Cro+L | 16 | 8 | 3 | - |
| | Standard disc | | | | 18 |

Table 5.10: Antibacterial activity of the complexes 2, 3, 4 against *Sarcina lutea.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | Pb+A+L | 20 | 12 | 6 | - |
| 3 | Pb+B+L | 18 | 9 | 4 | - |
| 4 | Pb+Cro+L | 16 | 7 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 18 |

Table 5.11: Antibacterial activity of the complexes 3, 4,5 against *Staphylococcus aureus.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Pb+B+L | 28 | 14 | 6 | - |
| 4 | Pb+Cro+L | 20 | 10 | 4 | - |
| 5 | Pb+Ci+L | 18 | 9 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 18 |

Table 5.12: Antibacterial activity of the complexes 3, 4, 5 against *Bacillus cereus*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Pb+B+L | 18 | 10 | 5 | - |
| 4 | Pb+Cro+L | 12 | 6 | 3 | - |

| | | | | | |
|---|---|---|---|---|---|
| 5 | Pb+Ci+L | 16 | 8 | 4 | - |
| | Control disc | Nil | - | - | |
| | Standard disc | | | | 20 |

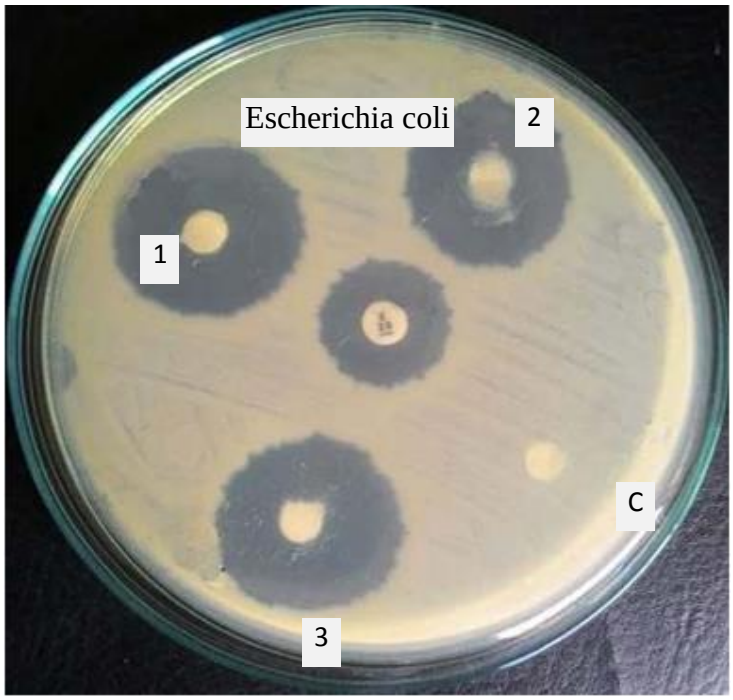


Fig: 5.1: Photographic representation of zone of inhibition of the complexes1,2, 3 the standard compound kanamycin against Escherichia coli.

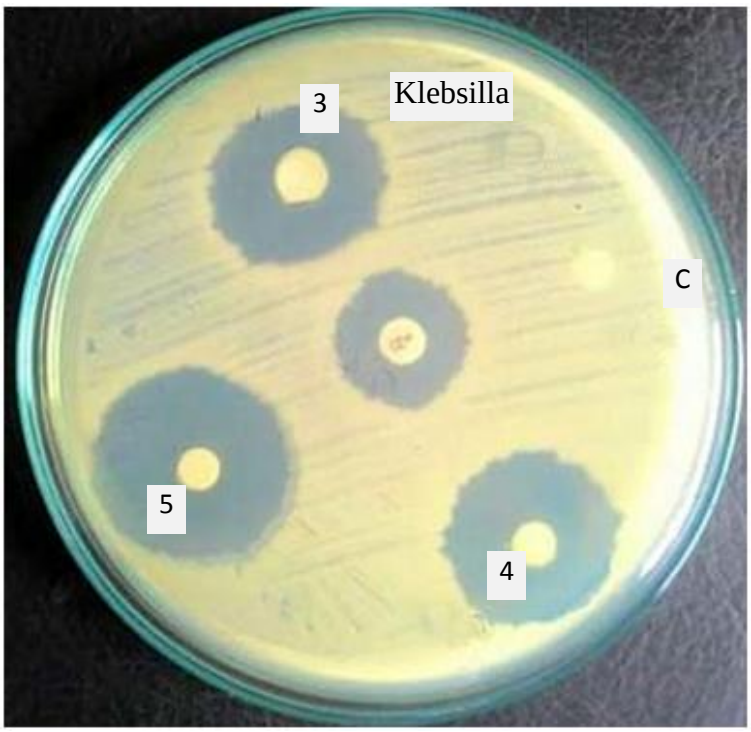


Fig: 5.2: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycin against Klebsilla.

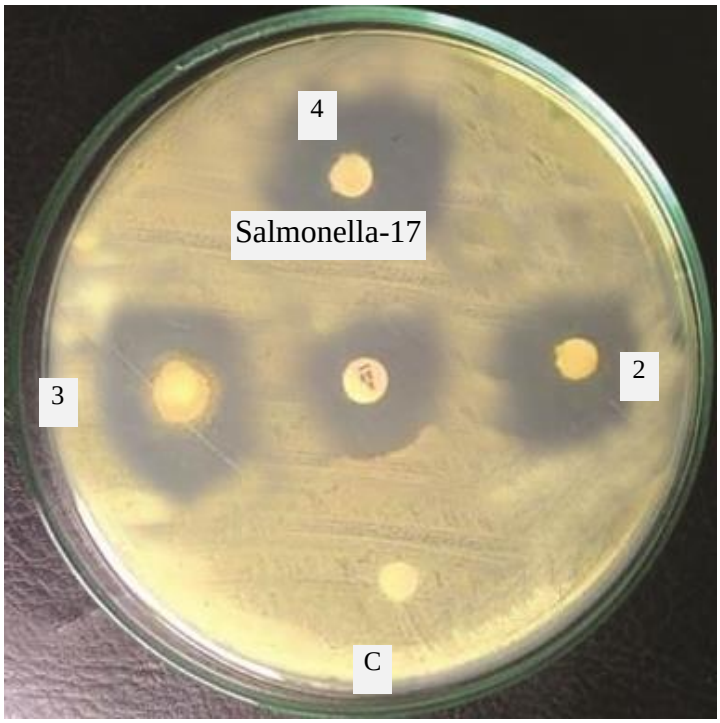

Fig: 5.3: Photographic representation of zone of inhibition of the complexes 2,3,4 the standard compound kanamycin against Salmonella-17.

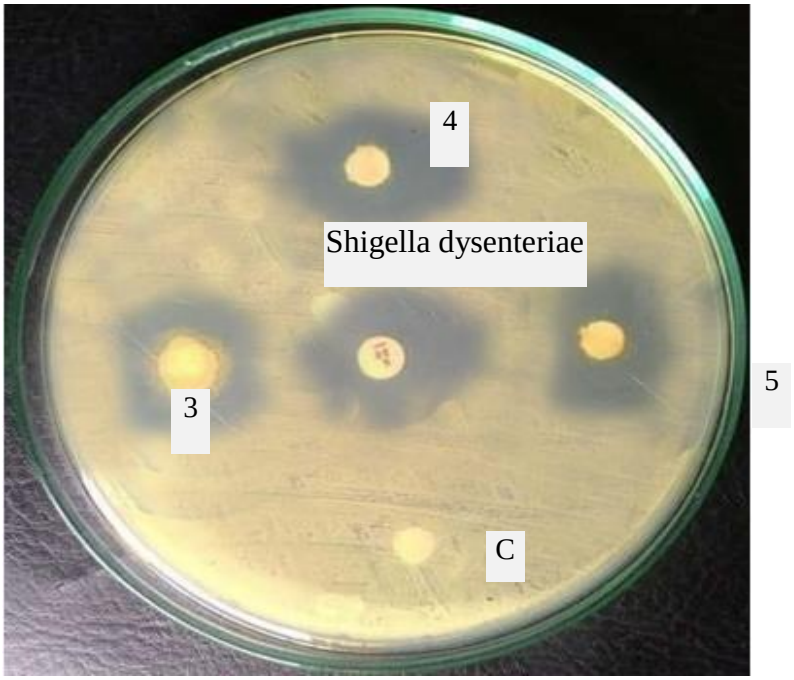


Fig: 5.4: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycin against Shigella dysenteriae.

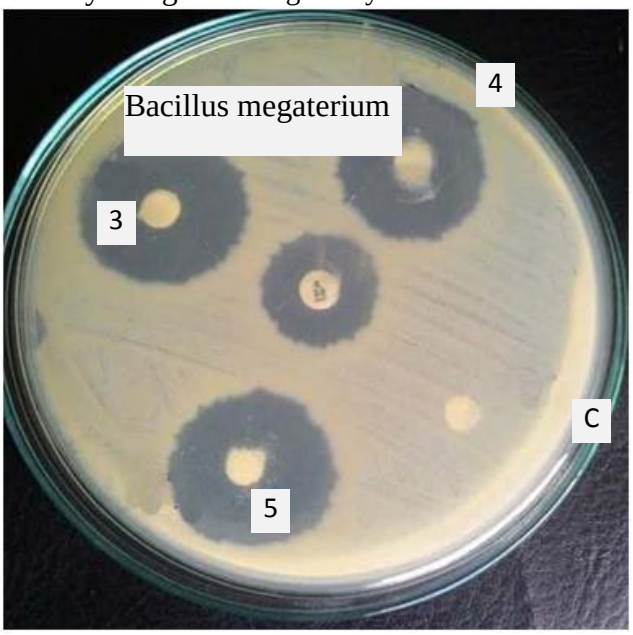


Fig: 5.5: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycin against Bacillus megaterium.

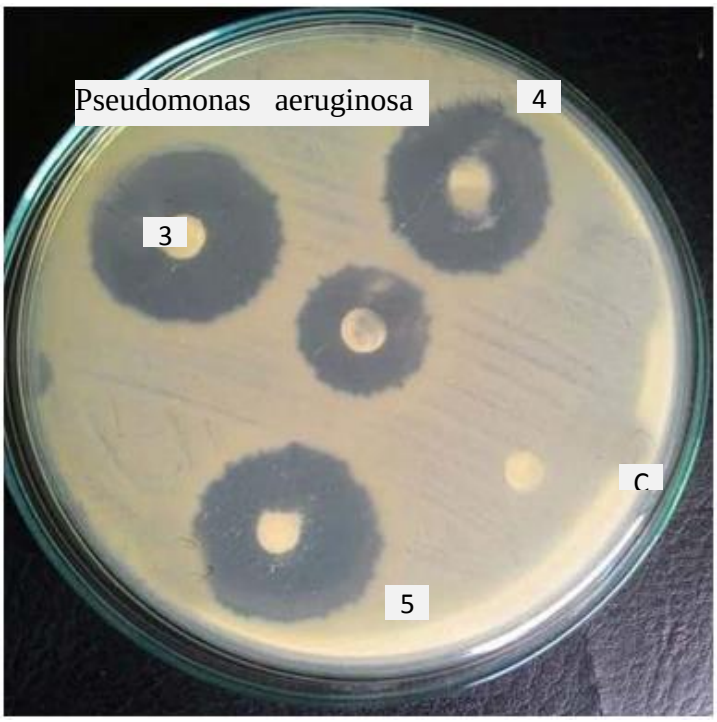


Fig: 5.6: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycin against Pseudomonas aeruginosa.

## 5.12 DISCUSSION

It has been observed that some drug increases the activity when administered as metal complexes or their metal chalets[4]. The antibacterial activity of the metal complexes 3, 4, 5 and other complexes are recorded against fourteen pathogenic bacteria viz.

| *Gram Negative* | *Gram Positive* |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | *14. Bacillus cereus* |

| | |
|---|---|
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

And the result is given in (Table 5.1-5.12) the complex 3,4,**5** showed the most acitivities above fourteen pathogenic bacteria as shown Table (Fig 5.1-5.6). It is evident from all the tables that the under investigation showed the most activity compared to the complex **2,6**

The malanodihydrazied complexes **1,2** and **6** have shown good activity against the above fourteen pathogenic bacteria as seen in (Table 5.1-5.12). The complex **1** showed the best activity against *E. Coli,* less activity against *Klebsilla.* The complex **3** showed the best activity *Salmonella-17, Shigella shiga, Shigella sonnei* and less activity against *Pseudomonas aeruginosa*. The complex **5** showed the best activity against *Bacillus cereus* and less activity against *Shigella boydii* the complex **6** are not showed good activities against the above fourteen pathogenic bacteria.

The good activity against *Bacillus cereus* and less activity *Shigella dysenteriae* and other bacteria was not seen. Similarly the complex **2** showed good activities *E.Coli* and less activity against *Bacillus megatrium* and other bacteria was not seen activities. The complex **4** showed good activities *Salmonella* and less activities against *shigella sonnei* and other bacteria was

not seen activities. The complex **6** showed good activities *Shigella dysenteriae* and less activities against *Shigella boydii, Bacillus megaterium* and other bacteria was not seen activities. All the result are compared with the standard compound, kanamycin as seen in the Table (5.1-5.12) the ligand malanodihydrazide ($C_3H_8N_4O_2$) did not show any activities against the above fourteen pathogenic bacteria.

From here it is concluded that the complex **3, 4** and **5** showed good activities against the fourteen pathogenic bacteria as compared to the standard compound, kanamycin. It is evident that the ligand malanodihydrazide did not show any activity.

## 5.13 DETERMINATION OF MINIMUM INHIBITORY CONCENTRATIONS (MIC) OF COMPLEXES

### 5.13.1 Introduction

Minimum inhibitory concentration (MIC) may be defined as the lowest concentration of antimicrobial drug to inhibit the growth of organism. The data derived from the test can be corrected with the knowledge of expected or measured antibiotic level in vivo to predict the efficacy of the sample.

There are two methods for determining the MIC are as follows.

i. Serial dilution technique or turbidimetric assay[5,6].

ii. Paper disc technique or agar diffusion assay[5].

Here "Serial dilution technique"[5,6] was followed using nutrient broth medium. The MIC values of complexes were determined against the following fourteen-test organisms.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | *14. Bacillus cereus* |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

### 5.13.2 Principle of serial tube dilution technique

The tubes of broth medium containing graded doses of sample are inoculated with the test organisms. After suitable incubation growth will occur in these inhibitory tubes were the concentration of sample is bellow the inhibitory level and the culture will become tabid (cloudy). Therefore the large number of microorganism's present growth will not occur above the inhibitory level and the tube will remain clear.

### 5.13.3 Preparation of the sample solution

2.048 mg of the test compound was taken in a vial 2 mL of methanol was added to the vial to dissolve the compound. Thus solution with a concentration of 1.024 mg/mL was obtained. The solution was added the medium on the serial dilution.

### 5.13.4 Preparation of Inoculum

Fresh culture of the test organisms was grown at $37.5^0$C for overnight on nutrient agar medium. Bacterial suspensions were then prepared in sterile saline TS and the turbidity was adjusted with saline TS to obtain the turbidity visually comparable to that of Farland 0.5 standard. The bacterial suspension was further diluted to 1:200 in Muller hinton broth. The resulting suspension contained 106 $_{CFU/mL}$.

### 5.13.5: Procedure

i. 12 test tubes were taken nine of which were marked 1, 2, 3, 4, 5, 6, 7, 8, 9 and rest three were assigned as $C_M$ (nutrient broth medium), $C_S$ (nutrient broth medium + compound), Ci [nutrient broth medium inoculum (organism)].

ii. 1 mL of nutrient broth medium was poured to each of the 12 test tubes.

iii. These test tubes were cotton plugged and sterilized in an autoclave for 15 minutes at $121^0$C temperature and 1 atm pressure.

iv. After cooling 1 mL of the sample solution was added to the 1st tube mixed well & then 1 mL of this content was transferred to the second test tube.

v. The content of the second test tube was mixed well and again 1 mL of this mixture was transferred to the 3rd tube this process of serial dilution was continued up to the 9th test tube and 1 mL mixed content was discareded from 9th test tube.

vi. 10 µL of properly diluted inoculums was added to each of the nine test tubes and mixed well.

vii. To the control test tube, $C_S$ 1 mL of the sample solution was added mixed well and 1 mL of this mixed content was discarded this is to check the clearity of the medium in presence of diluted.

viii. 10µL of the inoculums was added to the control test tube Ci to observe the growth of the organism in the medium used.

ix. The control test tubes $C_M$ containing medium only was used to confirm the sterility of the medium.

x. All the test tube was incubated at $37^0$C for 20 hours MIC is the lowest drug concentration at which there is no growth.

## 5.14 RESULT OF THE MINIMUM INHIBITORY CONCENTRATION OF THECOMPLEXES $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$, $[Pb(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ AGAINST THE FOURTEEN PATHOGENIC BACTERIA VIZ.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | *14. Bacillus cereus* |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

**Table 5.15:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Salmonella-17*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

Where

+ve = Growth

-ve = Not growth

The MIC of the complex **3** is 32 µg/mL

**Table 5.16:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Klebsilla*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.17:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella dysenteriae.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.18:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella shiga*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.19:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.20:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.21:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella flexneri.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.22:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Escherichia coli.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.23:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.24:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Salmonella.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.25:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.26:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Sarcina lutea*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.27:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Staphylococcus aureus*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.28:** Minimum inhibitory concentration of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.29:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Salmonella-17.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 5.30:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Klebsilla.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.31:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Shigella dysenteriae.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.32:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Shigella shiga.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.33:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.34:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.35:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Shigella flexneri*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.36:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] complex **5** against *Escherichia coli.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.37:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.38:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Salmonella*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.39:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.40:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Sarcina lutea*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.41:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Staphylococcus aureus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.42:** Minimum inhibitory concentration of [Pb $(C_{24}H_{28}N_8O_4)(ClO_4)_2$] Complex **5** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

## 5.43 DISCUSSION:

The minimum inhibitory concentrations (MIC) of the complexes $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ as **3,** $[Pb\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ as **5** were determined against fourteen pathogenic bacteria viz.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

***14. Bacillus cereus*** *by serial dilution technique. The results were shown in Table (5.44).*

**Table-5.44:** MIC of $[Pb(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3**
$[Pb\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **5**

| Test organism | Complex **3** | Complex **5** |
|---|---|---|
| | µg/mL | |
| *Salmonella-17* | 32 | 32 |
| *Klebsilla* | 32 | 32 |
| *Shigella dysenteriae* | 32 | 32 |
| *Shigella shiga* | 32 | 32 |
| *Shigella boydii* | 32 | 32 |
| *Shigella sonnei* | 32 | 32 |
| *Sigella flexneri* | 32 | 32 |
| *Escherichia coli* | 32 | 32 |
| *Pseudomonas aeruginosa* | 32 | 32 |
| *Salmonella* | 32 | 32 |
| *Bacillus megaterium* | 32 | 32 |
| *Sarcina lutea* | 32 | 32 |
| *Staphylococcus aureus* | 32 | 32 |
| *Bacillus cereus* | 32 | 32 |

## 6.11: RESULT OF Zn(II) CONTAIN COMPLEXES.

RESULT OF THE ANTIBACTERIAL ACTIVITY OF THE COMPLEXES (1-6) AGAINIST THE FOURTEEN PATHOGENIC BACTERIA VIZ. *SALMONELLA-17, KLEBSILLA, SHIGELLA DYSENTERIAE, SHIGELLA SHIGA, SHIGELLA BOYDII, SHIGELLA SONNEL, SHIGELLA FLEXNERI, ESCHERICHIA COLI, PSEUDOMONAS AERUGINOSA, SALMONELLA, BACILLUS MEGATERIUM, SARCINA LUTEA, STAPHYLOCOCCUS AUREUS, BACILLUS CEREUS.*

Table 6.0 Complexes abbreviation for antibacterial activity

| Complexes No. | Compound | Symbol |
|---|---|---|
| 1. | $[Zn(C_8H_{16}N_8O_4)(ClO_4)_2]$ | Zn +F+L |
| 2. | $[Zn\ (C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | Zn +A+L |
| 3. | $[Zn\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | Zn +B+L |
| 4. | $[Zn\ (\ C_{24}H_{24}N_8O_4)(ClO_4)_2]$ | Zn +Cro+L |
| 5. | $[Zn\ (C_{20}H_{28}N_8O_4)(ClO_4)_2]$ | Zn +Ci+L |
| 6. | $[Zn\ (C_{14}H_{\ 24}N_8O_4)(ClO_4)_2]$ | Zn +Banzal+L |
| Ligand | $C_3N_4H_8O_2$ | L |

Table 6.1: Antibacterial activity of the complexes 2.3,4 against *Salmonella-17.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | Zn+A+L | 20 | 10 | 6 | - |
| 3 | Zn +B+L | 17 | 8 | 3 | - |
| 4 | Zn +Cro+L | 18 | 10 | 5 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 16 |

Table 6.2: Antibacterial activity of the complexes 3.4,5 against *Klebsilla*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Zn +B+L | 18 | 8 | 4 | |
| 4 | Zn +Cro+L | 15 | 7 | 3 | |
| 5 | Zn +Ci+L | 17 | 9 | 4 | |
| | Control disc | Nil | | - | |
| | Standard disc | | | | 19 |

Table 6.3: Antibacterial activity of the complexes 4, 5, 6 against *Shigella dysenteriae.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 4 | Zn +Cro+L | 12 | 6 | 3 | - |

| | | | | | |
|---|---|---|---|---|---|
| 5 | Zn +Ci+L | 10 | 5 | 2 | - |
| 6 | Zn +Banzal+L | 6 | 3 | 2 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 21 |

Table 6.4: Antibacterial activity of the complexes 3.4.5 against *Shigella shiga*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Zn +B+L | 18 | 10 | 4 | - |
| 4 | Zn +Cro+L | 20 | 10 | 5 | - |
| 5 | Zn +Ci+L | 15 | 8 | 4 | - |
| | Control disc | Nil | | | - |
| | Standard disc | | | | 21 |

Table 6.5: Antibacterial activity of the complexes 3, 4, 5 against *Shigella boydii*.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Zn +B+L | 18 | 10 | 5 | - |
| 4 | Zn +Cro+L | 15 | 8 | 4 | - |
| 5 | Zn +Ci+L | 14 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 6.6: Antibacterial activity of the complexes 3, 4, 5 against *Shigella sonnei.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Zn +B+L | 18 | 10 | 5 | - |
| 4 | Zn +Cro+L | 12 | 6 | 3 | - |
| 5 | Zn +Ci+L | 16 | 8 | 4 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 6.7: Antibacterial activity of the complexes 1, 2, 3 against *Escherichia coli.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1. | Zn +F+L | 26 | 12 | 5 | - |
| 2. | Zn +A+L | 20 | 10 | 4 | - |
| 3 | Zn +B+L | 15 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 6.8: Antibacterial activity of the complexes 2, 3, 4 against *Salmonella.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | Zn +A+L | 10 | 8 | 2 | - |
| 3 | Zn +B+L | 18 | 14 | 4 | - |
| 4 | Zn +Cro+L | 18 | 9 | 3 | - |
| | Control disc | Nil | | - | - |

| | | | | | |
|---|---|---|---|---|---|
| | Standard disc | | | | 20 |

Table 6.9: Antibacterial activity of the complexes 2, 3, 4 against *Bacillus megaterium.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | Zn +A+L | 20 | 10 | 5 | - |
| 3 | Zn +B+L | 18 | 9 | 4 | - |
| 4 | Zn +Cro+L | 16 | 8 | 3 | - |
| | Standard disc | | | | 18 |

Table 6.10: Antibacterial activity of the complexes 2,3,4 against *Sarcina lutea.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | Zn +A+L | 20 | 12 | 6 | - |
| 3 | Zn +B+L | 18 | 9 | 4 | - |
| 4 | Zn +Cro+L | 16 | 7 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 18 |

Table 6.11: Antibacterial activity of the complexes 3, 4, 5 against *Staphylococcus aureus.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) |
|---|---|---|

| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
|---|---|---|---|---|---|
| 3 | Zn +B+L | 28 | 14 | 6 | |
| 4 | Zn +Cro+L | 20 | 10 | 4 | |
| 5 | Zn +Ci+L | 18 | 9 | 3 | |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 18 |

Table 6.12: Antibacterial activity of the complexes 3, 4, 5 against *Bacillus cereus*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Zn +B+L | 18 | 10 | 5 | - |
| 4 | Zn +Cro+L | 12 | 6 | 3 | - |
| 5 | Zn +Ci+L | 16 | 8 | 4 | - |
| | Control disc | Nil | - | - | |
| | Standard disc | | | | 20 |

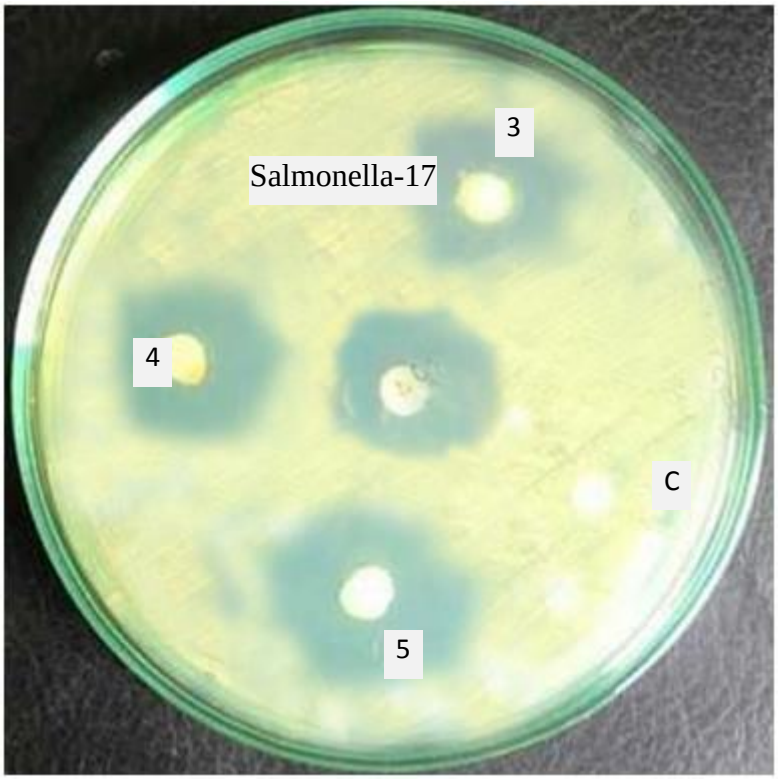


Fig: 6.1: Photographic representation of zone of inhibition of the complexes 3, 4, 5 the standard compound kanamycin against Salmonella-17.

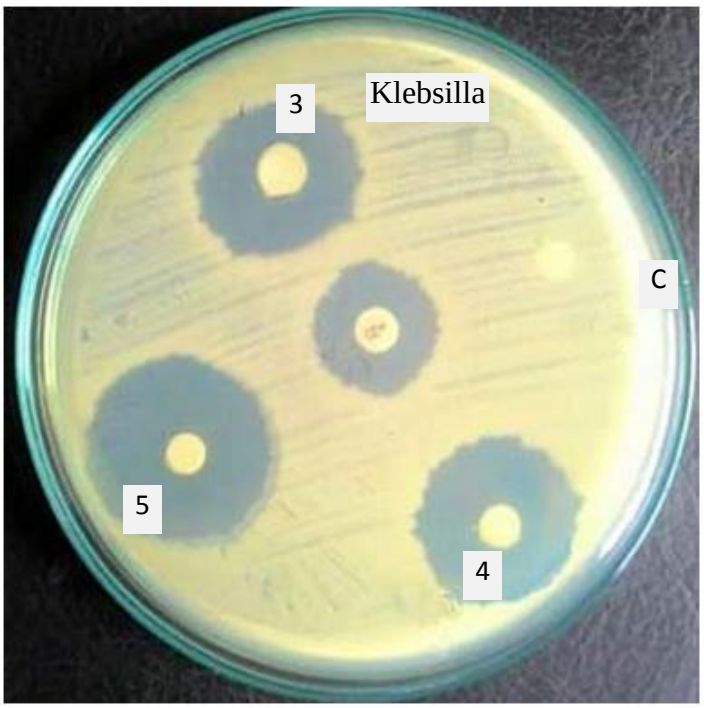

Fig: 6.2: Photographic representation of zone of inhibition of the complexes 3, 4, 5 the standard compound kanamycin against Klebsilla.

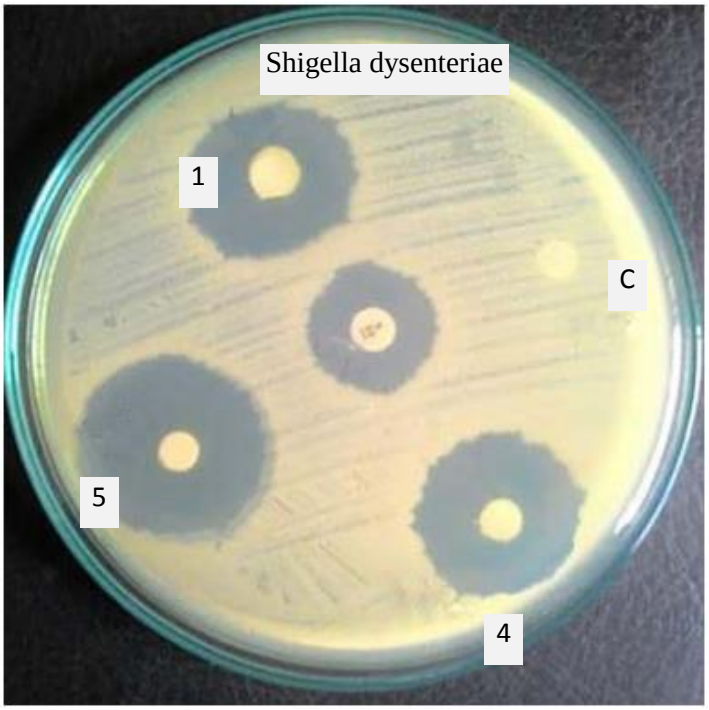


Fig: 6.3: Photographic representation of zone of inhibition of the complexes 1, 4, 5 the standard compound kanamycin against Shigella dysenteriae.

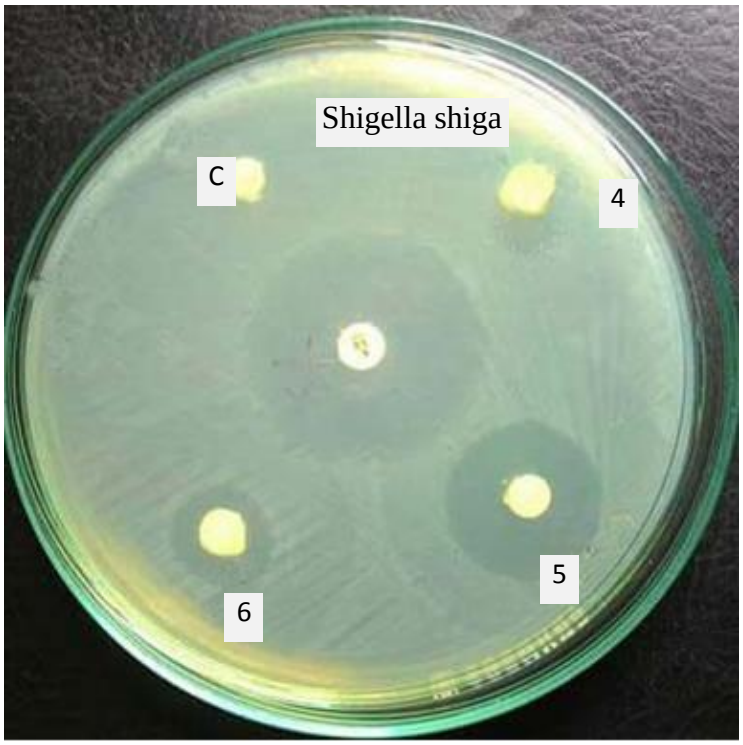


Fig: 6.4: Photographic representation of zone of inhibition of the complexes 4, 5, 6 the standard compound kanamycin against Shigella shiga.

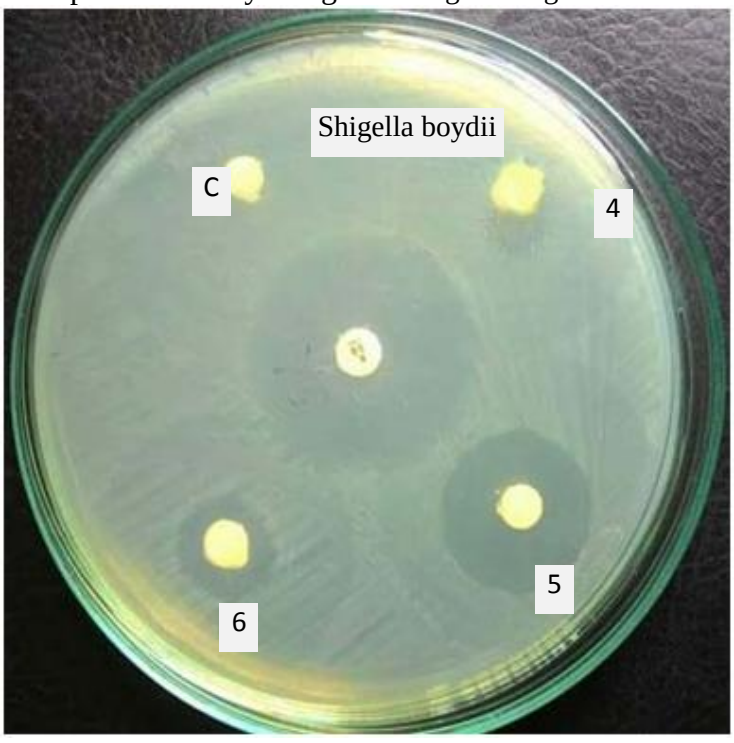


Fig: 6.5: Photographic representation of zone of inhibition of the complexes 4,5,6 the standard compound kanamycin against Shigella boydii.

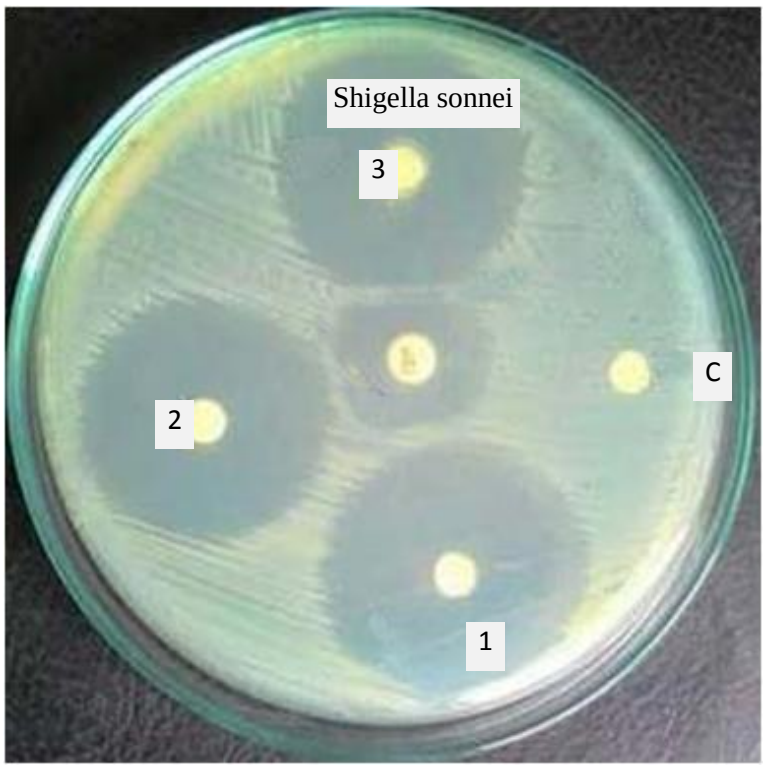


Fig: 6.6: Photographic representation of zone of inhibition of the complexes1, 2, 3 the standard compound kanamycin against Shigella sonnei.

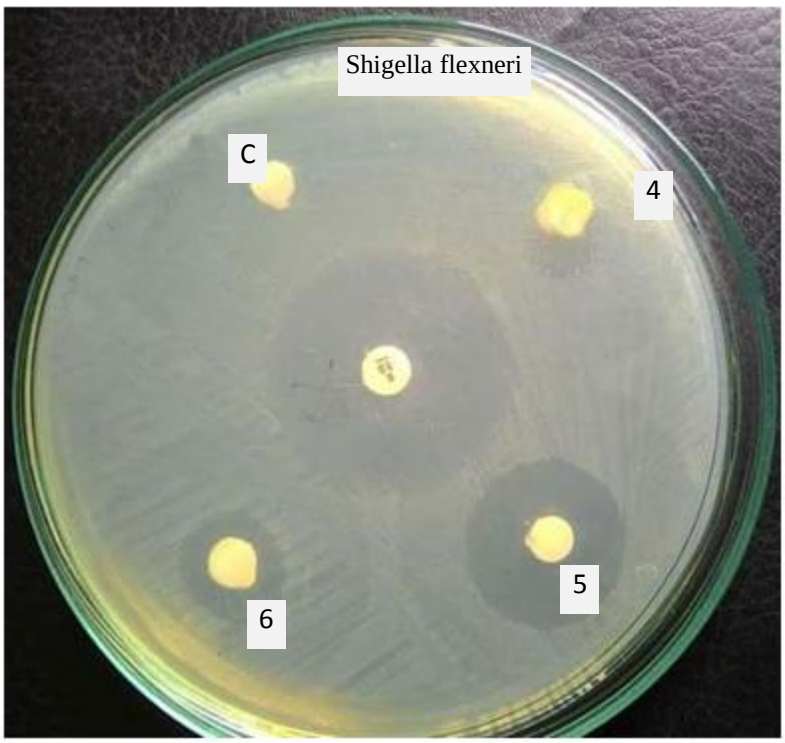

Fig: 6.7: Photographic representation of zone of inhibition of the complexes 4, 5, 6 the standard compound kanamycin against Shigella flexneri.

## 6.12 DISCUSSION

It has been observed that some drug increases the activity when administered as metal complexes or their metal chalets[4]. The antibacterial activity of the metal complexes 3, 4, 5 and other complexes are recorded against fourteen pathogenic bacteria viz.

| *Gram Negative* | *Gram Positive* |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | *14. Bacillus cereus* |

| | |
|---|---|
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

And the result is given in (Table 5.1-5.12) the complex 3,4,**5** showed the most acitivities above fourteen pathogenic bacteria as shown Table (Fig 5.1-5.6). It is evident from all the tables that the under investigation showed the most activity compared to the complex **2, 6**

The malanodihydrazied complexes **1,2** and **6** have shown good activity against the above fourteen pathogenic bacteria as seen in (Table 5.1-5.12). The complex **1** showed the best activity against *E. Coli,* less activity against *Klebsilla.* The complex **3** showed the best activity *Salmonella-17, Shigella shiga, Shigella sonnei* and less activity against *Pseudomonas aeruginosa*. The complex **5** showed the best activity against *Bacillus cereus* and less activity against *Shigella boydii* the complex **6** are not showed good activities against the above fourteen pathogenic bacteria.

The good activity against *Bacillus cereus* and less activity *Shigella dysenteriae* and other bacteria was not seen. Similarly the complex **2** showed good activities *E.Coli* and less activity against *Bacillus megatrium* and other bacteria was not seen activities. The complex **4** showed good activities *Salmonella* and less activities against *shigella sonnei* and other bacteria was

not seen activities. The complex **6** showed good activities *Shigella dysenteriae* and less activities against *Shigella boydii, Bacillus megaterium* and other bacteria were not seen activities. All the result are compared with the standard compound, kanamycin as seen in the Table (5.1-5.12) the ligand malanodihydrazide ($C_3H_8N_4O_2$) did not show any activities against the above fourteen pathogenic bacteria.

From here it is concluded that the complex **3, 4** and **5** showed good activities against the fourteen pathogenic bacteria as compared to the standard compound, kanamycin. It is evident that the ligand malanodihydrazide did not show any activity.

**6.14** RESULT OF THE MINIMUM INHIBITORY CONCENTRATION OF THE COMPLEXES $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$, $[Zn(C_{20}H_{28}N_8O_4)(ClO_4)_2]$ AGAINST THE FOURTEEN PATHOGENIC BACTERIA VIZ.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | *14. Bacillus cereus* |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

**Table 6.15:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Salmonella-17.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

Where

+ve = Growth

-ve = Not growth

The MIC of the complex **3** is 32 µg/mL

**Table 6.16:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Klebsilla*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.17:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella dysenteriae.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.18:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella shiga*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.19:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.20:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.21:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella flexneri*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.22:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Escherichia coli.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.23:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.24:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Salmonella.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.25:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.26:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Sarcina lutea*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.27:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Staphylococcus aureus*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.28:** Minimum inhibitory concentration of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.29:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **5** against *Salmonella-17*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 6.30:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Klebsilla.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.31:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Shigella dysenteriae.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.32:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Shigella shiga.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.33:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.34:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.35:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Shigella flexneri*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.36:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Escherichia coli.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.37:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.38:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Salmonella*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.39:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.40:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Sarcina lutea*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 5.41:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Staphylococcus aureus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**Table 6.42:** Minimum inhibitory concentration of $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 32 µg/mL

**6.15 DISCUSSION:**

The minimum inhibitory concentrations (MIC) of the complexes $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ as 3**,** $[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ as **5** were determined against fourteen pathogenic bacteria viz.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

***14. Bacillus cereus*** *by serial dilution technique. The results were shown in Table (5.43).*

**Table-6.43:** MIC of $[Zn(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex 3
$[Zn(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **5**

| Test organism | Complex 3 | Complex **5** |
|---|---|---|
| | µg/mL | |
| *Salmonella-17* | 32 | 32 |
| *Klebsilla* | 32 | 32 |
| *Shigella dysenteriae* | 32 | 32 |
| *Shigella shiga* | 32 | 32 |
| *Shigella boydii* | 32 | 32 |
| *Shigella sonnei* | 32 | 32 |
| *Sigella flexneri* | 32 | 32 |
| *Escherichia coli* | 32 | 32 |
| *Pseudomonas aeruginosa* | 32 | 32 |
| *Salmonella* | 32 | 32 |
| *Bacillus megaterium* | 32 | 32 |
| *Sarcina lutea* | 32 | 32 |
| *Staphylococcus aureus* | 32 | 32 |
| *Bacillus cereus* | 32 | 32 |

## 7.11: RESULT OF La(II) CONTAIN COMPLEXES.

RESULT OF THE ANTIBACTERIAL ACTIVITY OF THE COMPLEXES (1-6) AGAINIST THE FOURTEEN PATHOGENIC BACTERIA VIZ. *SALMONELLA-17, KLEBSILLA, SHIGELLA DYSENTERIAE, SHIGELLA SHIGA, SHIGELLA BOYDII, SHIGELLA SONNEL, SHIGELLA FLEXNERI, ESCHERICHIA COLI, PSEUDOMONAS AERUGINOSA, SALMONELLA, BACILLUS MEGATERIUM, SARCINA LUTEA, STAPHYLOCOCCUS AUREUS, BACILLUS CEREUS.*

Table 7.0 Complexes abbreviation for antibacterial activity

| Complexes No. | Compound | Symbol |
|---|---|---|
| 1. | $[La(C_8H_{16}N_8O_4)(ClO_4)_2]$ | La +F+L |
| 2. | $[La(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | La +A+L |
| 3. | $[La\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | La +B+L |
| 4. | $[La(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | La +Cro+L |
| 5. | $[La\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | La +Ci+L |
| 6. | $[La\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | La +Banzal+L |
| Ligand | $C_3N_4H_8O_2$ | L |

Table 7.1: Antibacterial activity of the complexes 2.3,4 against *Salmonella-17.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | La +A+L | 20 | 10 | 6 | - |
| 3 | La +B+L | 17 | 8 | 3 | - |
| 4 | La +Cro+L | 18 | 10 | 5 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 16 |

Table 7.2: Antibacterial activity of the complexes 3.4,5 against *Klebsilla*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | La +B+L | 18 | 8 | 4 | |
| 4 | La +Cro+L | 15 | 7 | 3 | |
| 5 | La +Ci+L | 17 | 9 | 4 | |
| | Control disc | Nil | | - | |
| | Standard disc | | | | 19 |

Table 7.3: Antibacterial activity of the complexes 4, 5, 6 against *Shigella dysenteriae.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 4 | La +Cro+L | 12 | 6 | 3 | - |
| 5 | La +Ci+L | 10 | 5 | 2 | - |

| | | | | | |
|---|---|---|---|---|---|
| 6 | La +Banzal+L | 6 | 3 | 2 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 21 |

Table 7.4: Antibacterial activity of the complexes 3, 4, 5 against *Shigella shiga*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | La +B+L | 18 | 10 | 4 | - |
| 4 | La +Cro+L | 20 | 10 | 5 | - |
| 5 | La +Ci+L | 15 | 8 | 4 | - |
| | Control disc | Nil | | | - |
| | Standard disc | | | | 21 |

Table 7.5: Antibacterial activity of the complexes 3, 4, 5 against *Shigella boydii*.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | La +B+L | 18 | 10 | 5 | - |
| 4 | La +Cro+L | 15 | 8 | 4 | - |
| 5 | La +Ci+L | 14 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 7.6: Antibacterial activity of the complexes 3, 4, 5 against *Shigella sonnei*.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | La +B+L | 18 | 10 | 5 | - |
| 4 | La +Cro+L | 12 | 6 | 3 | - |
| 5 | La +Ci+L | 16 | 8 | 4 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 7.7: Antibacterial activity of the complexes 1, 2,3 against *Escherichia coli.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1. | La +F+L | 26 | 12 | 5 | - |
| 2. | La +A+L | 20 | 10 | 4 | - |
| 3 | La +B+L | 15 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 7.8: Antibacterial activity of the complexes 2, 3, 4 against *Salmonella.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | La +A+L | 10 | 8 | 2 | - |
| 3 | La +B+L | 18 | 14 | 4 | - |
| 4 | La +Cro+L | 18 | 9 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 7.9: Antibacterial activity of the complexes 2, 3, 4 against *Bacillus megaterium.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | La +A+L | 20 | 10 | 5 | - |
| 3 | La +B+L | 18 | 9 | 4 | - |
| 4 | La +Cro+L | 16 | 8 | 3 | - |
| | Standard disc | | | | 18 |

Table 7.10: Antibacterial activity of the complexes 2, 3, 4 against *Sarcina lutea.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | La +A+L | 20 | 12 | 6 | - |
| 3 | La +B+L | 18 | 9 | 4 | - |
| 4 | La +Cro+L | 16 | 7 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 18 |

Table 7.11: Antibacterial activity of the complexes 3, 4, 5 against *Staphylococcus aureus.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | La +B+L | 28 | 14 | 6 | - |
| 4 | La +Cro+L | 20 | 10 | 4 | - |
| 5 | La +Ci+L | 18 | 9 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 18 |

Table 7.12: Antibacterial activity of the complexes 3, 4, 5 against *Bacillus cereus*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | La +B+L | 18 | 10 | 5 | - |

| | | | | | |
|---|---|---|---|---|---|
| 4 | La +Cro+L | 12 | 6 | 3 | - |
| 5 | La +Ci+L | 16 | 8 | 4 | - |
| | Control disc | Nil | - | - | |
| | Standard disc | | | | 20 |

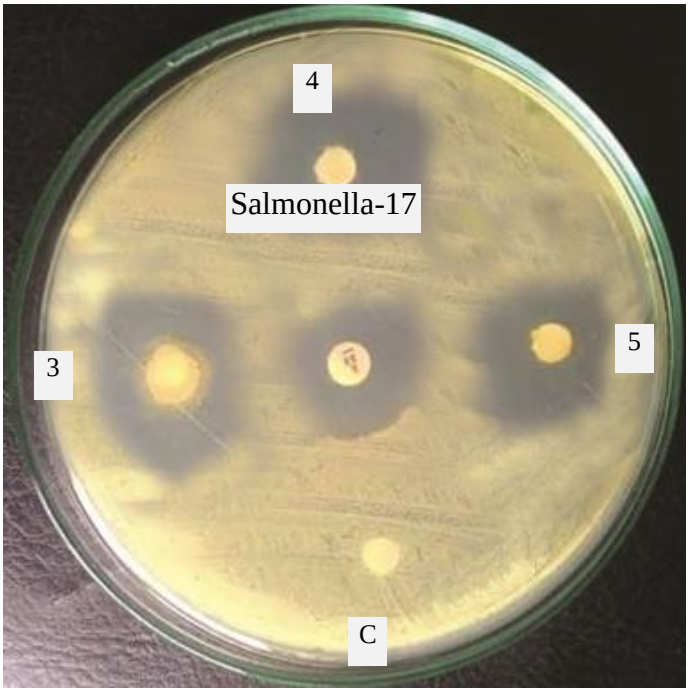


Fig: 7.1: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycin against Salmonella-17.

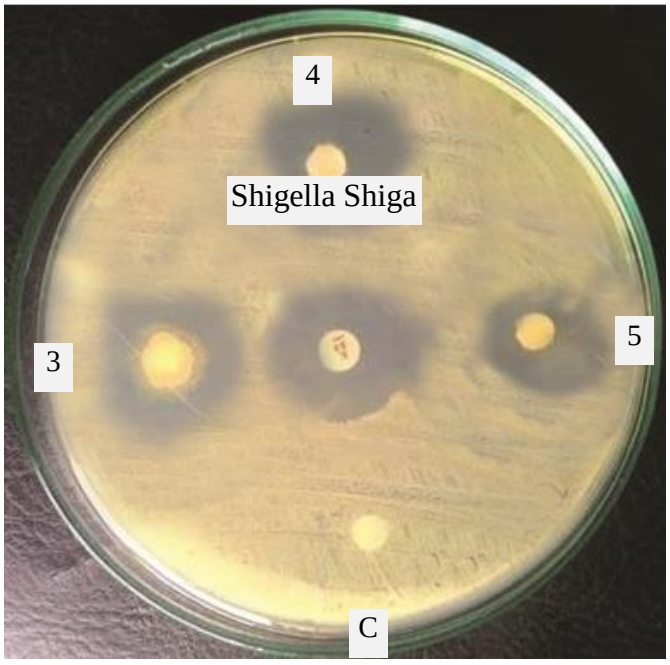


Fig: 7.2: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycin against Shigella Shiga.

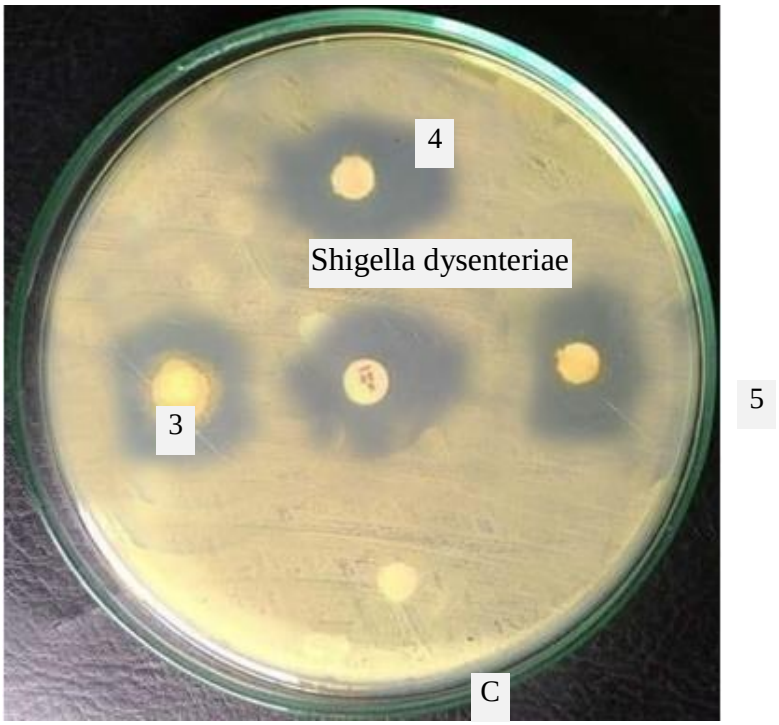


Fig: 7.3: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycin against Shigella dysenteriae.

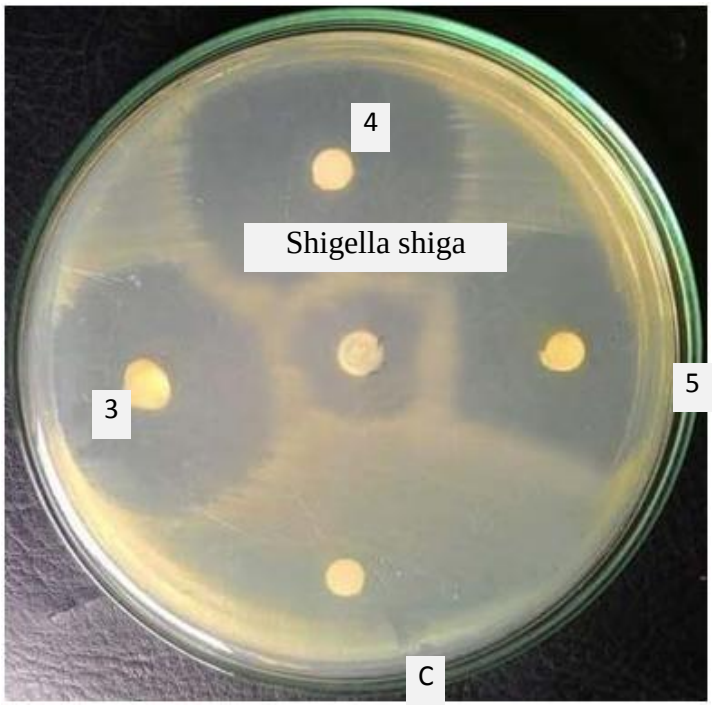


Fig: 7.4: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycin against Shigella shiga.

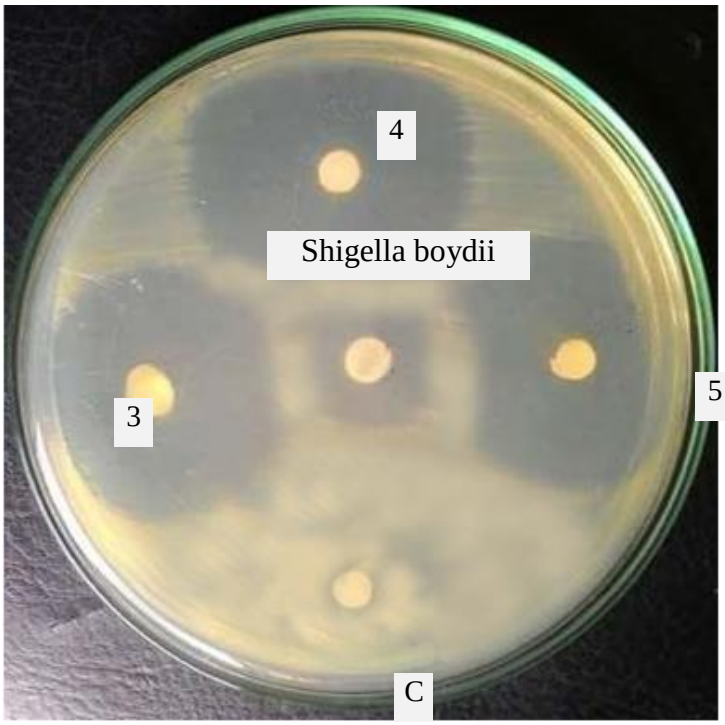


Fig: 7.5: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycinagain

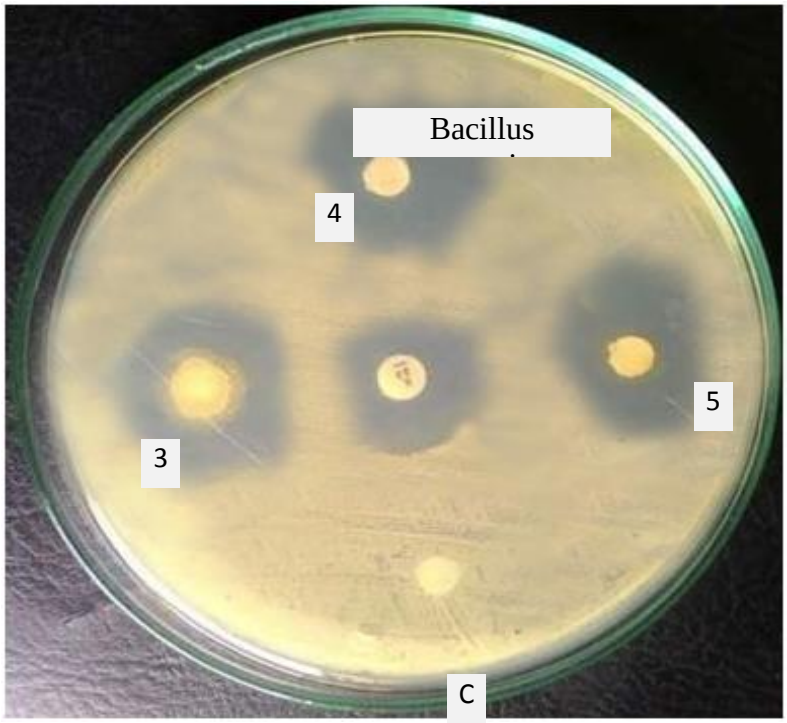


Fig: 7.6: Photographic representation of zone of inhibition of the complexes 3,4,5 the standard compound kanamycin against Bacillus megaterium.

## 7.12 DISCUSSION

It has been observed that some drug increases the activity when administered as metal complexes or their metal chalets[4]. The antibacterial activity of the metal complexes 3, 4, 5 and other complexes are recorded against fourteen pathogenic bacteria viz.

| *Gram Negative* | *Gram Positive* |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | *14. Bacillus cereus* |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

And the result is given in (Table 5.1-5.12) the complex 3, 4, **5** showed the most acitivities above fourteen pathogenic bacteria as shown Table (Fig 5.1-5.6). It is evident from all the tables that the under investigation showed the most activity compared to the complex **2, 6**

The malanodihydrazied complexes **1, 2** and **6** have shown good activity against the above fourteen pathogenic bacteria as seen in (Table 5.1-5.12).

The complex **1** showed the best activity against *E. Coli*, less activity against *Klebsilla*. The complex **3** showed the best activity *Salmonella-17, Shigella shiga, Shigella sonnei* and less activity against *Pseudomonas aeruginosa*. The complex **5** showed the best activity against *Bacillus cereus* and less activity against *Shigella boydii* the complex **6** are not showed good activities against the above fourteen pathogenic bacteria.

The good activity against *Bacillus cereus* and less activity *Shigella dysenteriae* and other bacteria was not seen. Similarly the complex **2** showed good activities *E.Coli* and less activity against *Bacillus megatrium* and other bacteria was not seen activities. The complex **4** showed good activities *Salmonella* and less activity against *shigella sonnei* and other bacteria were not seen activities. The complex **6** showed good activities *Shigella dysenteriae* and less activity against *Shigella boydii, Bacillus megaterium* and other bacteria was a not seen activity. All the result are compared with the standard compound, kanamycin as seen in the Table (5.1-5.12) the ligand malanodihydrazide ($C_3H_8N_4O_2$) did not show any activities against the above fourteen pathogenic bacteria.

From here it is concluded that the complex **3, 4** and **5** showed good activities against the fourteen pathogenic bacteria as compared to the standard compound, kanamycin. It is evident that the ligand malanodihydrazide did not show any activity.

**7.14** RESULT OF THE MINIMUM INHIBITORY CONCENTRATION OF THE COMPLEXES $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$, $[La(C_{20}H_{28}N_8O_4)(ClO_4)_2]$ AGAINST THE FOURTEEN PATHOGENIC BACTERIA VIZ.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | *14. Bacillus cereus* |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

**Table 7.15:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Salmonella-17.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

Where

+ve = Growth

-ve = Not growth

The MIC of the complex **3** is 64 µg/mL

**Table 7.16:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Klebsilla*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.17:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella dysenteriae.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.18:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella shiga*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 7.19:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 8.20:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.21:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella flexneri*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.22:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Escherichia coli.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.23:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.24:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Salmonella.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.25:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.26:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Sarcina lutea*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.27:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Staphylococcus aureus*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.28:** Minimum inhibitory concentration of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 64 µg/mL

**Table 7.29:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Salmonella-17.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 128 µg/mL

**Table 7.30:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Klebsilla.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.31:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **5** against *Shigella dysenteriae.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.32:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **5** against *Shigella shiga*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.33:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **5** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.34:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.35:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Shigella flexneri*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.36:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **5** against *Escherichia coli*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.37:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.38:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Salmonella*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.39:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.40:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **5** against *Sarcina lutea*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.41:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Staphylococcus aureus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 7.42:** Minimum inhibitory concentration of $[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ Complex **5** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**7.15 DISCUSSION:**

The minimum inhibitory concentrations (MIC) of the complexes [La ($C_{14}H_{28}N_8O_4$)($ClO_4$)$_2$] as **3,[**La ($C_{24}H_{28}N_8O_4$)($ClO_4$)$_2$] as **5** were determined against fourteen pathogenic bacteria viz.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

***14. Bacillus cereus*** *by serial dilution technique. The results were shown in Table (5.43).*

**Table-7.43:** MIC of $[La(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3**
$[La(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **5**

| Test organism | Complex **3** | Complex **5** |
|---|---|---|
| | µg/Ml | |
| *Salmonella-17* | 64 | 64 |
| *Klebsilla* | 64 | 64 |
| *Shigella dysenteriae* | 64 | 64 |
| *Shigella shiga* | 64 | 64 |
| *Shigella boydii* | 64 | 64 |
| *Shigella sonnei* | 64 | 64 |
| *Sigella flexneri* | 64 | 128 |
| *Escherichia coli* | 64 | 64 |
| *Pseudomonas aeruginosa* | 64 | 64 |
| *Salmonella* | 64 | 64 |
| *Bacillus megaterium* | 64 | 64 |
| *Sarcina lutea* | 64 | 64 |
| *Staphylococcus aureus* | 64 | 32 |
| *Bacillus cereus* | 64 | 64 |

## 8.11: RESULT OF Cd(II) CONTAIN COMPLEXES.

RESULT OF THE ANTIBACTERIAL ACTIVITY OF THE COMPLEXES (1-6) AGAINIST THE FOURTEEN PATHOGENIC BACTERIA VIZ. *SALMONELLA-17, KLEBSILLA, SHIGELLA DYSENTERIAE, SHIGELLA SHIGA, SHIGELLA BOYDII, SHIGELLA SONNEL, SHIGELLA FLEXNERI, ESCHERICHIA COLI, PSEUDOMONAS AERUGINOSA, SALMONELLA, BACILLUS MEGATERIUM, SARCINA LUTEA, STAPHYLOCOCCUS AUREUS, BACILLUS CEREUS.*

Table 8.0 Complexes abbreviation for antibacterial activity

| Complexes No. | Compound | Symbol |
|---|---|---|
| 1. | $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ | La +F+L |
| 2. | $[Cd\ (C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | La +A+L |
| 3. | $[Cd\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | La +B+L |
| 4. | $[Cd\ (\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | La +Cro+L |
| 5. | $[Cd\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | La +Ci+L |
| 6. | $[Cd\ (C_{20}H_{\ 24}N_8O_4)(ClO_4)_2]$ | La +Banzal+L |
| Ligand | $C_3N_4H_8O_2$ | L |

Table 8.1: Antibacterial activity of the complexes 2, 3, 4 against *Salmonella-17.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 20 | 10 | 6 | - |
| 2 | Cd +A+L | 17 | 8 | 3 | - |
| 6 | Cd +Banzal+L | 18 | 10 | 5 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 16 |

Table 8.2: Antibacterial activity of the complexes 3, 4, 5 against *Klebsilla*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 18 | 8 | 4 | - |
| 2 | Cd +A+L | 15 | 7 | 3 | - |
| 6 | Cd +Banzal+L | 17 | 9 | 4 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 19 |

Table 8.3: Antibacterial activity of the complexes 4, 5, 6 against *Shigella dysenteriae.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 12 | 6 | 3 | - |

| | | | | | |
|---|---|---|---|---|---|
| 2 | Cd +A+L | 10 | 5 | 2 | - |
| 6 | Cd +Banzal+L | 6 | 3 | 2 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 21 |

Table 8.4: Antibacterial activity of the complexes 2, 3, 5 against *Shigella shiga*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 18 | 10 | 4 | - |
| 2 | Cd +A+L | 20 | 10 | 5 | - |
| 6 | Cd +Banzal+L | 15 | 8 | 4 | - |
| | Control disc | Nil | | | - |
| | Standard disc | | | | 21 |

Table 8.5: Antibacterial activity of the complexes 3,4,5 against *Shigella boydii*.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 18 | 10 | 5 | - |
| 2 | Cd +A+L | 15 | 8 | 4 | - |
| 6 | Cd +Banzal+L | 14 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 8.6: Antibacterial activity of the complexes 3, 4,5 against *Shigella sonnei.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 18 | 10 | 5 | - |
| 2 | Cd +A+L | 12 | 6 | 3 | - |
| 6 | Cd +Banzal+L | 16 | 8 | 4 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 8.7: Antibacterial activity of the complexes 1, 2, 3 against *Escherichia coli.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 26 | 12 | 5 | - |
| 2 | Cd +A+L | 20 | 10 | 4 | - |
| 3 | Cd +B +L | 15 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 8.8: Antibacterial activity of the complexes 2,3,4 against *Salmonella.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 2 | Cd +A+L | 10 | 8 | 2 | - |
| 3 | Cd +B+L | 18 | 14 | 4 | - |
| 4 | Cd +Cro +L | 18 | 9 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 8.9: Antibacterial activity of the complexes 1, 2, 6 against *Salmonella.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 10 | 8 | 2 | - |
| 2 | Cd +A+L | 18 | 14 | 4 | - |
| 6 | Cd +Banzal+L | 18 | 9 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 8.10: Antibacterial activity of the complexes 1, 2, 6 against *Sarcina lutea.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 20 | 12 | 6 | - |
| 2 | Cd +A+L | 18 | 9 | 4 | - |
| 6 | Cd +Banzal+L | 16 | 7 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 18 |

Table 8.11: Antibacterial activity of the complexes 1, 2, 6 against *Staphylococcus aureus.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Cd +F+L | 28 | 14 | 6 | |
| 2 | Cd +A+L | 20 | 10 | 4 | |
| 6 | Cd +Banzal+L | 18 | 9 | 3 | |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 18 |

Table 8.12: Antibacterial activity of the complexes 3, 4, 5 against *Bacillus cereus*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 3 | Cd +F+L | 18 | 10 | 5 | - |
| 4 | Cd +A+L | 12 | 6 | 3 | - |

| | | | | | |
|---|---|---|---|---|---|
| 5 | Cd +Banzal+L | 16 | 8 | 4 | - |
| | Control disc | Nil | - | - | |
| | Standard disc | | | | 20 |

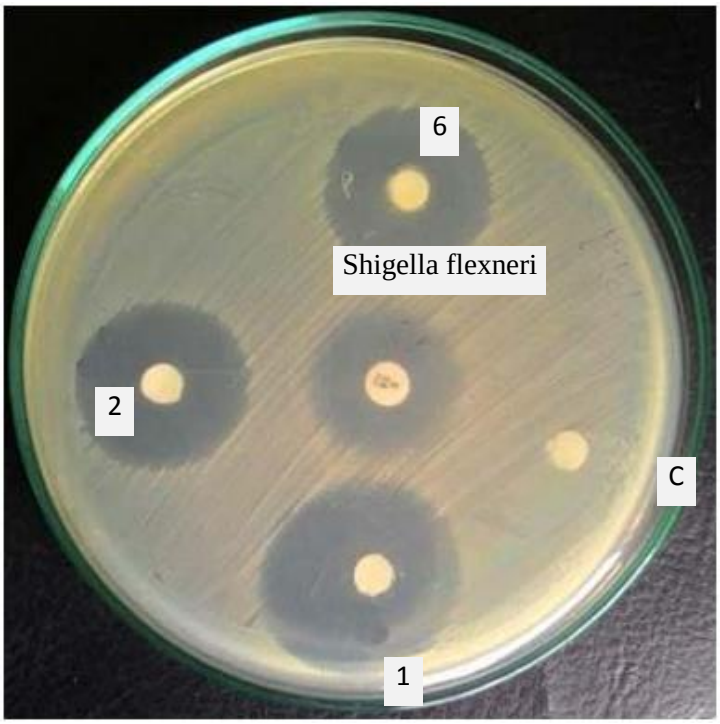


Fig: 8.1: Photographic representation of zone of inhibition of the complexes 1,2, 6 the standard compound kanamycin against Shigella flexneri.

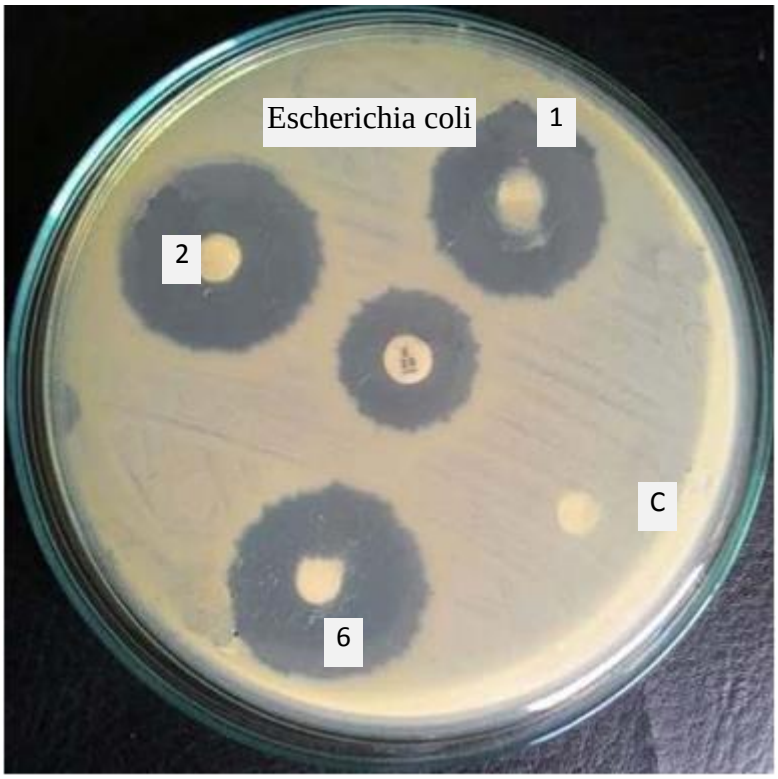


Fig: 8.2: Photographic representation of zone of inhibition of the complexes 1,2, 6 the standard compound kanamycin against Escherichia coli.

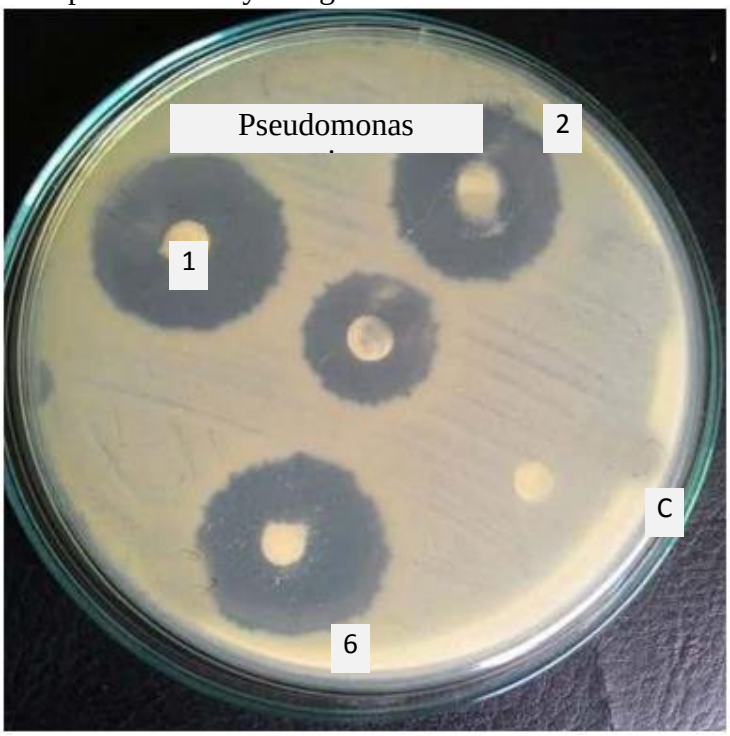

Fig: 8.3: Photographic representation of zone of inhibition of the complexes 1, 2, 6 the standard compound kanamycin against Pseudomonas aeruginosa.

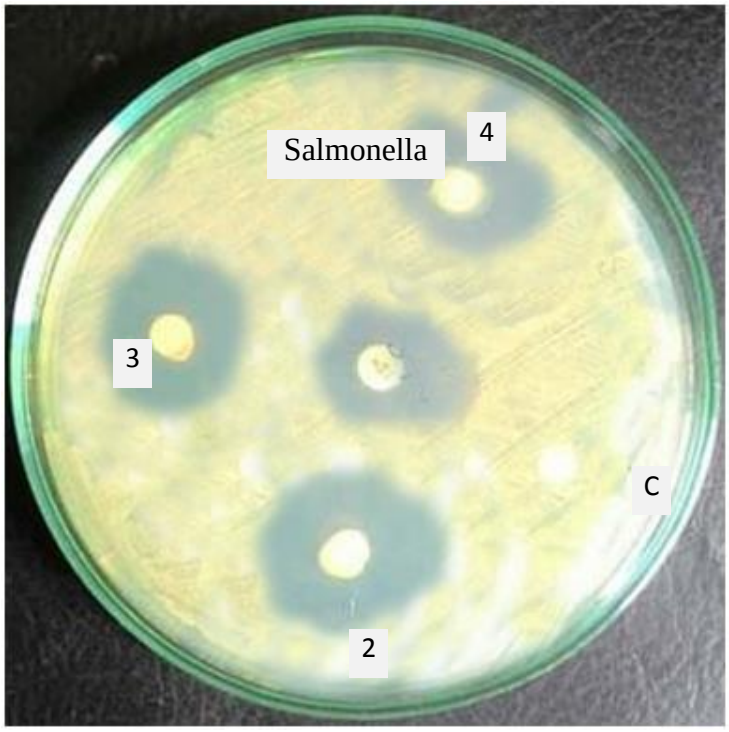


Fig: 8.4: Photographic representation of zone of inhibition of the complexes 2, 3, 4 the standard compound kanamycin against Salmonella.

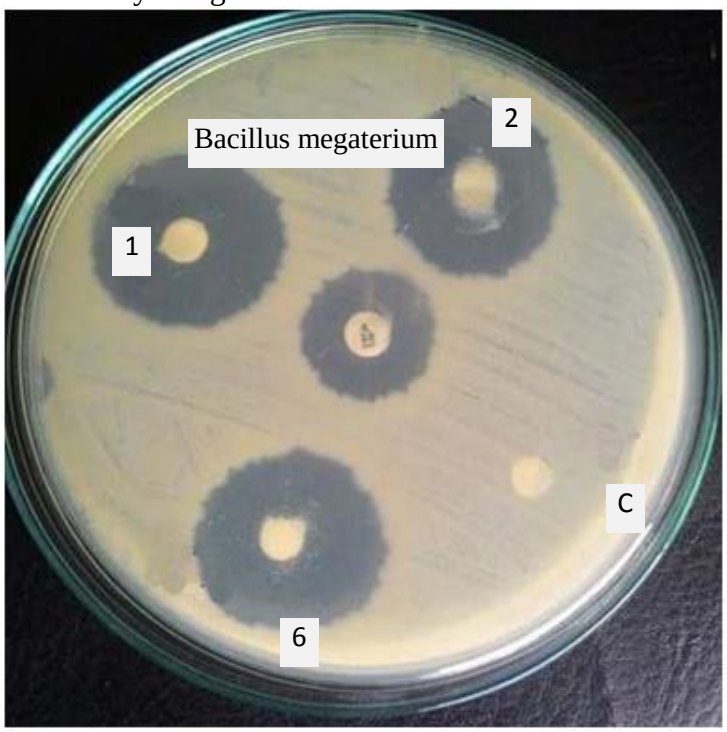

Fig: 8.5: Photographic representation of zone of inhibition of the complexes 1, 2, 6 the standard compound kanamycin against Bacillus megaterium.

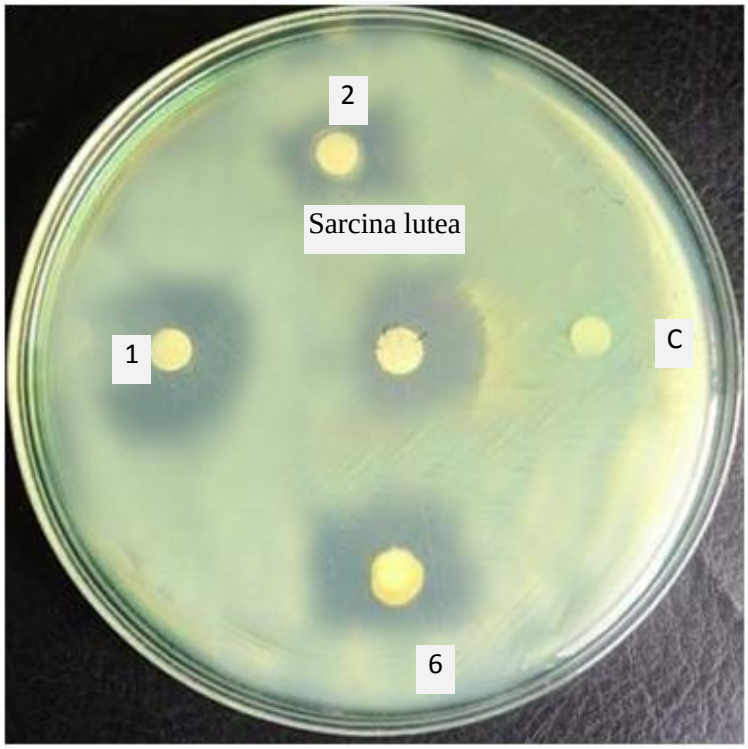


Fig: 8.6: Photographic representation of zone of inhibition of the complexes 1, 2, 6 the standard compound kanamycin against Sarcina lutea.

## 8.12 DISCUSSION

It has been observed that some drug increases the activity when administered as metal complexes or their metal chalets[4]. The antibacterial activity of the metal complexes 3, 4, 5 and other complexes are recorded against fourteen pathogenic bacteria viz.

| *Gram Negative* | *Gram Positive* |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |

| | |
|---|---|
| *4. Shigella shiga* | *14. Bacillus cereus* |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

And the result is given in (Table 5.1-5.12) the complex 3,4,**5** showed the most acitivities above fourteen pathogenic bacteria as shown Table (Fig 5.1-5.6). It is evident from all the tables that the under investigation showed the most activity compared to the complex **2, 6.**

The malanodihydrazied complexes **1, 2** and **6** have shown good activity against the above fourteen pathogenic bacteria as seen in (Table 5.1-5.12). The complex **1** showed the best activity against *E. Coli,* less activity against *Klebsilla.* The complex **3** showed the best activity *Salmonella-17, Shigella shiga, Shigella sonnei* and less activity against *Pseudomonas aeruginosa*. The complex **5** showed the best activity against *Bacillus cereus* and less activity against *Shigella boydii* the complex **6** are not showed good activities against the above fourteen pathogenic bacteria.

The good activity against *Bacillus cereus* and less activity *Shigella dysenteriae* and other bacteria was not seen. Similarly the complex **2** showed good activities *E.Coli* and less activity against *Bacillus megatrium* and other bacteria was not seen activities. The complex **4** showed good activities

*Salmonella* and less activities against *shigella sonnei* and other bacteria was not seen activities. The complex **6** showed good activities *Shigella dysenteriae* and less activities against *Shigella boydii*, *Bacillus megaterium* and other bacteria was not seen activities. All the result are compared with the standard compound, kanamycin as seen in the Table (5.1-5.12) the ligand malanodihydrazide ($C_3H_8N_4O_2$) did not show any activities against the above fourteen pathogenic bacteria.

From here it is concluded that the complex **3, 4** and **5** showed good activities against the fourteen pathogenic bacteria as compared to the standard compound, kanamycin. It is evident that the ligand malanodihydrazide did not show any activity.

**8.14** RESULT OF THE MINIMUM INHIBITORY CONCENTRATION OF THE COMPLEXES $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$, $[Cd(C_{20}H_{28}N_8O_4)(ClO_4)_2]$ AGAINST THE FOURTEEN PATHOGENIC BACTERIA VIZ.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | *14. Bacillus cereus* |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

**Table 8.15:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Salmonella-17.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

Where

+ve = Growth

-ve = Not growth

The MIC of the complex **1** is 64 µg/mL

**Table 8.16:** Minimum ihibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **3** against *Klebsilla*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.17:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Shigella dysenteriae.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 8.18:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Shigella shiga*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.19:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 128 µg/mL

**Table 8.20:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.21:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Shigella flexneri*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.22:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Escherichia coli.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.23:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.24:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Salmonella.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.25:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.26:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Sarcina lutea*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.27:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Staphylococcus aureus*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.28:** Minimum inhibitory concentration of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 64 µg/mL

**Table 8.29:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] Complex **6** against *Salmonella-17*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.30:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] Complex **6** against *Klebsilla.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.31:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] Complex **6** against *Shigella dysenteriae.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.32:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] Complex **6** against *Shigella shiga.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.33:** Minimum inhibitory concentration of $[Cd(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex **6** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.34:** Minimum inhibitory concentration of $[Cd(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex **6** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.35:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] Complex **6** against *Shigella flexneri*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.36:** Minimum inhibitory concentration of $[Cd(C_{20}H_{24}N_8O_4)(ClO_4)_2]$ complex **6** against *Escherichia coli.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.37:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] complex **6** against *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **5** is 64 µg/mL

**Table 8.38:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] complex **6** against *Salmonella*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.39:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] complex **6** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.40:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] complex **6** against *Sarcina lutea.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.41:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] complex **6** against *Staphylococcus aureus*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

**Table 8.42:** Minimum inhibitory concentration of [Cd ($C_{20}H_{24}N_8O_4$)($ClO_4$)$_2$] complex **6** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **6** is 64 µg/mL

## 8.15 DISCUSSION:

The minimum inhibitory concentrations (MIC) of the complexes $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ as **1,** $[Cd\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ as **6** were determined against fourteen pathogenic bacteria viz.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

***14. Bacillus cereus*** *by serial dilution technique. The results were shown in Table (8..43).*

**Table-8.43:** MIC of $[Cd(C_8H_{16}N_8O_4)(ClO_4)_2]$ as **1,**
$[Cd\ (C_{20}H_{24}N_8O_4)(ClO_4)_2]$ as **6**

| Test organism | Complex **1** | Complex **6** |
|---|---|---|
| | µg/mL | |
| *Salmonella-17* | 64 | 64 |
| *Klebsilla* | 64 | 64 |
| *Shigella dysenteriae* | 64 | 64 |
| *Shigella shiga* | 64 | 64 |
| *Shigella boydii* | 64 | 64 |
| *Shigella sonnei* | 64 | 64 |
| *Sigella flexneri* | 64 | 128 |
| *Escherichia coli* | 64 | 64 |
| *Pseudomonas aeruginosa* | 64 | 64 |
| *Salmonella* | 64 | 64 |
| *Bacillus megaterium* | 64 | 64 |
| *Sarcina lutea* | 64 | 64 |
| *Staphylococcus aureus* | 64 | 32 |
| *Bacillus cereus* | 64 | 64 |

## 9.11: RESULT OF Ag (II) CONTAIN COMPLEXES.

RESULT OF THE ANTIBACTERIAL ACTIVITY OF THE COMPLEXES (1-6) AGAINIST THE FOURTEEN PATHOGENIC BACTERIA VIZ. *SALMONELLA-17, KLEBSILLA, SHIGELLA DYSENTERIAE, SHIGELLA SHIGA, SHIGELLA BOYDII, SHIGELLA SONNEL, SHIGELLA FLEXNERI, ESCHERICHIA COLI, PSEUDOMONAS AERUGINOSA, SALMONELLA, BACILLUS MEGATERIUM, SARCINA LUTEA, STAPHYLOCOCCUS AUREUS, BACILLUS CEREUS.*

Table 9.0 Complexes abbreviation for antibacterial activity

| Complexes No. | Compound | Symbol |
|---|---|---|
| 1. | $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ | Ag +F+L |
| 2. | $[Ag(C_{10}H_{20}N_8O_4)(ClO_4)_2]$ | Ag+A+L |
| 3. | $[Ag\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ | Ag+B+L |
| 4. | $[Ag(\ C_{14}H_{24}N_8O_4)(ClO_4)_2]$ | Ag+Cro+L |
| 5. | $[Ag\ (C_{24}H_{28}N_8O_4)(ClO_4)_2]$ | Ag +Ci+L |
| 6. | $[Ag\ (C_{20}H\ _{24}N_8O_4)(ClO_4)_2]$ | Ag+Banzal+L |
| Ligand | $C_3N_4H_8O_2$ | L |

Table 9.1: Antibacterial activity of the complexes 1, 2, 3 against *Salmonella-17.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 20 | 10 | 6 | - |
| 2 | Ag+A+L | 17 | 8 | 3 | - |
| 3 | Ag+B+L | 18 | 10 | 5 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 16 |

Table 9.2: Antibacterial activity of the complexes 1, 2, 3 against *Klebsilla*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 18 | 8 | 4 | |
| 2 | Ag+A+L | 15 | 7 | 3 | |
| 3 | Ag+B+L | 17 | 9 | 4 | |
| | Control disc | Nil | | - | |
| | Standard disc | | | | 19 |

Table 9.3: Antibacterial activity of the complexes 1, 2, 3 against *Shigella dysenteriae.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 12 | 6 | 3 | - |

| | | | | | |
|---|---|---|---|---|---|
| 2 | Ag+A+L | 10 | 5 | 2 | - |
| 3 | Ag+B+L | 6 | 3 | 2 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 21 |

Table 9.4: Antibacterial activity of the complexes 1, 2, 3 against *Shigella shiga*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 18 | 10 | 4 | - |
| 2 | Ag+A+L | 20 | 10 | 5 | - |
| 3 | Ag+B+L | 15 | 8 | 4 | - |
| | Control disc | Nil | | | - |
| | Standard disc | | | | 21 |

Table 9.5: Antibacterial activity of the complexes 1, 2, 3 against *Shigella boydii*.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 18 | 10 | 5 | - |
| 2 | Ag+A+L | 15 | 8 | 4 | - |
| 3 | Ag+B+L | 14 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 9.6: Antibacterial activity of the complexes 1, 2, 3 against *Shigella sonnei*.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 18 | 10 | 5 | - |
| 2 | Ag+A+L | 12 | 6 | 3 | - |
| 3 | Ag+B+L | 16 | 8 | 4 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 9.7: Antibacterial activity of the complexes 1, 2, 3 against *Escherichia coli.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 26 | 12 | 5 | - |
| 2 | Ag+A+L | 20 | 10 | 4 | - |
| 3 | Ag+B+L | 15 | 7 | 3 | - |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 20 |

Table 9.8: Antibacterial activity of the complexes 1, 2, 3 against *Salmonella.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 10 | 8 | 2 | - |
| 2 | Ag+A+L | 18 | 14 | 4 | - |
| 3 | Ag+B+L | 18 | 9 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 20 |

Table 9.9: Antibacterial activity of the complexes 1, 2, 3 against *Bacillus megaterium.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 20 | 10 | 5 | - |
| 2 | Ag+A+L | 18 | 9 | 4 | - |
| 3 | Ag+B+L | 16 | 8 | 3 | - |
| | Standard disc | | | | 18 |

Table 9.10: Antibacterial activity of the complexes 1,2, 3 against *Sarcina lutea.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 20 | 12 | 6 | - |
| 2 | Ag+A+L | 18 | 9 | 4 | - |
| 3 | Ag+B+L | 16 | 7 | 3 | - |
| | Control disc | Nil | | - | - |
| | Standard disc | | | | 18 |

Table 9.11: Antibacterial activity of the complexes 1, 2, 3 against *Staphylococcus aureus.*

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |
| 1 | Ag +F+L | 28 | 14 | 6 | |
| 2 | Ag+A+L | 20 | 10 | 4 | |
| 3 | Ag+B+L | 18 | 9 | 3 | |
| | Control disc | Nil | - | - | - |
| | Standard disc | | | | 18 |

Table 9.12: Antibacterial activity of the complexes 1, 2, 3 against *Bacillus cereus*

.

| Complex No | Symbol | Zone of inhibition of mycelia growth (in mm) | | | |
|---|---|---|---|---|---|
| | | 200µg/disc | 100µg/disc | 50µg/disc | 30µg/disc |

| | | | | | |
|---|---|---|---|---|---|
| 1 | Ag +F+L | 18 | 10 | 5 | - |
| 2 | Ag+A+L | 12 | 6 | 3 | - |
| 3 | Ag+B+L | 16 | 8 | 4 | - |
| | Control disc | Nil | - | - | |
| | Standard disc | | | | 20 |

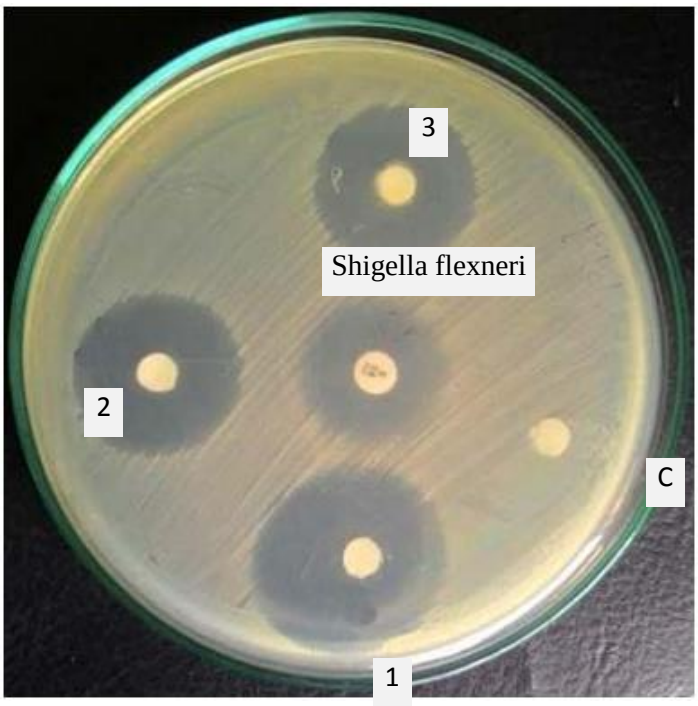


Fig: 9.1: Photographic representation of zone of inhibition of the complexes1,2, 3 the standard compound kanamycin against Shigella flexneri.

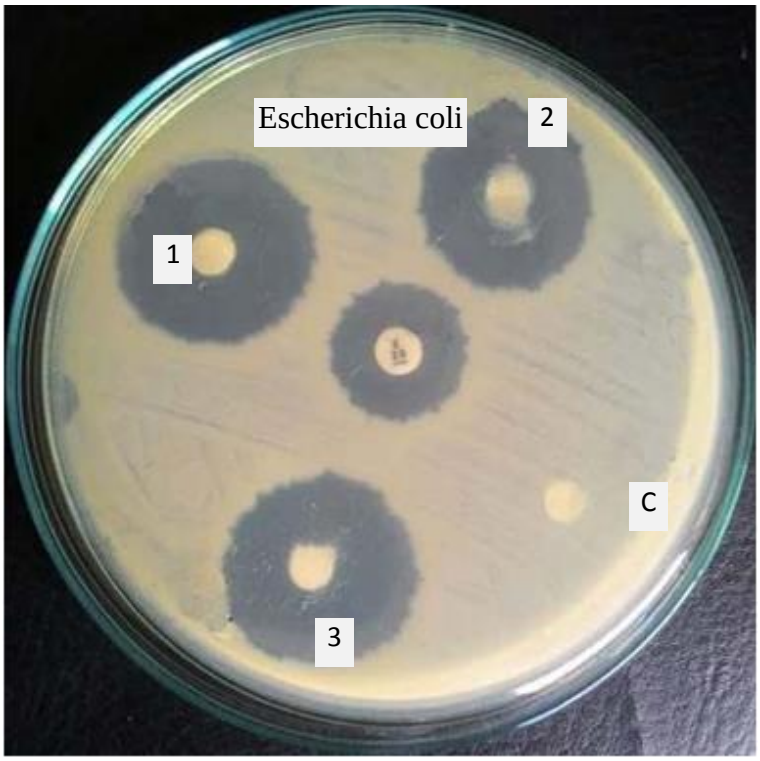


Fig: 9.2: Photographic representation of zone of inhibition of the complexes 1, 2, 3 the standard compound kanamycin against Escherichia coli.

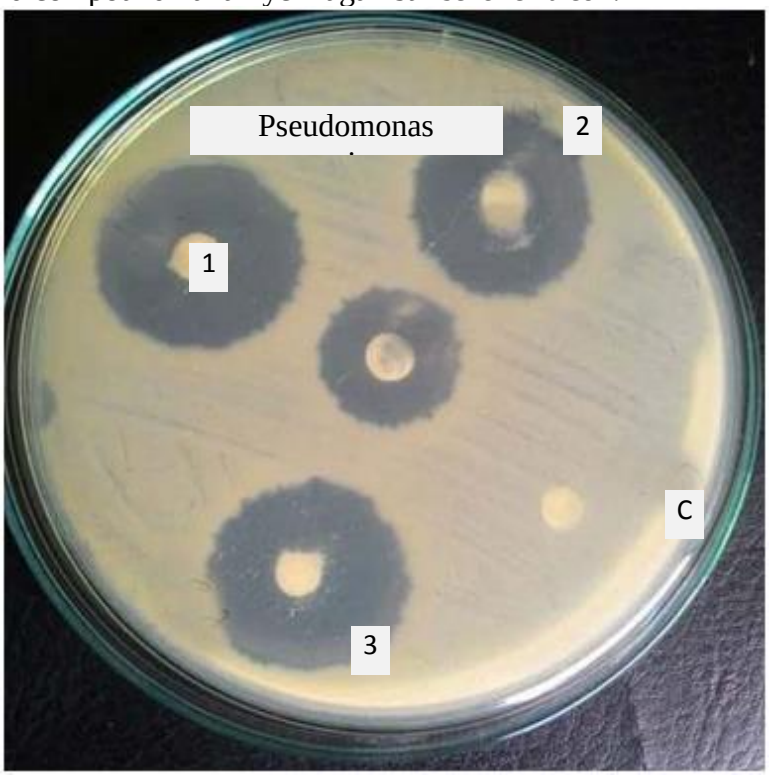

Fig: 9.3: Photographic representation of zone of inhibition of the complexes 1, 2, 3 the standard compound kanamycin against Pseudomonas aeruginosa.

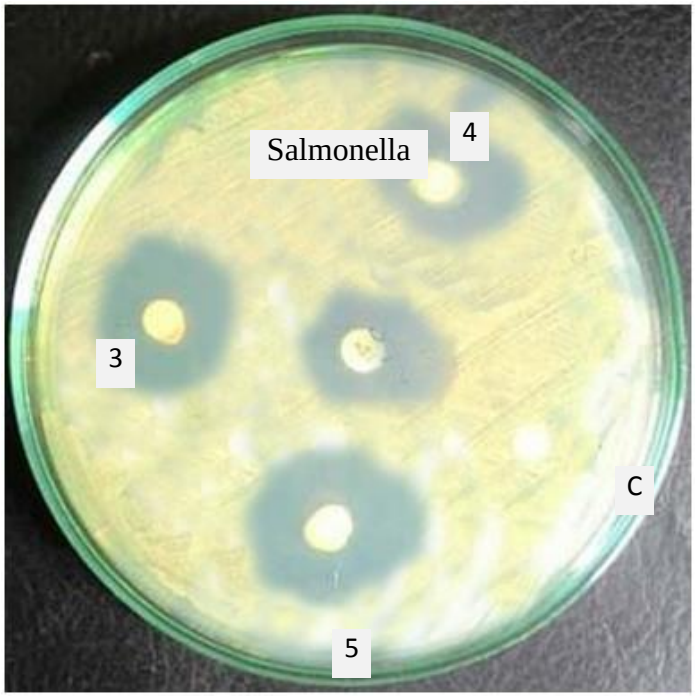


Fig: 9.4: Photographic representation of zone of inhibition of the complexes 3, 4, 5 the standard compound kanamycin against Salmonella.

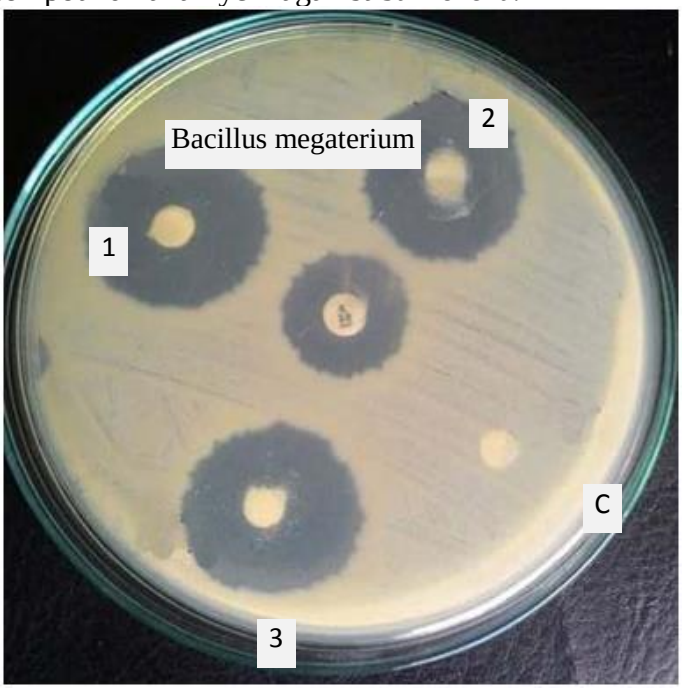

Fig: 9.5: Photographic representation of zone of inhibition of the complexes 1, 2, 3 the standard compound kanamycin against Bacillus megaterium.

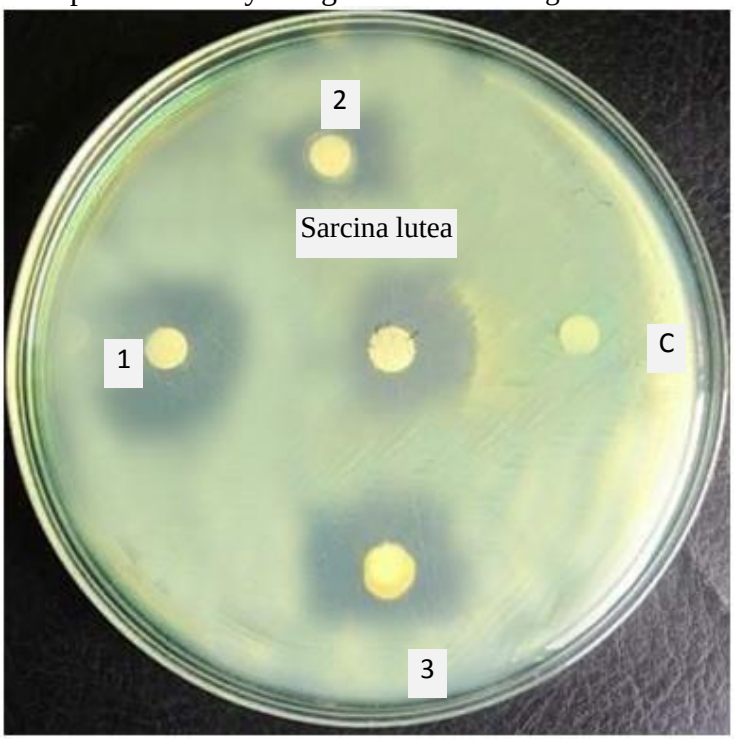


Fig: 9.6: Photographic representation of zone of inhibition of the complexes 1, 2, 3 the standard compound kanamycin against Sarcina lutea.

## 9.12 DISCUSSION

It has been observed that some drug increases the activity when administered as metal complexes or their metal chalets[4]. The antibacterial activity of the metal complexes 3, 4, 5 and other complexes are recorded against fourteen pathogenic bacteria viz.

| *Gram Negative* | *Gram Positive* |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |

| | |
|---|---|
| *4. Shigella shiga* | *14. Bacillus cereus* |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

And the result is given in (Table 5.1-5.12) the complex 3,4,**5** showed the most acitivities above fourteen pathogenic bacteria as shown Table (Fig 5.1-5.6). It is evident from all the tables that the under investigation showed the most activity compared to the complex **2,6**

The malanodihydrazied complexes **1,2** and **6** have shown good activity against the above fourteen pathogenic bacteria as seen in (Table 5.1-5.12). The complex **1** showed the best activity against *E. Coli,* less activity against *Klebsilla.* The complex **3** showed the best activity *Salmonella-17, Shigella shiga, Shigella sonnei* and less activity against *Pseudomonas aeruginosa*. The complex **5** showed the best activity against *Bacillus cereus* and less activity against *Shigella boydii* the complex **6** are not showed good activities against the above fourteen pathogenic bacteria.

The good activity against *Bacillus cereus* and less activity *Shigella dysenteriae* and other bacteria was not seen. Similarly the complex **2** showed good activities *E.Coli* and less activity against *Bacillus megatrium* and other bacteria was not seen activities. The complex **4** showed good activities

*Salmonella* and less activities against *shigella sonnei* and other bacteria was not seen activities. The complex **6** showed good activities *Shigella dysenteriae* and less activities against *Shigella boydii*, *Bacillus megaterium* and other bacteria was not seen activities. All the result are compared with the standard compound, kanamycin as seen in the Table (5.1-5.12) the ligand malanodihydrazide ($C_3H_8N_4O_2$) did not show any activities against the above fourteen pathogenic bacteria.

From here it is concluded that the complex **3, 4** and **5** showed good activities against the fourteen pathogenic bacteria as compared to the standard compound, kanamycin. It is evident that the ligand malanodihydrazide did not show any activity.

**9.14** RESULT OF THE MINIMUM INHIBITORY CONCENTRATION OF THE COMPLEXES $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$, $[Ag(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ AGAINST THE FOURTEEN PATHOGENIC BACTERIA VIZ.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |

| | |
|---|---|
| *4. Shigella shiga* | *14. Bacillus cereus* |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

**Table 9.15:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$, complex **1** against *Salmonella-17.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

Where

+ve = Growth

-ve = Not growth

The MIC of the complex **1** is 32 µg/mL

**Table 9.16:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$, complex **1** against *Klebsilla*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.17:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$, complex **1** against *Shigella dysenteriae.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | -ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.18:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$, complex **1** against *Shigella shiga*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.19:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.20:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.21:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Shigella flexneri*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.22:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Escherichia coli.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.23:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.24:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Salmonella.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.25:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.26:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$, complex **1** against *Sarcina lutea*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.27:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Staphylococcus aureus*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.28:** Minimum inhibitory concentration of $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ complex **1** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **1** is 32 µg/mL

**Table 9.29:** Minimum inhibitory concentration of $[Ag(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Salmonella-17.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.30:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Klebsilla*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.31:** Minimum inhibitory concentration of $[Ag(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **3** against *Shigella dysenteriae*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.32:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Shigella shiga.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.33:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Shigella boydii*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.34:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Shigella sonnei*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.35:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Shigella flexneri*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.36:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Escherichia coli.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.37:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Pseudomonas aeruginosa*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.38:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Salmonella*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.39:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Bacillus megaterium.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.40:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Sarcina lutea*.

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.41:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Staphylococcus aureus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | +ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**Table 9.42:** Minimum inhibitory concentration of [Ag $(C_{14}H_{28}N_8O_4)(ClO_4)_2$] complex **3** against *Bacillus cereus.*

| Test tube No | Nutrient broth medium added (mL) | Diluted solution of complex (µg/mL) | Inoculum added (µL) | Observation |
|---|---|---|---|---|
| 1. | 1 | 512 | 10 | -ve |
| 2. | 1 | 256 | 10 | -ve |
| 3. | 1 | 128 | 10 | -ve |
| 4. | 1 | 64 | 10 | -ve |
| 5. | 1 | 32 | 10 | +ve |
| 6. | 1 | 16 | 10 | +ve |
| 7. | 1 | 8 | 10 | +ve |
| 8. | 1 | 4 | 10 | +ve |
| 9. | 1 | 2 | 10 | +ve |
| $C_S$ | 1 | 512 | 0 | -ve |
| $C_i$ | 1 | 0 | 10 | +ve |
| $C_M$ | 1 | 0 | 0 | -ve |

The MIC of the complex **3** is 32 µg/mL

**9.15 DISCUSSION:**

The minimum inhibitory concentrations (MIC) of the complexes $[Ag(C_8H_{16}N_8O_4)(ClO_4)_2]$ as **1,** $[Ag\ (C_{14}H_{28}N_8O_4)(ClO_4)_2]$ as **3** were determined against fourteen pathogenic bacteria viz.

| ***Gram Negative*** | ***Gram Positive*** |
|---|---|
| *1. Salmonella-17* | *11. Bacillus megaterium* |
| *2. Klebsilla* | *12. Sarcina lutea* |
| *3. Shigella dysenteriae* | *13. Staphylococcus aureus* |
| *4. Shigella shiga* | |
| *5. Shigella boydii* | |
| *6. Shigella sonnei* | |
| *7. Shigella flexneri* | |
| *8. Escherichia coli* | |
| *9. Pseudomonas aeruginosa* | |
| *10. Salmonella* | |

***14. Bacillus cereus*** *by serial dilution technique. The results were shown in Table (9.43).*

**Table-9.43:** MIC of $[Ag(C_{14}H_{28}N_8O_4)(ClO_4)_2]$ complex **1**
$[Ag(C_{24}H_{28}N_8O_4)(ClO_4)_2]$ complex **3**

| Test organism | Complex **1** | Complex **3** |
|---|---|---|
| | µg/Ml | |
| *Salmonella-17* | 32 | 32 |
| *Klebsilla* | 32 | 32 |
| *Shigella dysenteriae* | 32 | 32 |
| *Shigella shiga* | 32 | 32 |
| *Shigella boydii* | 32 | 32 |
| *Shigella sonnei* | 32 | 32 |
| *Sigella flexneri* | 32 | 32 |
| *Escherichia coli* | 32 | 32 |
| *Pseudomonas aeruginosa* | 32 | 32 |
| *Salmonella* | 32 | 32 |
| *Bacillus megaterium* | 32 | 32 |
| *Sarcina lutea* | 32 | 32 |
| *Staphylococcus aureus* | 32 | 32 |
| *Bacillus cereus* | 32 | 32 |

## 10.01 ANTIFUNGAL ACTIVITY

The minimum effective comcentration (MEC) of the complexes were determined four used to assess the in vitro antifungal activity of against Aspergillus Niger, Socehoramyces cerevisae, Pericillium notatum, mueor sp. I cound not found any activity all the complexes. Photograp of the activity of complexes as flows.

Fig: 10.01: Photographic representation of zone of inhibition of the complexes in antifungul activity.

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# *10.02 ABSTRACTS OF THE PUBLICATIONS BASED ON THE PRESENT WORK*

*Internatioal journal of advance research (IJOAR) Org.*
*Volum 2, Issuse 12, December 2014, Online: ISSN 2320-9178*

# THE SYNTHESIZED OF MACRO CYCLIC COMPLEX COMPOUNDS OF Pb(II) CONTAINING A LIGAND HAVING TETRAOXOTETRAHYDRAZIN MOITY ON SOME PATHOGENIC BACTERIA.

M.R. Ullah, M. J. Hossain,
*Department of Chemistry, BUET, Dhaka.*

Abstract:
The macrocyclic complex compounds of Pb(II) containing a ligand having tetraoxotetrahydrazin moity are synthesized by template condensation of malonodihydrazide ($C_3H_8N_4O_2$) with different aldehydes. The complexes are characterized on the basis of elemental analysis, UV-visible & IR spectroscopy, magnetic moment and conductance measurement and other physical properties**.** The IR spectrum study of the complex compounds suggests that ligand coordinates to metal ions through the nitrogen atoms from the tetraoxotetrahydrazin moity. Antibacterial activity of the derived complex compounds, as well as already used standard compound kanamycin, was tested on fourteen pathogenic bacteria. Given results were then compared to the efficacy of the Antibacterial activity of standard compound kanamycin used for control of these pathogenic bacteria.

*International organization of sciencetific Research (IOSR)*
*IOSR Journal of applied chemistry (IOSR JAC)*
*Volum 9, Issuse 12, December 2016, PP 13-19 Online: ISSN 2278-5736*

# COMPLEX FORMATION OF Zn(II) PERCHLORATE IONS USING A LIGAND HAVING TETRAOXOTETRAHYDRAZIN MOITY ON SOME PATHOGENIC BACTERIA.

M.R. Ullah, M. J. Hossain,
*Department of Chemistry, BUET, Dhaka.*

Abstract:
Six new macrocyclic complexes were synthesized by template reaction of malanodihydrazide with Zn(II) perchlorate. The metal perchlorate to ligand proper molar ratio and containing a ligand having tetraoxotetrahydrazin moity are synthesized by template condensation of malonodihydrazide ($C_3H_8N_4O_2$) with different aldehydes. The complexes are characterized on the basis of elemental analysis, UV-visible & IR spectroscopy, T.G.A. magnetic moment and conductance measurement and other physical properties**.** The IR spectrum study of the complex compounds suggests that ligand coordinates to metal ions through the nitrogen atoms from the

tetraoxotetrahydrazin moity**.** Antibacterial activity of the derived complex compounds, as well as already used standard compound kanamycin, was tested on fourteen pathogenic bacteria. Given results were then compared to the efficacy of the Antibacterial activity of standard compound kanamycin used for control of these pathogenic bacteria.

*International Journal of Mathematics and Statistics Invension (IJMSI)*
*Volum 5, Issuse 2, December 2017, PP 58-67 Online: ISSN 2321-4759*

# SYNTHESIS AND CHARACTERIZATION OF THERMAL ANALYSIS OF La(II) PERCHLORATE CONTAINING A LIGAND HAVING TETRAOXOTETRAHYDRAZIN MOITY ON SOME PATHOGENIC BACTERIA.

M.R. Ullah, M. J. Hossain
*Department of Chemistry, BUET, Dhaka.*

.

Abstract:
The macrocyclic complex compounds of La(II) containing a ligand having tetraoxotetrahydrazin moity are synthesized by template condensation of malonodihydrazide ($C_3H_8N_4O_2$) with different aldehydes. The complexes are characterized on the basis of elemental analysis, UV-visible & IR spectroscopy, magnetic moment and conductance measurement and other physical properties. The IR spectrum study of the complex compounds

suggests that ligand coordinates to metal ions through the nitrogen atoms from the tetraoxotetrahydrazin moity. Antibacterial activity of the derived complex compounds, as well as already used standard compound kanamycin, was tested on fourteen pathogenic bacteria. Given results were then compared to the efficacy of the Antibacterial activity of standard compound kanamycin used for control of these pathogenic bacteria.

*International journal of Engineering Research and Application (IJERA)*
*(Wating date 29/05/2017)*

# THE SYNTHESIZED OF MACRO CYCLIC COMPLEX COMPOUNDS OF Cd (II) CONTAINING A LIGAND HAVING TETRAOXOTETRAHYDRAZIN MOITY ON SOME PATHOGENIC BACTERIA.

M.R. Ullah, M. J. Hossain
*Department of Chemistry, BUET, Dhaka.*

Abstract:
The macrocyclic complex compounds of Cd(II) containing a ligand having tetraoxotetrahydrazin moity are synthesized by template condensation of malonodihydrazide ($C_3H_8N_4O_2$) with different aldehydes. The complexes are characterized on the basis of elemental analysis, UV-visible & IR

spectroscopy, TGA, DTA, magnetic moment and conductance measurement and other physical properties. The IR spectrum study of the complex compounds suggests that ligand coordinates to metal ions through the nitrogen atoms from the tetraoxotetrahydrazin moity. Antibacterial activity of the derived complex compounds, as well as already used standard compound kanamycin, was tested on fourteen pathogenic bacteria. Given results were then compared to the efficacy of the Antibacterial activity of standard compound kanamycin used for control of these pathogenic bacteria.

*International Journal of contemporary Research & Review. (Waiting 30/05/2017)*

# SYNTHESIZED, COMPLEX COMPOUNDS OF SILVER PERCHLORATE CONTAINING A LIGAND HAVING TETRAOXOTETRAHYDRAZIN MOITY ON SOME PATHOGENIC BACTERIA.

M.R. Ullah, M. J. Hossain

*Department of Chemistry, BUET, Dhaka.*

.

Abstract

The macrocyclic complex compounds of Ag(I) containing a ligand having tetraoxotetrahydrazin moity are synthesized by template condensation of malonodihydrazide ($C_3H_8N_4O_2$) with different aldehydes. The complexes are characterized on the basis of elemental analysis, UV-visible & IR spectroscopy, magnetic moment and conductance measurement and other physical properties. The IR spectrum study of the complex compounds suggests that ligand coordinates to metal ions through the nitrogen atoms from the tetraoxotetrahydrazin moity. Antibacterial activity of the derived complex compounds, as well as already used standard compound kanamycin, was tested on fourteen pathogenic bacteria. Given results were then compared to the efficacy of the Antibacterial activity of standard compound kanamycin used for control of these pathogenic bacteria.