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Structural Studies of some Oxo-metal Complexes of Tetra dentate (2N,2O) Schiff bases.

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Department of Chemistry

Title of Thesis

Structural Studies of some Oxo-metal Complexes of Tetra dentate (2N, 2O) Schiff bases

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بسم الله الرحمن الرحيم

﴿ وَمَا تَكُونُ فِي شَأْنٍ وَمَا تَتْلُوا۟ مِنْهُ مِن قُرْءَانٍ وَلَا تَعْمَلُونَ مِنْ عَمَلٍ إِلَّا
كُنَّا عَلَيْكُمْ شُهُودًا إِذْ تُفِيضُونَ فِيهِ وَمَا يَعْزُبُ عَن رَّبِّكَ مِن مِّثْقَالِ ذَرَّةٍ فِي
الْأَرْضِ وَلَا فِي السَّمَاءِ وَلَا أَصْغَرَ مِن ذَٰلِكَ وَلَا أَكْبَرَ إِلَّا فِي كِتَٰبٍ مُّبِينٍ ﴾

[يونس : 61]

صدق الله العظيم

Dedication

To My

wife
Parents, brothers, waif and babies

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Acknowledgment

I would Like to express my great gratitude to the almighty Allah for his well and help to accomplish this work I would like to give special thanks to Supervisor me Dr.Abdulhakem A.Ahmed for his invaluable effort and guideness throughout the past three years of work. Finally I would like to the thank faculties of science at AL-tahady University and the University of Garyounis for providing the required facilities for the research.

Otman Almajdoub Alsusi

ABSTRACT

The oxo-metal complexes were synthesized using tetra dentate Schiff base ligands (2N,2O). The Schiff base ligands were obtained by the condensation of some primary amine (ethylenediamine and 1,6-hexandiamine) with some ketone (Benzoylacetone, Acetylacetone and Benzoin) which are produced the following ligands, H_2L^1=[$C_{22}H_{24}N_2O_2$], H_2L^2=[$C_{26}H_{32}N_2O_2$], H_2L^3=[$C_{16}H_{28}N_2O_2$], H_2L^4=[$C_{12}H_{20}N_2O_2$] and H_2L^5=[$C_{30}H_{28}N_2O_2$]. These tetradentate ligands have the 2N, 2O donor sites. The tetradentate ligands were reacted with some oxo-metal cations namely, $UO2^{2+}$, VO^{2+}, $MoO2^{2+}$ and ZrO^{2+}. This study confirmed the formation of mono-nuclear complexes for all the cations under investigation. The Schiff base ligands and their complexes have been were characterized by elemental analysis(C,H,N) ,infrared (IR), electronic (UV) and mass spectroscopy to verify their structures. The elemental analysis obtained for H%, C% and N% were in good agreements with the calculated values for the compounds. The IR showed the expected absorption bands for the compounds, since the frequencies attributed to the C=N, C=O, C-O and O-H groups were observed on the ligands spectra. And these bands in complexation have been shifted to lower frequencies and new bands due to M-C and M-N were also apparent. The mass spectrum data analysis for the ligands under

consideration showed a fragmentation pattern which were in excellent agreement with the molecular structures. The electronic spectra of complexes in 10^{-3} M DMF or chloroform showed the expected transitions. In addition, the measured values of the magnetic moment suggested the square pyramidal stereochemistry of most complexes.

Chapter(I)

Introduction

1. Introduction

1.1. Schiff base

Since the discovery of Schiff base (azomethine), by Hugo Schiff, the German chemist, there have been extensive work carried on the Schiff base and their complexes due to their biological and industrial applications. The Schiff base is a functional group that contains a carbon-nitrogen double bond with the nitrogen atom connected to an aryl or alkyl group but not hydrogen.[1]

1.2. Schiff base complexes:

1.2.1. Vanadyl complexes

The synthesis and characterization of the V(IV) complex of a salicylaldimine, $[V(C_{20}H_{14}N_2O_4)O].2C_2H_6OS.2H_2O$, has been described. The reaction of 2,4-dihydroxybenzaldehyde and 1,2-phenylenediamine with vanadyl sulfate produces a monomeric V(IV) complex. Its structure reveals that the vanadium(IV) ion is pentacoordinated and situated in a distorted square-pyramidal environment[1].

Structural modification of organic molecule has considerable biological relevance. Further, coordination of a biomolecules to the metal ions significantly alters the effectiveness of the biomolecules. In view of the antimicrobial activity ligand [bis-(2-aminobenzaldehyde)] malonoyl dihydrazone], metal complexes with Cu(II), Ni(II), Zn(II) and oxovanadium(IV) have been synthesized and found to be potential antimicrobial agents. An attempt is also made to correlate the biological activities with geometry of the complexes. The complexes have been characterized by elemental analysis, molar conductance, spectra and cyclicvoltammetric measurements. The structural assessment of the complexes has been carried out based on electronic, infrared and molar conductivity values [2].

The electrochemical properties of vanadyl(IV) derivatives, namely salen Schiff base complexes of the type [VO(Salen)] (5-BrSalen, 5-NO_2Salen, 5-MeOSalen, salpn (bis(salicylaldehyde)-1,3-propanediamine, 5-BrSalpn, 5-NO_2Salpn, 5-MeOSalpn,Me_2Salen, Salophen, 5-BrSalophen, and 5-MeOSalophen) have been investigated. The equatorial Schiff base ligands affect the oxidation potentials *via* interaction with the d-orbitals of the vanadyl metal ion. The cathodic peak potential (Epc) becomes less negative according to the sequence $MeO^- < H^- < Br^- < NO_2^-$[3].

Two series of the unsymmetrical diimino tetradentate Schiff bases derived from phenylenediamine and 1,3-naphthalene diamine and their vanadyl complexes have been made and characterized by 1H, 13C NMR, IR, UV-Vis and elemental analysis [4].

Fifteen Schiff base ligands were synthesized and used to form complexes with vanadium in oxidation states III, IV, and V. Electrochemical and spectral characteristics of the complexes were evaluated and compared. In acidified solutions in acetonitrile the vanadium(IV) complexes undergo reversible disproportionation to form V(III) and V(V) complexes. With several of the ligands the V(III) complexes are much more stable in the presence of acid than is the previously studied complex with salen, an unelaborated Schiff base ligand (H_2 salen = *N,N'*-ethylenebis (salicylideneiminato)). Equilibrium constants for the disproportionation were evaluated. The vanadium(III) complexes reduce dioxygen to form two oxo ligands. The reaction is stoichiometric in the absence of acid, and second-order rate constants were evaluated. In the presence of acid some of the complexes investigated participate in a catalytic electroreduction of dioxygen [5].

Neutral tetradentate N_2O_2 type complexes of Cu(II), Ni(II), Mn(II), Zn(II) and VO(II) have been prepared using a Schiff base ligand formed by the condensation of *o*-phenylenediamine with acetoacetanilide in alcohol medium. All the complexes have been characterised on the basis of their microanalytical data, molar conductance, magnetic susceptibility, IR, UV–Vis 1H NMR and ESR spectra. IR and UV–Vis spectral data suggest that all the complexes are square-planar except the Mn(II) and VO(II) chelates, which are of octahedral and square pyramidal geometry respectively. The monomeric and neutral nature of the complexes was confirmed by their magnetic susceptibility data and low conductance values. The ESR spectra of copper and vanadyl complexes in DMSO solution at 300 K and 77 K were recorded and their salient features are reported.[6]

The saccharinato complexes of Au(III), ZrO(II), VO(II) and UO_2(II) metal ions have been prepared and the coordination of saccharin in these complexes has been investigated through their ^{1}HNMR and IR spectra as well as by thermal analysis. It was found that saccharin interacts with all of these metal ions in the anionic form and coordinates in a monodentate fashion through its nitrogen to Au(III), ZrO(II) and VO(II) ions, whereas it coordinates to UO_2(II) ion as a bidentate ligand using its carbonyl and sulphonyl groups. A square structure has been proposed for Au(III) complexes, polymeric chain structures for ZrO(II) and VO(II) complexes and an octahedral structure for UO_2-saccharin complex. The thermal properties of these complexes were shown to be consistent with the proposed structures and indicate that metallic gold, ZrO_2, V_2O_5 and UO_2SO_4 are obtained as final thermal decomposition products of these complexes [7].

1.2.2. Uranyl complexes

Four new complexes $[UO_2(HNTA)(H_2O)].3H_2O$, $Na[UO_2(NTA)].(H_2O)$, $[ZrO(HNTA)(H_2O)].3H_2O$ and $Na[ZrO(NTA)].H_2O$ were obtained by the reactions of UO_2^{2+} and ZrO^{2+} salts with nitrilotriacetic acid (NTA) dissolved in water and NaOH aqueous solution. Infrared spectra of the solid complexes have been obtained and assigned on the basis of Cs point group. Octahedral and polymeric chain structures are adopted for the uranyl and zirconyl NTA complexes, respectively. Thermogravimetric (TG) and differential thermal analysis (DTA) data are in accordance with the proposed structures and showed that the complexes decompose to metal oxides.[8]

terdentate neutral complexes of Cu(II), Ni(II), Co(II), Mn(II), Zn(II), Cd(II), Hg(II), VO(II), ZrO(II) and UO_2 II have been made using a Schiff base derived from 1-phenyl-2,3-dimethyl-4-aminopyrazol-5-one (4-aminoantipyrine) and acetoacetanilide. The structural features of the chelates have been confirmed by microanalytical data, IR, UV-Vis, ^{1}HNMR, ESR and mass spectral techniques. Electronic absorption and IR spectra of the complexes indicate an octahedral geometry around the central metal ion, except for the VOII and ZrOII complexes which show square pyramidal geometry.[9]

The synthesis and characterization of 2-acetylpyridine-(2-aminobenzoylhydrazone)(HL1) and its first dioxouranium complex has been described. The single crystal X-ray structural analysis of the hydrazone, HL1, reveals an almost planar structure with intermolecular hydrogen bonds. The complex $[UO_2(L1)(DMF)_2](NO_3)$ is formed by refluxing a suspension of HL1 with uranyl nitrate in methanol in the presence of a small quantity of triethylamine. Recrystallisation from DMF results in single crystals suitable for X-ray crystallography. The single crystal X-ray study of the complex shows a distorted pentagonal-bipyramidal coordination geometry around the uranium(VI) center, determined by two axial oxo ligands, two nitrogen and one oxygen atoms from a chelating monodeprotonated hydrazone and the oxygen donor atoms of two dimethylformamide molecules.[10]

The reaction of uranyl nitrate with asymmetric Schiff base ligands in the presence of base yields dinuclear uranyl complexes, $[UO_2(HL1)]_2{\cdot}DMF$ (1), $[UO_2(HL2)]_2{\cdot}2DMF{\cdot}H_2O$ (2), and $[UO_2(HL3)]_2{\cdot}2DMF$ (3) with 3-(2-hydroxybenzylideneamino)propane-1,2-diol (H_3L1), 4-((2,3-dihydroxy-propylimino)methyl)benzene-1,3-diol (H_3L2), and 3-(3,5-di-tert-butyl-2-hydroxybenzylideneamino)propane-1,2-diol (H_3L3), respectively. All complexes exhibit a symmetric U_2O_2 core featuring distorted pentagonal bipyramidal geometry around each uranyl center. The hydroxyl groups on the ligands are attached to the uranyl ion in chelating, bridging, and coordinate covalent bonds. Distortion in the backbone is more pronounced in 1, where the phenyl groups are on the same side of the planar U_2O_2 core. The phenyl groups are present on the opposite side of U_2O_2 core in 2 and 3 due to electronic and steric effects. A similar hydrogen-bonding pattern is observed in the solid-state structures of 1 and 3 with terminal hydroxyl groups and DMF molecules, resulting in discrete molecules. Free aryl hydroxyl groups and water molecules in 2 give rise to a two-dimensional network with water molecules in the channels of an extended corrugated sheet structure. Compound 1 in the presence of excess $Ag(NO_3)$ yields $\{[(UO_2)(NO_3)(C_6H_4OCOO)](NH(CH2CH3)3)\}2$ (4), where the geometry around the uranyl center is hexagonal bipyrimidal. Two-phase extraction studies of uranium from aqueous media employing H_3L_3 indicate 99% reduction of uranyl ion at higher pH. [11]

A new approach to the molecular recognition of metal oxo cations was introduced based on a ligand design strategy that provides at least one hydrogen bond donor for interaction with oxo group(s) as well as conventional electron pair donor ligands for coordination to the metal center. This concept of stereognostic coordination of oxo metal ions is exemplified in the design of four tripodal ligands tris[2-(2-carboxyphenoxy)ethyl]amine [NEB], tris[3-(2- carboxyphenoxy)propyl] amine [NPB], tris[3-(2-carboxynaphthyl-3-oxy)propyl]amine [NPN] , and tris[3-(2-carboxy-4-octadecylphenoxy)propyl]amine[NPodB]or sequestrate of the uranyl ion. The ligands NEB, NPB, and NPN form 1:1 complexes with $U0_2^{2+}$. The bidentate coordination of carboxyl groups of these compounds is indicated by the infrared spectra, which offer some support for the presence of a hydrogen bond to the uranyl group. [12]

1.2.3. Zirconyl and Molybdenyl complexes

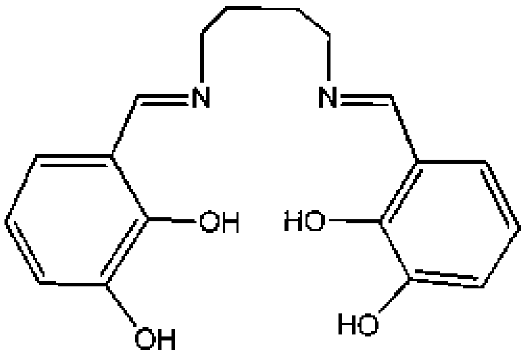
Fewer zirconyl and molybdyl Schiff base complexes have been reported in comparison with uarnyl and vandyl counter parts. Pyridine N-oxide complexes having the composition $ZrO(Py.O).(ClO_4)_2$, $Th(Py.O)_8(ClO_4)_4$, and $U02(Py.O)_6(ClO_4)_2$ have been prepared. The infrared and electronic absorption spectra show that the bonding between the metal and pyridine N-oxide in the complexes has occurred by donation of the lone pair of p-electrons on oxygen to the metal, and that the T-bondcharacter of NO group increases in the complexes as uranyl < thorium < zirconyl. The decrease in the vibrational structure of the UO_2^{2+} spectrum in the visible region indicates strong coordination of pyridine N-oxide to the uranyl group. The decomposition temperatures of zirconyl, thorium, and uranyl complexes are 307, 350, and 319° C respectively [13].

The synthesis of two monomeric molybdenum(VI) Schiff base complexes derived from N-phenyl-2-hidroxy-1-naphthaldimine and N-benzyl-2-hidroxy-1-naphthaldimine: $[MoO_2(C_5H_7O_2)(OCH_3)(2-OC_{10}H_6CH=NHC_6H_5)]$ and $[MoO_2(C_5H_7O_2)(OCH_3)(2-OC_{10}H_6CH=NH-CH_2C_6H_5)]$ have been described. These complexes are the first examples in which potentially bidentate Schiff bases are monodentately bonded to molybdenum as zwitterionic ligands. The structure of first complex was determined by X-ray analysis. Within the same ligand, the hydrogen atom attached to nitrogen forms an intramolecular hydrogen bond of 2.548 Å with the oxygen atom coordinated to molybdenum. The octahedral coordination of molybdenum is completed by two oxo-oxygens, two acetylacetonato oxygens and one methoxy oxygen. The compounds were characterized by chemical analysis, IR spectroscopy and single crystal structure determination. [14]

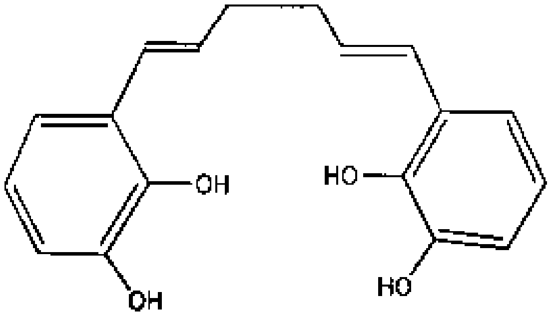
Six new dioxobridged complexes of molybdenum (IV) and tungsten (IV) with N-alkylphenothiazines having the general formula $M_2O_4(L)_2(H_2O)_2$ [whereM = molybdenum or tungsten and L = N-alkyl-phenothiazines] have been synthesized. The complexes have been characterised on the basis of analytical, molar conductance ,magnetic susceptibility, spectral data, TGA and DTA. The low magnetic moments for the complexes are due to spin–spin interaction or metal–metal bonding. The interactions of these complexes with some biologically important amino acids have been studied. [15]

Three optically active Molybdenum (VI) dioxo complexes with tetrahydro salen and substituted tetrahydro salen derivatives as ligands were synthesized and examined as catalysts for asymmetric epoxidation. Complexes of the type MoO2(L)(Solv) and WO2(L) (L = tridentate, trans-2-aminocyclohexanol derived chiral Schiff base, Solvent = alcohol) were prepared and characterized by elemental analysis, NMR and IR spectroscopy. These complexes were applicable as catalysts for olefin epoxidation reactions with tert-butyl hydroperoxide being the oxidizing agent. In case of cis- β-methylstyrene moderate enantiomeric excesses of up to 26% can be reached when the reaction is carried out at 0°C. [16]

The metal ions use to control the self-assembly of multicomponent supramolecular structures is a subject of such investigation, with particular attention to multihelical species and those containing internal cavities.[17]



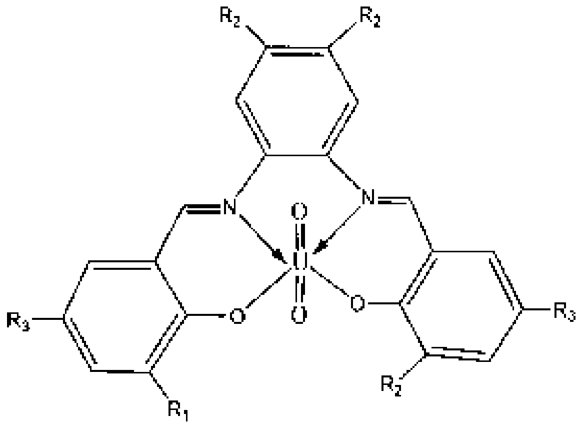
H_4L



H_4L

Likewise, the chemistry of metal complexes with dicompartmental ligands has arisen a growing interest because of their biomimetic possibilities, catalytic properties, ability to stabilize unusual or mixed oxidation states and their usefulness to design molecular magnetic materials.[17]

Uranyl salophenes, for the first time, have been utilized in the experiments with complexation and recognition of polar neutral molecules, i.e. formamide, acetamide, dimethylsulfoxide, urea derivatives, etc.[18]



I. $R_1=R_2=R_3=H$
II $R_1=R_3=H$, $R_2=.OC_{12}H_{25}$
III $R_1=R_2,R_3=t\text{-}Bu$

Chapter (II)

Experimental

2. Experimental

2.1. Materials and reagents

All materials and reagents used in this study were laboratory pure chemical. They include, as shown in table.

Materials and reagents	M.Wt	Formula	The company
Ethylenediamine	60.09	C2H8N2	Aldrich
1,6-hexanediamine	116.2	C6H16N2	Aldrich
Benzoylacetone	162.18	$C_{10}H_{10}O_2$	Riedel-dehaen
Acetyl acetone	100.11	$C_5H_8O_2$	Sigma Chemical CO. Steinheim, Germany
Benzoin	212.24	$C_{14}H_{12}O_2$	Aldrich
Ethanol	46.07	C_2H_5OH	Carblo erba regenti
petroleum ether 60 – 80 C°		-----------	Nice
Dimethylformamide	73.09	C_3H_7NO	Riedel-dehaen
Uranyl Nitrate. hexahedrate	502.20	$UO_2\,(NO_3)_2.6H_2O$	Koch-Liht Laboratories LTD England
Zirconium oxide chloride.octahedrate	322.25	$ZrOCL_2.8H_2O$	Koch-Liht Laboratories LTD England
Sodium molybdate	241.96	$Na_2MoO_4.2H_2O$	Riedel-Dehaenag
Ammonium meta vanadat	116.96	NH_4VO_3	Riedel-Dehaenag

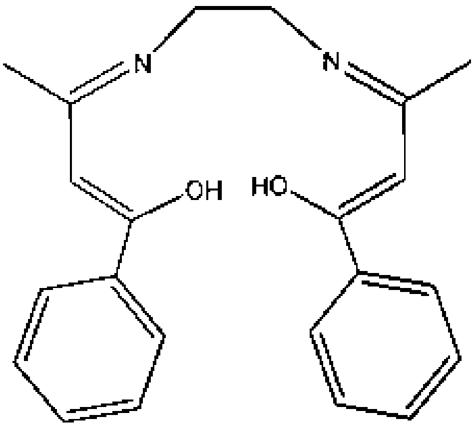
2.2. The synthesis of the Schiff base ligands

The synthesis of the Schiff base ligands was prepared by the literature procedure. An ethanolic solution (40ml) of primary diamine (1 mole) was refluxed with diketones (2mole) for about 12h. The volume of the solution was reduced to one third and 10ml of petroleum ether (60-80C°) was added with constant stirring. The solid product formed was filtered and recrystallized from ethanol and dried in vacuo. This method was based on the previous methods for the preparation Schiff base ligands.[19]

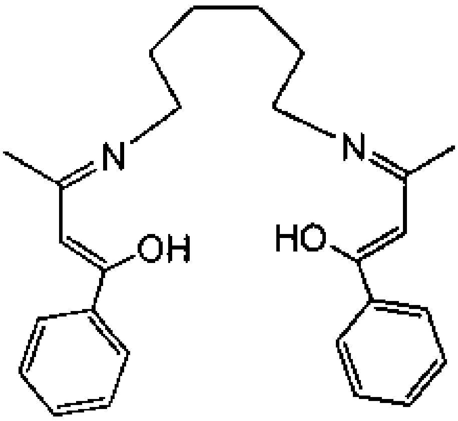
2.2.1. The preparation of ($H_2L^1 = C_{22}H_{24}N_2O_2$) ligand

An ethanolic solution (40ml) of Ethylenediamine (0.6 ml, 0.01mole) was refluxed with Benzoylacetone (3.24g, 0.02mole) for about 12h. The volume of the solution was reduced to one third and 10ml of petroleum ether (60-80C°) was added with constant stirring. The solid product formed was filtered and recrystallized from ethanol and dried in vacuo.

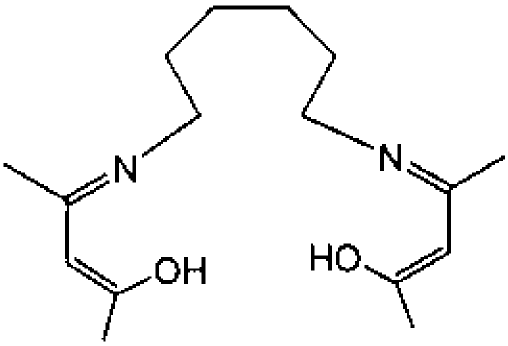
The same procedures was used for the synthesis of ($H_2L^1= C_{22}H_{24}N_2O_2$) from Ethylenediamine with Benzoylacetone , ($H_2L^2= C_{26}H_{32}N_2O_2$) from 1, 6-hexanediamine with Benzoylacetone , ($H_2L^3 = C_{16}H_{28}N_2O_2$) from 1, 6-hexanediamine with acetylacetone, ($H_2L^4 = C_{12}H_{20}N_2O_2$) from Ethylenedi amine with acetylacetone and ($H_2L^5 = C_{30}H_{28}N_2O_2$) from Ethylenediamine with Benzoin.



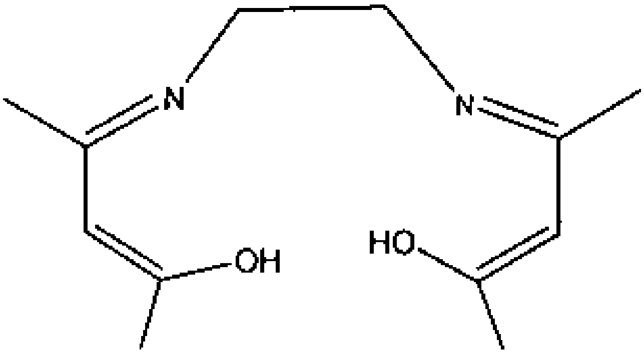
Structure of (H_2L^1) ligand



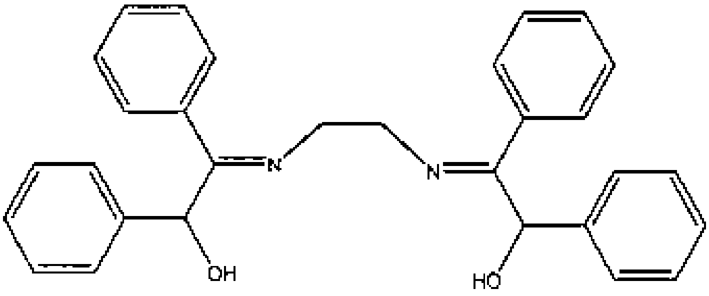
Structure of (H_2L^2) ligand



Structure of (H_2L^3) ligand



Structure of (H_2L^4) ligand



Structure of (H_2L^5) ligand

2.3. The synthesis of metal Complexes:

The Schiff base complexes under investigation were synthesized by one method, an ethanolic solution of Schiff base ligands (1mol) was refluxed with oxo-metal salt (1 mol) in ethanol for about 6h. The volume of the solution was reduced to one third and the concentrate cooled at 0C°. The solid complex formed was filtered, washed with ethanol and dried in vacuo.[19]

2.4. The studies on ligands and its metal Complexes

2.4.1. Physical techniques measurements and spectroscopic

2.4.1.1. Elemental analysis

All the Schiff base ligands and their complexes under investigation, were subjected to (C, H and N) elemental analysis which performed at micro analytical center faculty of Science-Cairo University (Egypt) and the Complexation at laboratories of RASCO company Libya.

2.4.1.2. Mass spectra

Mass spectra were recorded with a Shimadzo 24 GCMS-QP 1000EX, at micro analytical center University of Cairo.

2.4.1.3. Melting point

The melting point of ligands and their complexes were measured in capillary tubes Philip Haris, Shenston-England, serial No.B/A-211, at chemistry department. Faculty of Science, Garyounis (UOG) University.

2.4.1.4. Magnetic moment measurements

The magnetic moment measurements of complexes under investigation were determined by using a modified Goy type magnetic balance Herts SG8 SHJ, England, at Cairo. University.

2.4.1.5 Infrared spectra

The IR spectra of the Schiff base ligands and their complexes under investigation were recorded using BRUKER IFS 25 spectrophotometer using KBr Discs, at the region 400-4000cm^{-1} at Advanced Laboratory for Chemical Analysis, Al Fatah University Tripoli and micro analytical center faculty of Science-Cairo University (Egypt)

2.4.1.6 Electronic spectra

In 10^{-3} M solution of Schiff base ligands and their complexes were prepared by dissolving the accurate weight of the compound in 25 cm^3 of DMF or $CHCl_3$. The spectra of the obtained solutions were scanned within the visible and ultraviolet measured using a 640S UV-vis spectrophotometer using 1cm matched silica cells, at Advanced Laboratory for Chemical Analysis, Al Fatah University Tripoli, and Micro Analytical Center, Cairo University, Giza-Egypt.

Chapter (III)

Results and discussion

3. Results and discussion:

3.1. The studies on Schiff base ligands

The Schiff base ligands under investigation were formed by the condensation reaction of Ethylenediamine with Benzoylacetone obtained H_2L^1=[$C_{22}H_{24}N_2O_2$], 1,6-hexanediamine with Benzoylacetone obtained H_2L^2= [$C_{26}H_{32}N_2O_2$], 1, 6-hexanediamine with Acetylaceton obtained H_2L^3= [$C_{16}H_{28}N_2O_2$], Ethylenediamine with Acetylaceton obtained H_2L^4= [$C_{12}H_{20}N_2O_2$] and Ethylenediamine with Benzoin obtained H_2L^5= [$C_{30}H_{28}N_2O_2$]. The Schiff base ligands were subjected to elemental analysis, Infrared, Mass spectroscopy and Electronic spectroscopy to identify their structures.

3.1.1 Elemental analysis of the Schiff base ligands

Elemental analysis results of the ligands are given in table (1); the results are in good agreements for the ligands and correlated well with the calculated.

3.1.2. Infrared spectrum of the Schiff base ligands

The infrared spectral data is shown in Table (2).The assignment of The infrared bands the Schiff base are in good agreement with our expected Fig.1-5 the band at 3418 $cm^{-1}$is attributed to the OH group [20-22]. A band at 1597-1657 $cm^{-1}$are due to v(C=N) vibration [23-28].

The band at 1547-1657 $cm^{-1}$are assigned to ν(C=C) stretching frequency [21,23,28,29]. The C—O stretching absorption are seen at range 1263-1354cm^{-1}[21,30,31].

The infrared spectrum of the ligand H_2L^1= $C_{22}H_{24}N_2O_2$. Showed a strong band at 1606 cm^{-1} is assigned to ν(**C**=N) azomethine group stretching. The absorption band in the region 1547cm^{-1} is assigned to the ν(C=C) vibration. Finally strong band at 1288 cm^{-1} is attributed to the existence of ν(C—O).

The infrared spectrum of the ligand H_2L^2= $C_{26}H_{32}N_2O_2$. Showed a strong band at 1597cm^{-1} is assigned to ν(**C**=N) azomethine group stretching. The absorption band in the region 1582cm^{-1} is assigned to the ν(C=C) vibration. Finally strong band at 1289 cm^{-1} is attributed to the existence of ν(C—O).

The infrared spectrum of the ligand H_2L^3= $C_{16}H_{28}N_2O_2$. Showed a strong band at 1605 cm^{-1} is assigned to ν(C=N) azomethine group stretching. The absorption band in the region 1564cm^{-1} is assigned to the ν(C=C) vibration. Finally strong band at 1354cm^{-1} is attributed to the existence of ν(C—O).

The infrared spectrum of the ligand H_2L^4= $C_{12}H_{20}N_2O_2$. Showed a strong band at 1609 cm^{-1} is assigned to ν(**C**=N) azomethine group stretching. The absorption band in the region 1579cm^{-1} is assigned to the ν(C=C) vibration. Finally strong band at 1353cm^{-1} is attributed to the existence of ν(C—O).

The infrared spectrum of the ligand H_2L^5- $C_{30}H_{28}N_2O_2$. Showed a strong band at 1681cm^{-1} which is assigned to ν(C=N) azomethine group stretching. The absorption band in the region 1657cm^{-1} was assigned to the ν(C=C) vibration. The band at 3418 cm^{-1} is due to hydrogen bonding of (OH) group ·Finally the strong band at 1263cm^{-1} was attributed to the existence of ν(C—O).

3.1.3 Electronic spectrum of Schiff base ligands

The electronic spectrum of all ligands were recorded in 10^{-3}M DMF are presented in table (3). Fig.(6-10). The Schiff bases were characterized mainly by absorption bands in the region (28490-47146cm^{-1}). The bands were attributed to aromatic $\pi - \pi^*$ or azomethine group at range1263-1354cm^{-1} [30,32].

3.1.4 Mass spectrum of Schiff base ligands

The mass spectra of shiff base "ligands" are given in the figures (11-15) and Schemes (1-5). The compound peaks are in good agreements with their empirical formula as indicated from elemental analyses. The other peaks represent fragments of the ligands.

The mass spectrum of (H_2L^1) showed the following fragmentation scheme the molecular ion peak at m/z 348 is corresponding to the molecular weight of ($C_{22}H_{24}N_2O_2$). The peak at m/z 304 which is the base peak is assigned to ($C_{20}H_{20}N_2O$)$^+$ fragment, A peak at m/z 119 and 77 are due to (C_8H_8O)$^+$ and (C_6H_6)$^+$ fragment.

The mass spectrum of (H_2L^2) showed the following fragmentation scheme 2. The molecular ion peak at m/z 404 is corresponding to the molecular weight of ($C_{26}H_{32}N_2O_2$). The peak at m/z 299 is due to ($C_{19}H_{28}N_2O$)$^+$ fragment. A peak at m/z 244 is due to ($C_{15}H_{20}N_2O$)$^+$ fragment. A peak at m/z 188 is assigned to ($C_{12}H_{15}NO$)$^+$ fragment. The peak at m/z 105 which is the base peak is assigned to (C_7H_6O)$^+$ fragment.

The mass spectrum of (H_2L^3) showed the following fragmentation scheme 3. The molecular ion peak at m/z 280 is corresponding to the molecular weight of ($C_{16}H_{28}N_2O_2$). The peak at m/z 237 which is assigned to ($C_{14}H_{26}N_2O$)$^+$ fragment.. The peak at m/z 180 which is the base peak is assigned to ($C_{11}H_{20}NO$)$^+$ fragment. A peak at m/z 138 is due to ($C_9H_{17}N$)$^+$. A peak at m/z 98 is due to (C_5H_8NO)$^+$ fragment. A peak at m/z 55 is due to (C_4H_{10})$^+$ fragment.

The mass spectrum of (H_2L^4) showed the following fragmentation scheme 4. The molecular ion peak at m/z 224 is corresponding to the molecular weight of ($C_{12}H_{20}N_2O_2$) fragment. The peak at m/z 175 which is the base peak is assigned to ($C_{11}H_{18}N_2$)$^+$ fragment. A peak at m/z 105 is due to $(C_6H_{10}N_2)^+$ fragment. A peak at m/z 51 is assigned to $(C_3H_4N)^+$ fragment

The mass spectrum of (H_2L^5) in scheme 5 showed the followings fragmentation. The molecular ion peak at m/z 448 is corresponding to the molecular weight of $(C_{30}H_{28}N_2O_2)$. The peak at m/z 174 is assigned to $(C_{10}H_{10}N_2O)^+$ fragment. A peak at m/z 105 is the base peak due to $(C_7H_6O)^+$ fragment . A peak at m/z 77 is due to $(C_6H_8)^+$ fragment

3.2 Studies of Schiff base complexes

In this section, we focus our attention on the analysis of the entire complexes, the analysis included the elemental analysis, magnetic susceptibility, IR and UV-vis spectra.

3.2.1 Elemental analysis

Analytical results for complexes. C, H and N, all the synthesized compounds are given in table (4).

3.2.2 Infrared spectra of the complexes

The IR spectra of the complexes are found in table (5) and Figures (16-26).the IR spectra of the complexes compared with those of the ligand indicate that the ν (C=N) band at (1563 to 1652 cm^{-1}) shifted to lower or higher frequencies at ranging from 1263-1354cm^{-1}[25,26,30,33,34]., band at (1231 to 1362 cm^{-1}) for ν(C—O) stretching[30,31,35-37] and region at

(3306 to 3446 cm^{-1}) for O-H group stretching[22,28]. Conclusive evidence of bonding is also shown by the observation that new bands in the spectra of the metal complexes appear at (473 to 570 cm^{-1}) and (417 to 546 cm^{-1}) these are assigned to ν(M—O) and ν(M—N) stretching vibrations and are not observed in the spectra of the ligands [22,27,28,31,37]. and new band region at (818 to 987cm^{-1}) is attributed to the existence of M=O band [22, 38].

The infrared spectrum of the $C_{22}H_{22}N_2O_4U.8H_2O$ Complex showed a peak at 1600cm^{-1} which is assigned to the ν(C=N) stretching frequency.. The spectra of the complex exhibited the presence of a band at 1284cm^{-1} which is due to the ν(C—O) vibration. The broad band at 3445 cm^{-1} is attributed to the presence of (OH) group of water molecules. The coordination mode of ligand is further supported by the appearance of new bands in the range of 507 to 434cm^{-1} indicating the formation of (U—O) and (U—N) bonds respectively. Finally new strong band at 919 cm^{-1} is attributed to the existence of U=O band.

The infrared spectrum of the $C_{22}H_{22}N_2O_4Mo.4H_2O$ Complex showed a strong peak at 1604cm^{-1} which is assign to the ν(C=N) stretching frequency.. The spectra of the complex exhibited the presence of a band at 1289cm^{-1} which is due to the ν(C—O) vibration. The strong band at 3306 cm^{-1} is attributed to the presence of (OH) group of water molecules. The coordination mode of ligand is further supported by the appearance of new bands in the range of 570 to 546 indicating the formation of (M_O—O) and (M_O—N) bonds respectively. Finally new band at 900 cm^{-1} is attributed to the existence of Mo=O band.

The infrared spectrum of the $C_{22}H_{22}N_2O_3V$. H_2O Complex showed a strong peak at 1604cm^{-1} which is assign to the ν(C=N) stretching frequency. The spectra of the complex exhibited the presence of a band at 1289cm^{-1} which is due to the ν(C—O) vibration. The broad band at 3446 cm^{-1} is attributed to the presence of (OH) group of water molecules. The coordination mode of ligand is further supported by the appearance of new bands in the range of 570 to 504cm^{-1} indicating the formation of (V—O) and (V—N) bonds respectively. Finally new band at 884 cm^{-1} is attributed to the existence of V=O band.

The infrared spectrum of the $C_{22}H_{22}N_2O_3Zr.2H_2O$ Complex showed a strong peak at 1601cm^{-1} which is assign to the ν(C=N) stretching frequency.. The spectra of the complex exhibited the presence of a band at 1288cm^{-1} which is due to the ν(C—O) vibration. The coordination mode of ligand is further supported by the appearance of new bands in the range of 570 to 450cm^{-1} indicating the formation of (Zr—O) and (Zr—N) bonds respectively. Finally new a strong band at 818 cm^{-1} is attributed to the existence of Zr=O band.

The infrared spectrum of the $C_{26}H_{30}N_2O_4U.6H_2O$ Complex showed a peak at 1605cm^{-1} which is assign to the ν(C=N) stretching frequency. The spectra of the complex exhibited the presence of a band at 1278cm^{-1} which is due to the ν(C—O) vibration. The broad band at 3425 cm^{-1} is attributed to the presence of (OH) group of water molecules. The coordination mode of ligand is further supported by the appearance of new bands in the range of 513 to 435cm^{-1} indicating the formation of (U—O) and (U—N) bonds respectively. Finally new strong band at 924 cm^{-1} is attributed to the existence of U=O band.

The infrared spectrum of the $C_{26}H_{30}N_2O_3Zr.6H_2O$ Complex showed a strong peak at 1563cm^{-1} which is assign to the ν(C=N) stretching frequency.. The spectra of the complex exhibited the presence of a band at 1245cm^{-1} which is due to the ν(C—O) vibration. The band at 3419 cm^{-1} is attributed to the presence of (OH) group of water molecules The coordination mode of ligand is further supported by the appearance of new bands in the range of 476 to 420cm^{-1} indicating the formation of (Zr—O) and (Zr—N) bonds respectively. Finally new a strong band at 942 cm^{-1} is attributed to the existence of Zr=O band.

The infrared spectrum of the $C_{16}H_{26}N_2O_4U.2H_2O$ Complex showed a peak at 1603cm^{-1} which is assign to the ν(C=N) stretching frequency.. The spectra of the complex exhibited the presence of a band at 1328cm^{-1} which is due to the ν(C—O) vibration. The broad band at 3424 cm^{-1} is attributed to the presence of (OH) group of water molecules. The coordination mode of ligand is further supported by the appearance of new bands in the range of 545 to 417 indicating the formation of (U—O) and (U—N) bonds respectively. Finally new strong band at 920 cm^{-1} is attributed to the existence of U=O band.

The infrared spectrum of the $C_{16}H_{26}N_2O_3Zr.2H_2O$ Complex showed a peak at 1601cm^{-1} which is assign to the ν(C=N) stretching frequency.. The spectra of the complex exhibited the presence of a band at 1342cm^{-1} which is due to the ν(C—O) vibration. The broad band at 3374 cm^{-1} is attributed to the presence of (OH) group of water molecules. The coordination mode of ligand is further supported by the appearance of new bands in the range of 473 to 422cm^{-1} indicating the formation of (Zr—O) and (Zr—N) bonds respectively. Finally new strong band at 818 $cm^{-1}$is attributed to the existence of Zr=O band.

The infrared spectrum of the $C_{12}H_{18}N_2O_4U.H_2O$ Complex showed a peak at 1600cm^{-1} which is assign to the ν(C=N) stretching frequency.. The spectra of the complex exhibited the presence of a band at 1337cm^{-1} which is due to the ν(C—O) vibration. The broad band at 3422 cm^{-1} is attributed to the presence of (OH) group of water molecules. The coordination mode of ligand is further supported by the appearance of new bands in the range of 505 to 432cm^{-1} indicating the formation of (U—O) and (U—N) bonds respectively. Finally new strong band at 930 cm^{-1} is attributed to the existence of U=O band.

The infrared spectrum of the $C_{12}H_{18}N_2O_3V.6H_2O$ Complex showed a strong peak at 1556cm^{-1} which is assign to the ν(C=N) stretching frequency. The spectra of the complex exhibited the presence of a band at 1362cm^{-1} which is due to the ν(C—O) vibration. The broad band at 3396 cm^{-1} is attributed to the presence of (OH) group of water molecules. The coordination mode of ligand is further supported by the appearance of new bands in the range of 542 to 424 cm^{-1} indicating the formation of (V—O) and (V—N) bonds respectively. Finally new band at 938 cm^{-1} is attributed to the existence of V=O band

The infrared spectrum of the $C_{30}H_{26}N_2O_4Mo.H_2O$ Complex showed a strong peak at 1652cm^{-1} which is assign to the ν(C=N) stretching frequency.. The spectra of the complex exhibited the presence of a band at 1231cm^{-1} which is due to the ν(C—O) vibration. The band at 3314 cm^{-1} is attributed to the presence of (OH) group of water molecules. The coordination mode of ligand is further supported by the appearance of new bands in the range of 555 and 460 cm-^{1} indicating the formation of (M_O—O) and (M_O—N) bonds respectively. Finally new strong band at 987 cm^{-1} is attributed to the existence of Mo=O band

3.2.3 Electronic spectra and magnetic measurements of complexes

The magnetic moment values, μ_{eff} measured for all complexes at room temperature (298 K) and the recorded in the solid state are given in table (6). The electronic spectra of complexes in 10^{-3} M DMF or chloroform shows a d-d transition at region (465 - 835)nm and the show several bands at region (325 - 379)nm transition in the aromatic ring or azomethine group. $\pi \rightarrow \pi^*$ assigned to the data are recorded in table (6). [39,40-43]

3.2.3.1 Electronic spectra and magnetic measurements of complex square pyramidal structures:

The electronic spectrum of the vanadyl complex $C_{22}H_{22}N_2O_3V$. H_2O exhibits bands at 11976 $cm^{-1}$and 21505cm^{-1} which can be assigned to $^2B_2 \rightarrow {}^1A_1$ and $^2B_{2(P)} \rightarrow E$ transition' And the band at 26385 cm^{-1} is assigned to the $\pi \rightarrow \pi^*$ transition of the aromatic ring or azomethine group which agreeable with the reported results. These transitions, as well as the measured value of magnetic moment (1.74 B.M) suggest square pyramidal stereochemistry of the compound.

The electronic spectrum of the vanadyl complex $C_{12}H_{18}N_2O_3V$. $6H_2O$ exhibits bands at 13245 $cm^{-1}$and 21276cm^{-1} which can be assigned to $^2B_2 \rightarrow {}^1A_1$ and $^2B_{2(P)} \rightarrow E$ transition' And the band at 30769 cm^{-1} is assigned to the $\pi \rightarrow \pi^*$ transition of the aromatic ring or azomethine group which agreeable with the reported results. these transition, as well as the measured value of magnetic moment (1.76 B.M) suggest square pyramidal stereochemistry of the compound.

The Zirconyl Complexes are Dia magnetic moment, and square Pyramidal geometry.

3.2.3.2 Electronic spectra and magnetic measurements of complexs octahedral structures:

The Uranyl and molabadat Complexes are Dia magnetic moment, and octahedral geometry [30,32].

Table (1) *Elemental analysis data, colors and melting points of Schiff bases under investigation.*

The ligands	M.Wt	Found (Calc.) C%	H	N%	M.P	Color
$C_{22}H_{24}N_2O_2$	348.47	75.16 (75.82)	6.67 (6.95)	8.11 (8.04)	177C°	white Crystal
$C_{26}H_{32}N_2O_2$	404.59	76.25 (77.18)	7.11 (7.98)	6.97 (6.92)	99C°	white Crystal
$C_{16}H_{28}N_2O_2$	280.44	68.09 (68.52)	9.74 (10.08)	10.14 (9.99)	110C°	bright Brown
$C_{12}H_{20}N_2O_2$	224.32	63.68 (64.25)	8.58 (9.00)	12.53 (12.48)	85C°	white
$C_{30}H_{28}N_2O_2$	448.55	80.10 (80.33)	5.92 (6.29)	6.35 (6.25)	95C°	orange Yellow

Table (2) *Infrared spectra of ligands under investigation.*

Ligands	**ν (C=N) cm^{-1}**	**ν (C-O) cm^{-1}**	**ν (OH) cm^{-1}**	**ν (C=C) cm^{-1}**
$C_{22}H_{24}N_2O_2$	1606	1288	—	1547
$C_{26}H_{32}N_2O_2$	1597	1289	—	1582
$C_{16}H_{28}N_2O_2$	1605	1354	—	1564
$C_{12}H_{20}N_2O_2$	1609	1353	—	1579
$C_{30}H_{28}N_2O_2$	1657	1263	3418	1657

Table (3) *Electronic spectra of ligands under investigation*

Ligands	λ **nm**	ε **(L.mol^{-1}cm^{-1})**	**ν cm^{-1}**	**Transitions**
$C_{22}H_{24}N_2O_2$	351	0.974	28490	$\pi \rightarrow \pi^*$
$C_{26}H_{32}N_2O_2$	343	2.51	29154	$\pi \rightarrow \pi^*$
$C_{16}H_{28}N_2O_2$	311	2.18	32154	$\pi \rightarrow \pi^*$
$C_{12}H_{20}N_2O_2$	322	2.128	31055	$\pi \rightarrow \pi^*$
$C_{30}H_{28}N_2O_2$	209	3.65	47146	$\pi \rightarrow \pi^*$

Table (4) *Elemental analysis, Color and M.P of Schiff base complexes under investigation.*

Complex	M.wt	color	m.p C°	Found (calc.)		
				C%	H%	N%
$C_{22}H_{22}N_2O_4U\ .8H_2O$	760.57	Orange	185	34.49 (34.74)	5.44 (5.04)	3.41 (3.68)
$C_{22}H_{22}N_2O_4Mo\ .4H_2O$	546.42	white Crystal	250	48.70 (48.36)	4.92 (5.53)	5.21 (5.13)
$C_{22}H_{22}N_2O_3V.\ H_2O$	447.38	Pale Green	220	60.22 (61.25)	6.79 (5.61)	6.26 (6.49)
$C_{22}H_{22}N_2O_3Zr.2H_2O$	489.67	white	150	54.22 (53.96)	4.88 (5.35)	6.17 (5.72)
$C_{26}H_{30}N_2O_4U.6H_2O$	780.64	Orange	214	40.04 (40.00)	5.95 (5.42)	3.42 (3.59)
$C_{26}H_{30}N_2O_3Zr.6H_2O$	617.84	white	177	51.20 (50.54)	6.48 (6.85)	5.07 (4.53)
$C_{16}H_{26}N_2O_4U.2H_2O$	584.44	Yellow	250	32.76 (32.88)	5.58 (5.17)	4.94 (4.79)
$C_{16}H_{26}N_2O_3Zr.2H_2O$	421.64	white	165	46.09 (45.58)	6.85 (7.17)	6.37 (6.64)
$C_{12}H_{18}N_2O_4U.H_2O$	510.18	Yellow	210	28.56 (28.24)	4.37 (3.95)	5.25 (5.49)
$C_{12}H_{18}N_2O_3V.6H_2O$	397.31	white	143	35.20 (36.28)	7.96 (7.61)	7.01 (7.05)
$C_{30}H_{26}N_2O_4Mo.\ H_2O$	592.48	Pale Yellow	190	61.16 (60.81)	4.83 (4.76)	4.87 (4.73)

Table (5) *Infrared bands assignments (cm^{-1}) of the Schiff base complexes under investigation.*

The complex	**ν(C=N) cm^{-1}**	**ν(C−O) cm^{-1}**	**ν(OH) cm^{-1}**	**ν(M=O) cm^{-1}**	**ν(M-O) cm^{-1}**	**ν(M-N) cm^{-1}**
$C_{22}H_{22}N_2O_4U\ .8H_2O$	1600	1284	3445	919	507	434
$C_{22}H_{22}N_2O_4Mo\ .4H_2O$	1604	1289	3306	900	570	546
$C_{22}H_{22}N_2O_3V.\ H_2O$	1604	1289	3446	884	570	504
$C_{22}H_{22}N_2O_3Zr\ .2H_2O$	1601	1288	___	818	570	450
$C_{26}H_{30}N_2O_4U.6H_2O$	1605	1278	3425	924	513	435
$C_{26}H_{30}N_2O_3Zr.6H_2O$	1563	1245	3419	942	476	420
$C_{16}H_{26}N_2O_4U.2\ H_2O$	1603	1328	3424	920	545	417
$C_{16}H_{26}N_2\ O_3Zr.2\ H_2O$	1601	1342	3374	818	473	422
$C_{12}H_{18}N_2\ O_4U.\ H_2O$	1600	1337	3422	930	505	432
$C_{12}H_{18}N_2\ O_3V.6\ H_2O$	1565	1362	3396	938	542	424
$C_{30}H_{26}N_2O_4Mo.\ H_2O$	1652	1231	3314	987	555	460

Table (6) *Electronic Spectra and magnetic moment of the Schiff base complexes under investigation*

The complex	λ nm	ε $L.mol^{-1}cm^{-1}$	ν cm^{-1}	μ_{eff} B.M	**Geometry**
$C_{22}H_{22}N_2O_3V\ .\ H_2O$	835 465 379	2.135 1.564 0.936	11976 21505 26385	1.74	Square pyramidal
$C_{12}H_{18}N_2O_3V.6H_2O$	755 470 325	2.354 2.106 1.098	13245 21276 30769	1.76	Square pyramidal

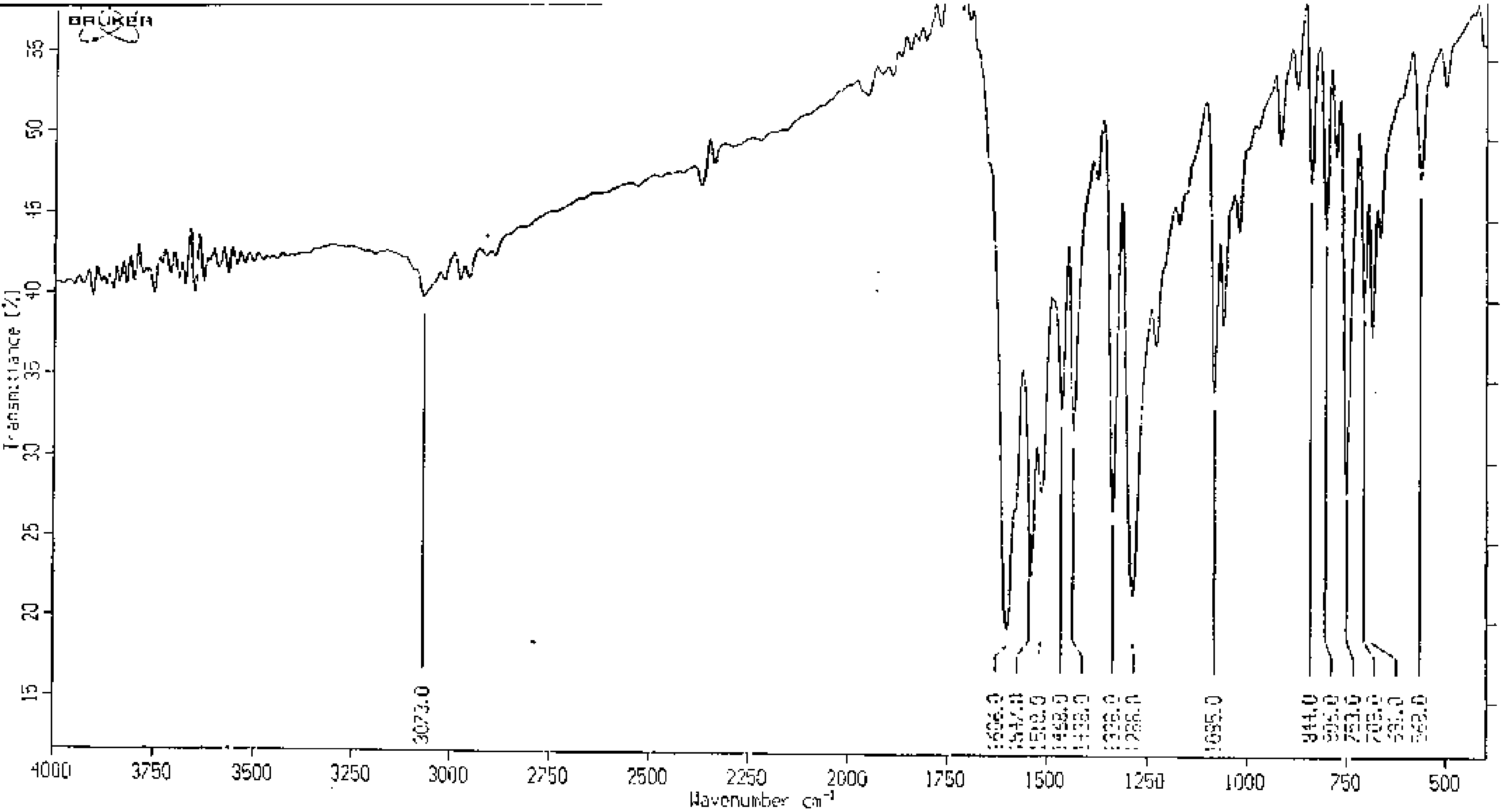


Fig.(1) IR spectrum of (H_2L^1) ligand

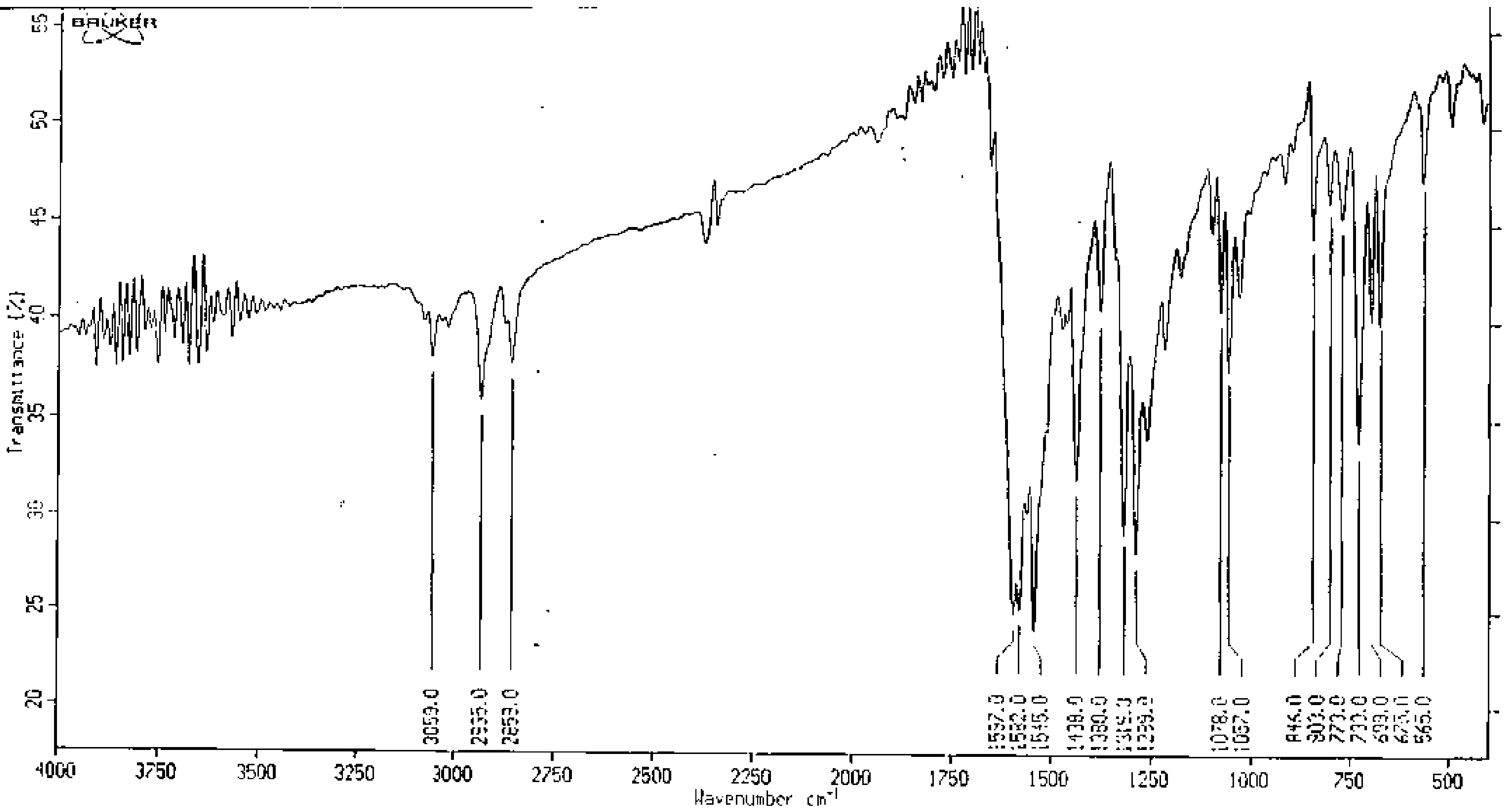


Fig.(2) IR spectrum of (H_2L^2) ligand

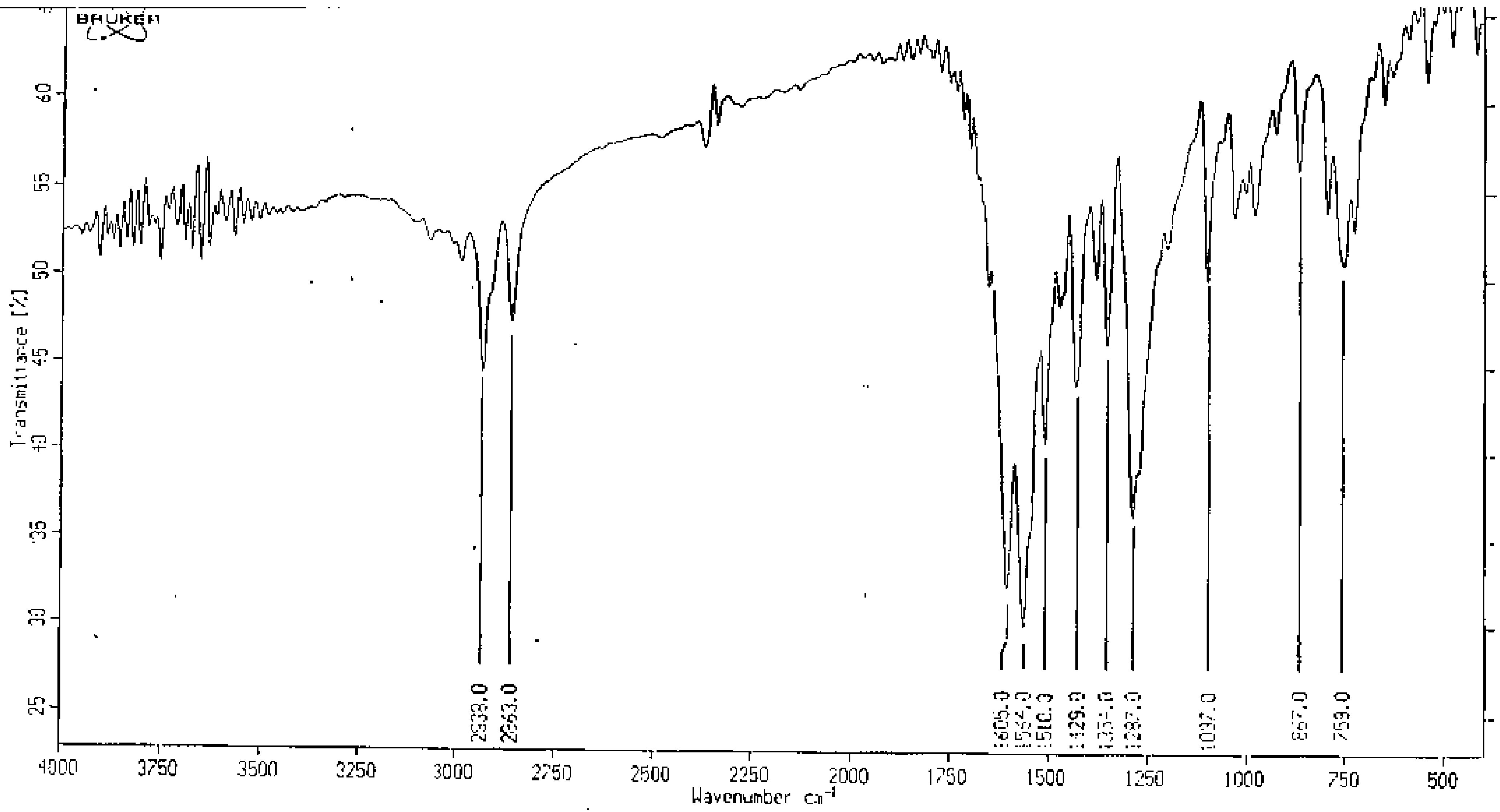


Fig.(3) IR spectrum of (H_2L^3) ligand

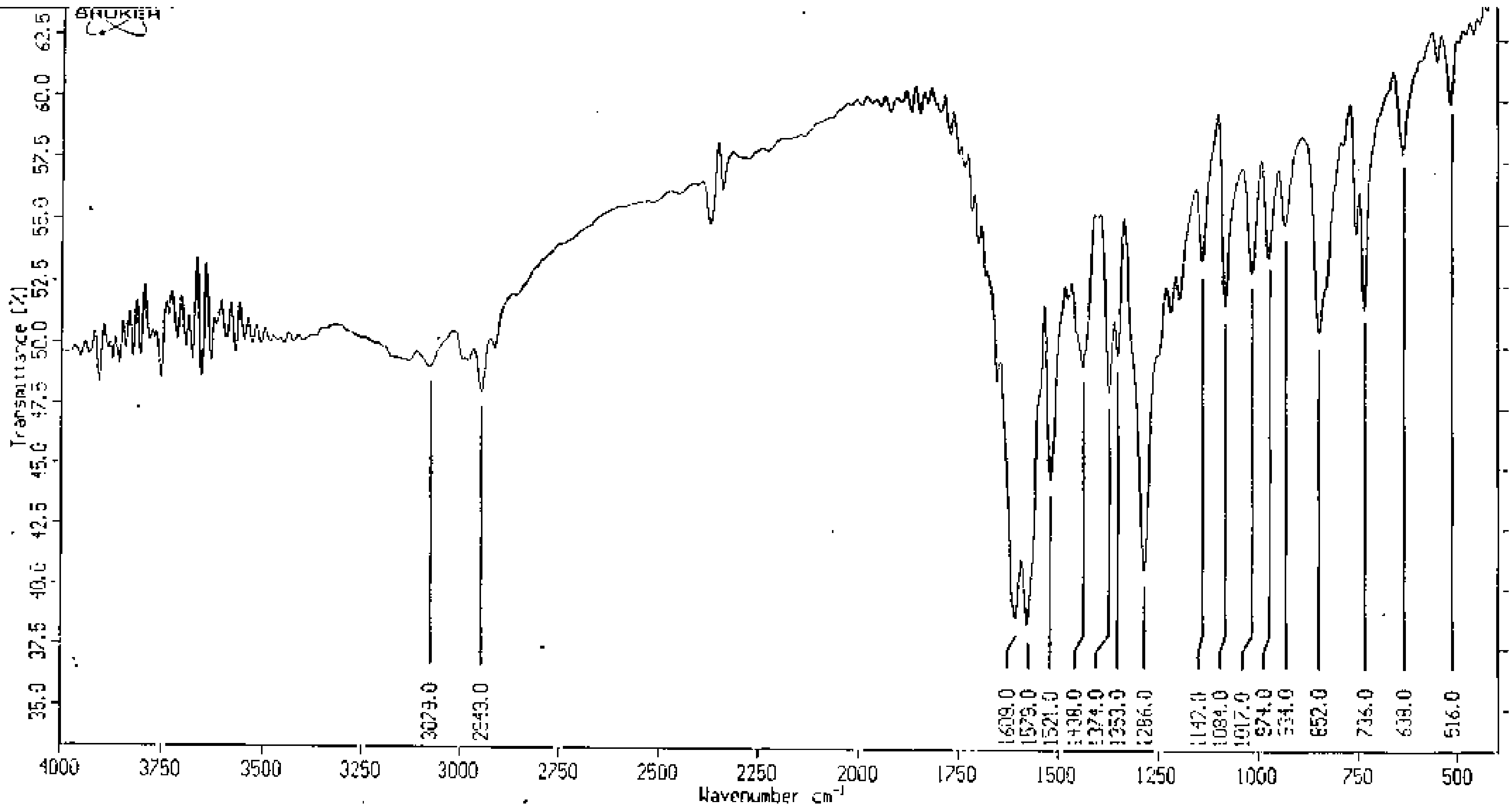


Fig.(4) IR spectrum of (H_2L^4) ligand

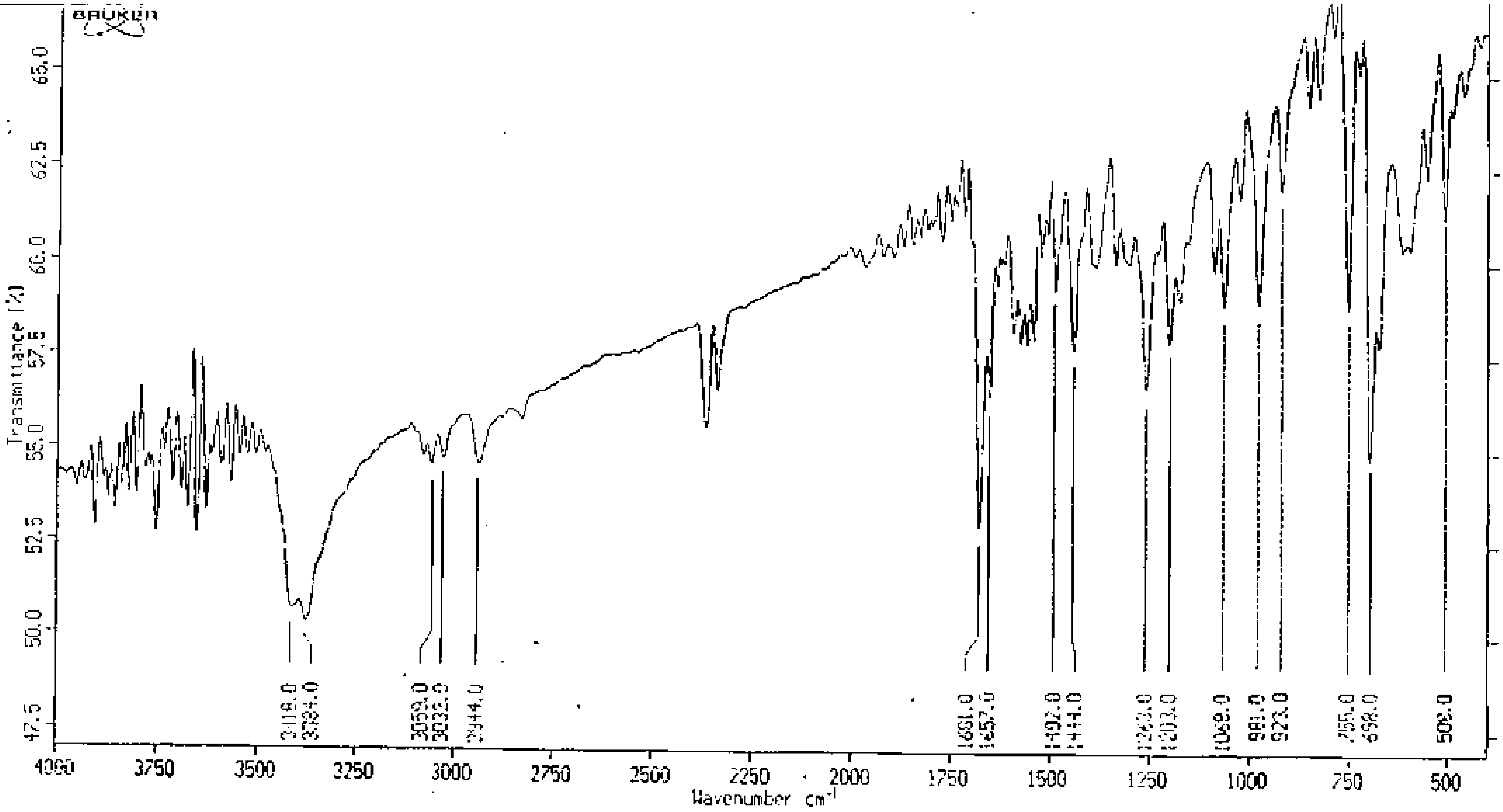


Fig.(5) IR spectrum of (H_2L^5) ligand

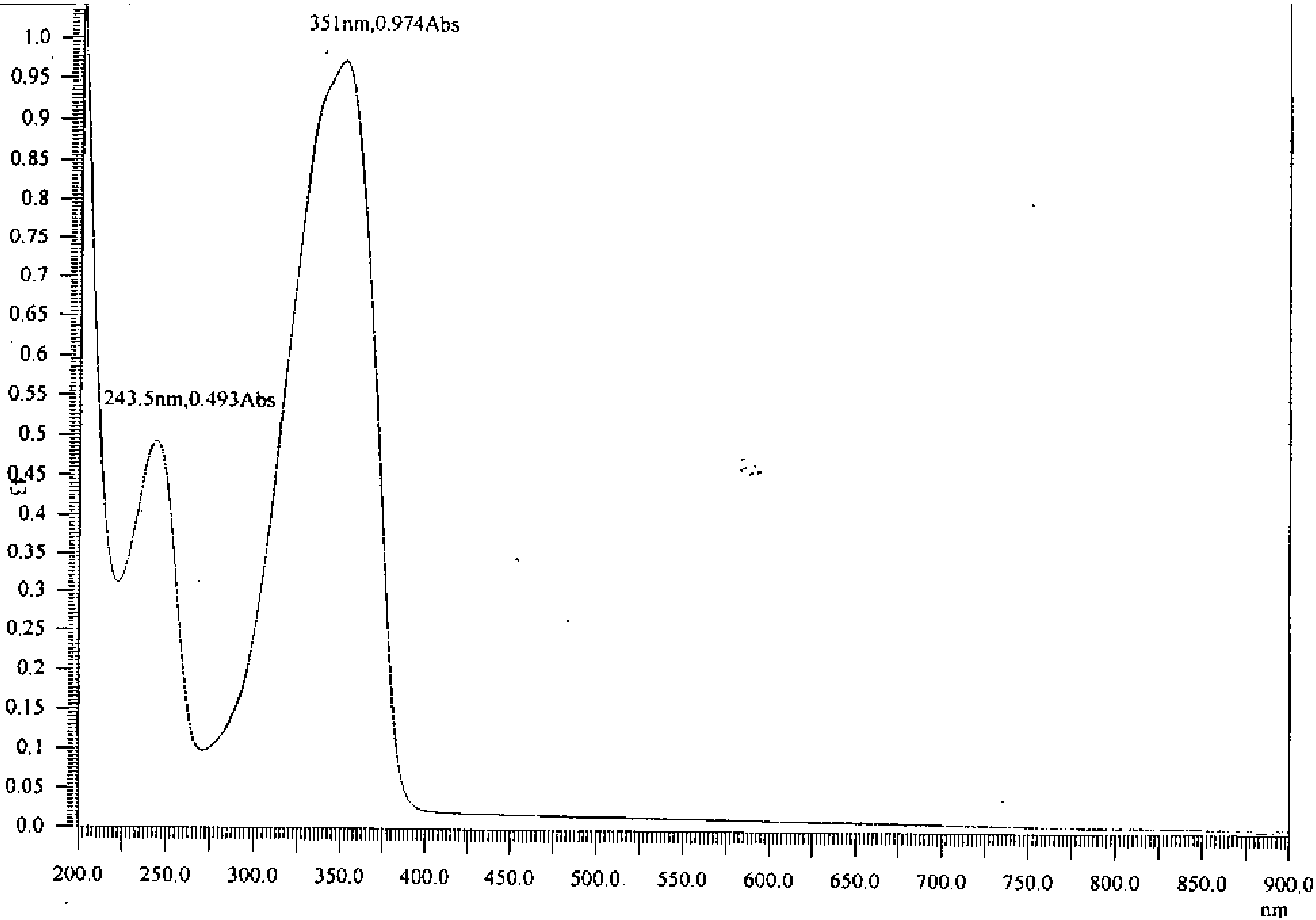


Fig.(6) Electronic spectra of (H_2L^1) ligand

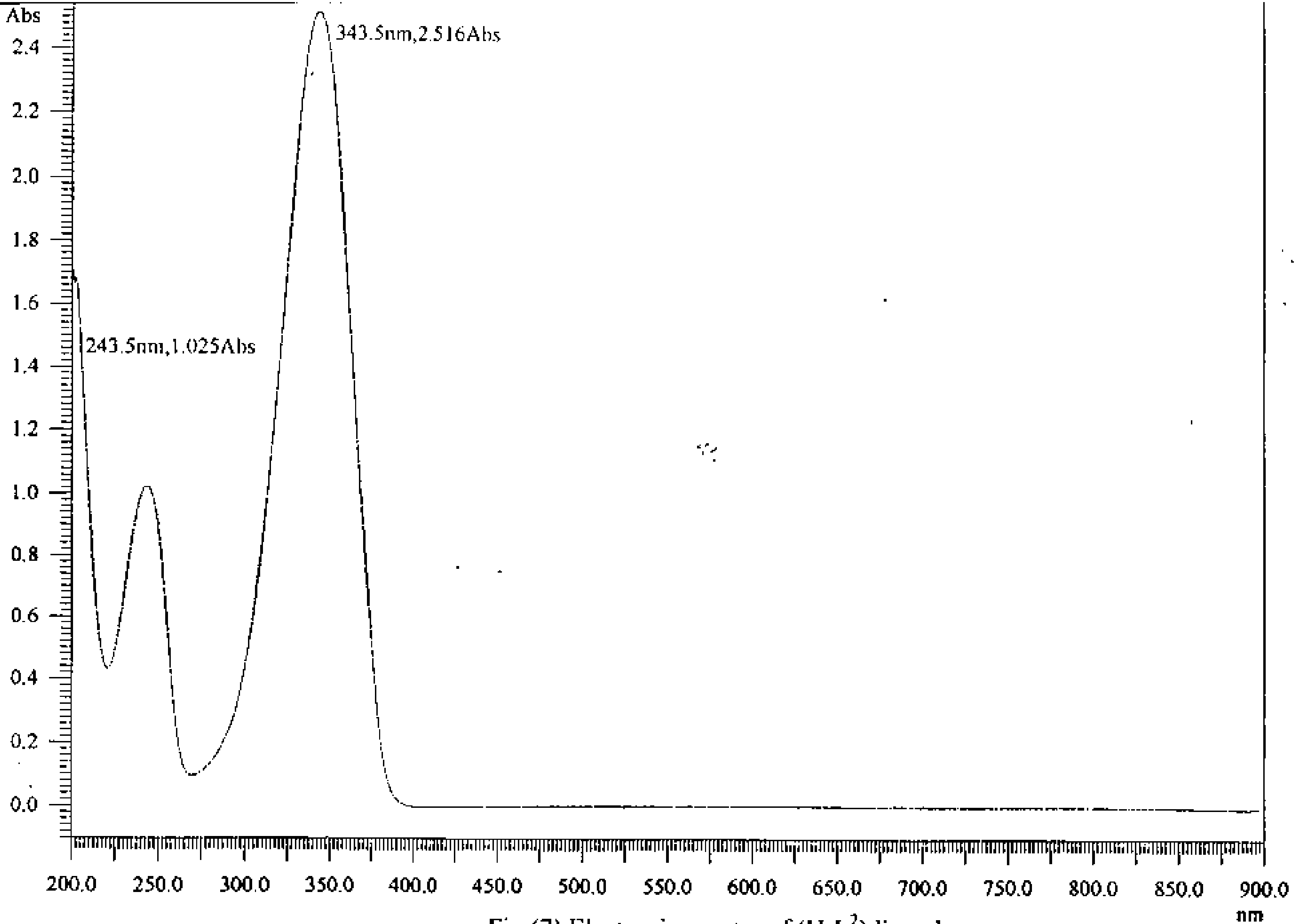


Fig.(7) Electronic spectra of (H_2L^2) ligand

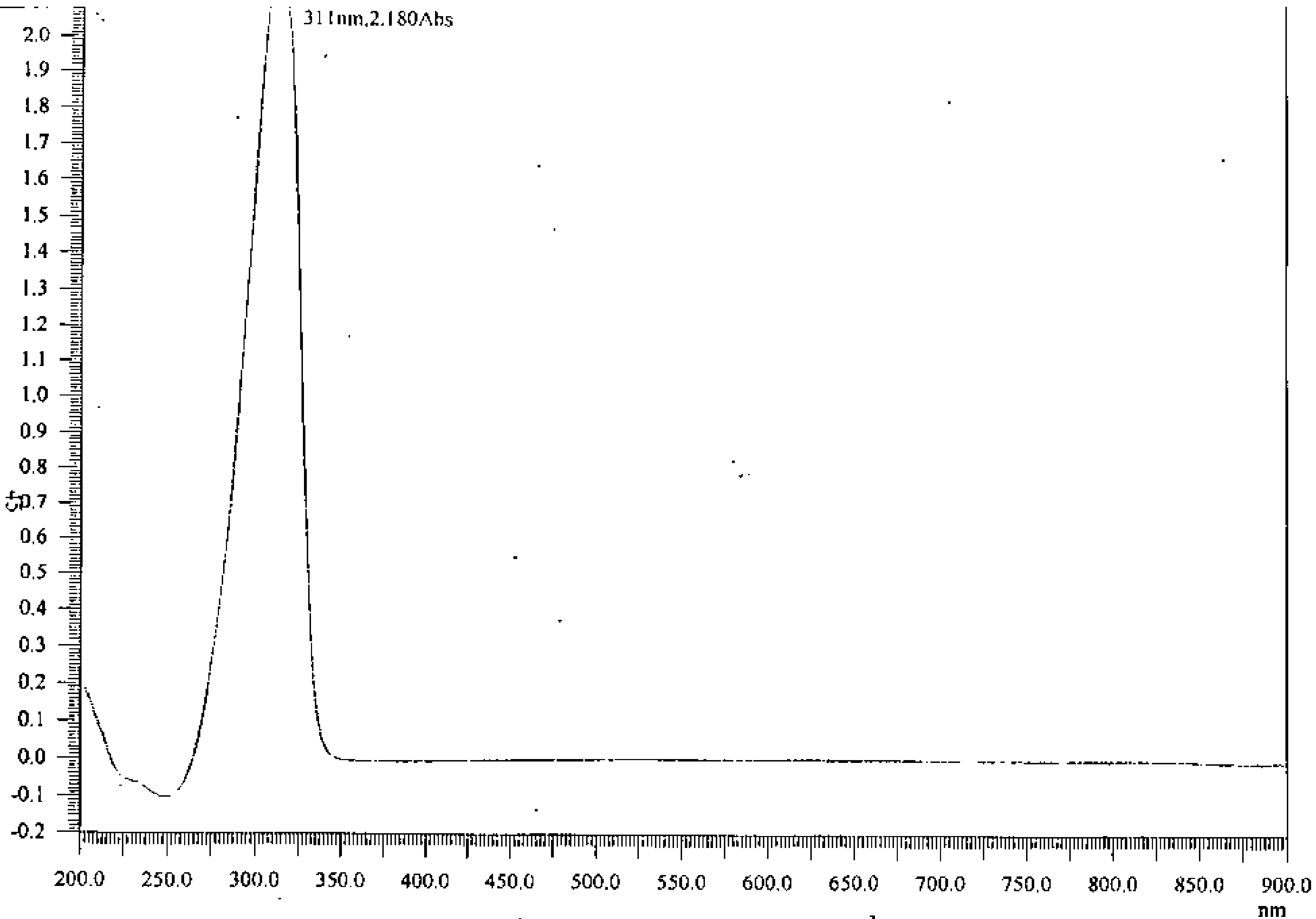


Fig.(8) Electronic spectra of (H_2L^3) ligand

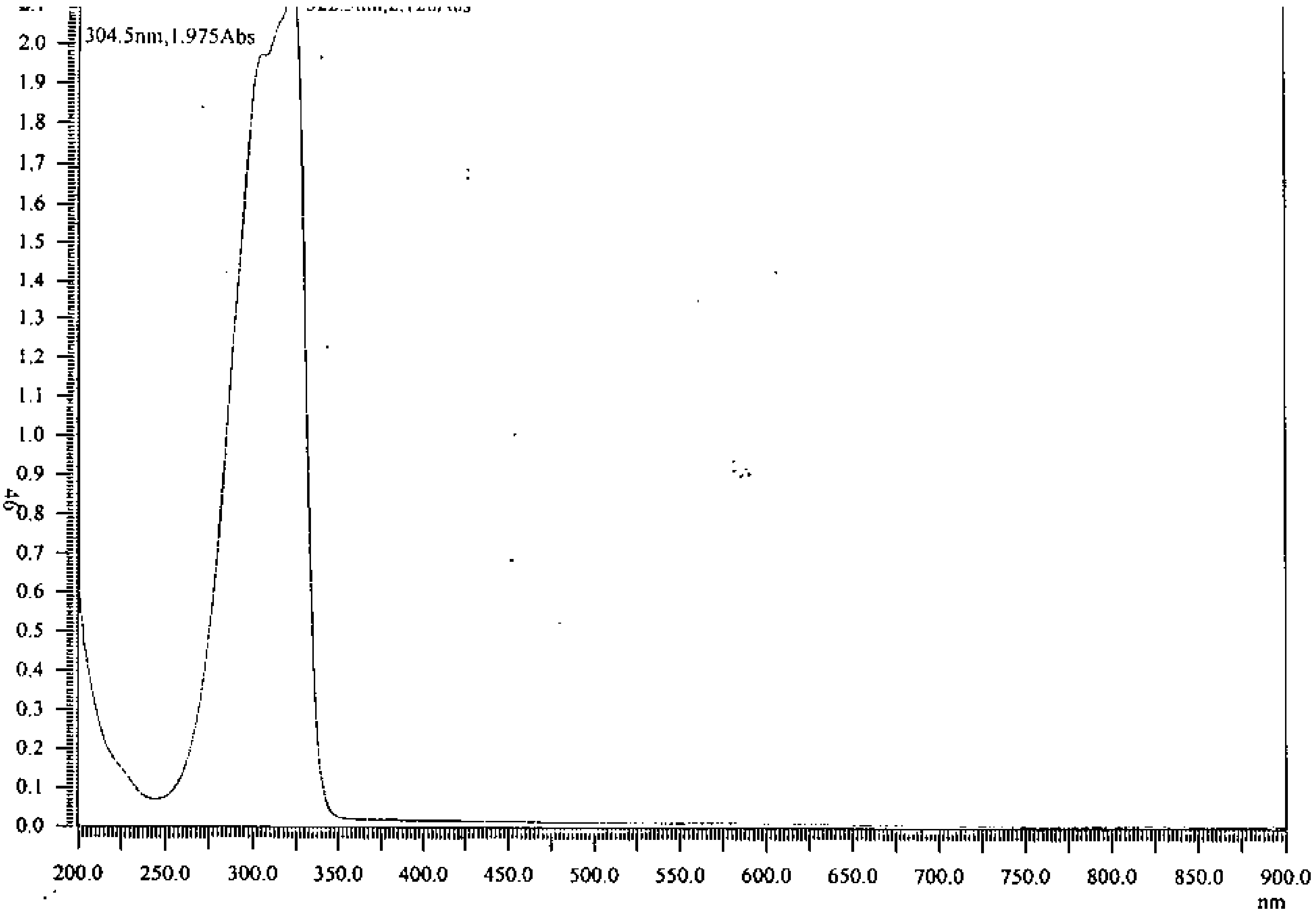


Fig.(9) Electronic spectra of (H_2L^4) ligand

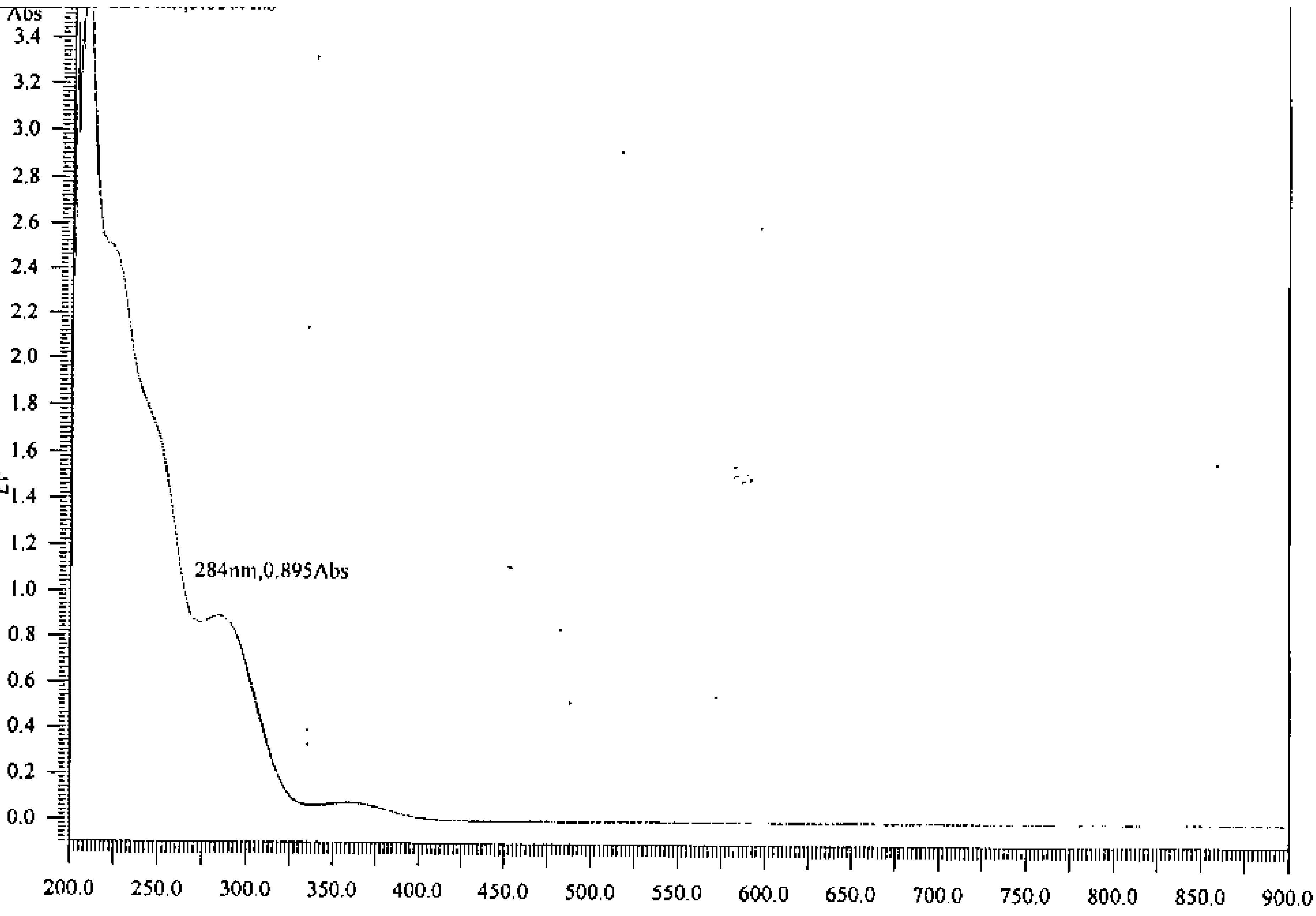


Fig.(10) Electronic spectra of (H_2L^5) ligand

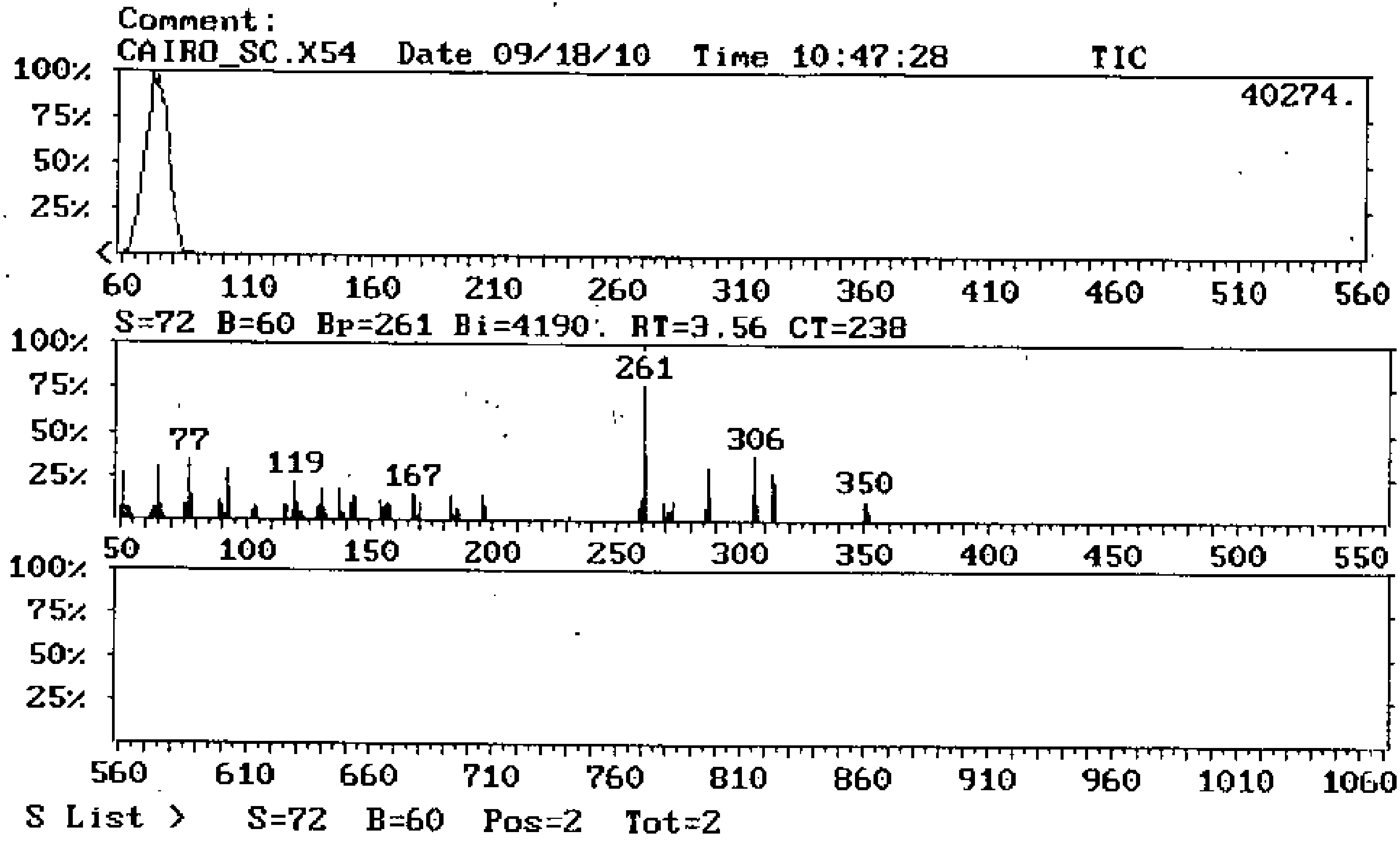


Fig.(11) Mass spectrum of (H_2L^1) ligand

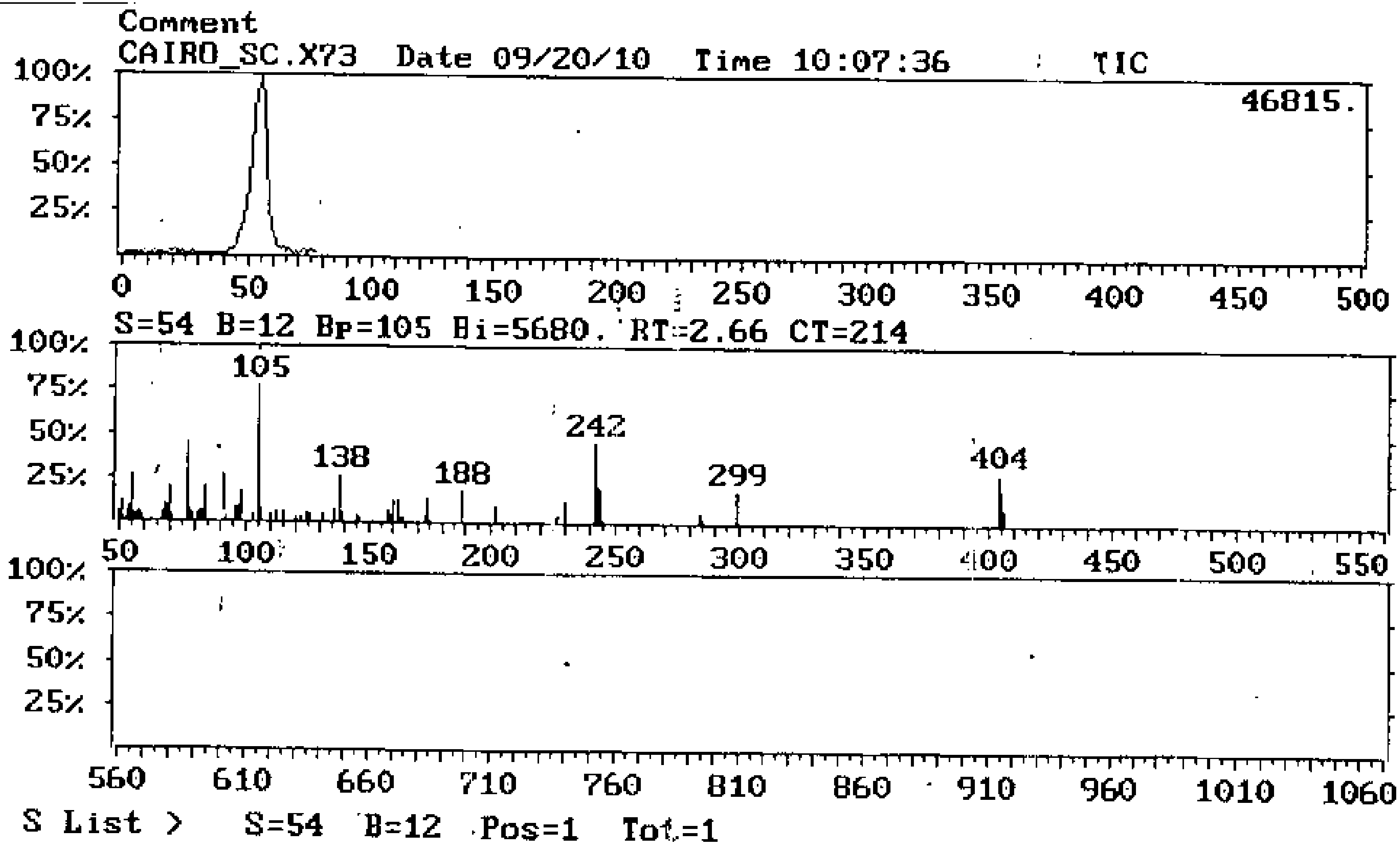


Fig.(12) Mass spectrum of (H_2L^2) ligand

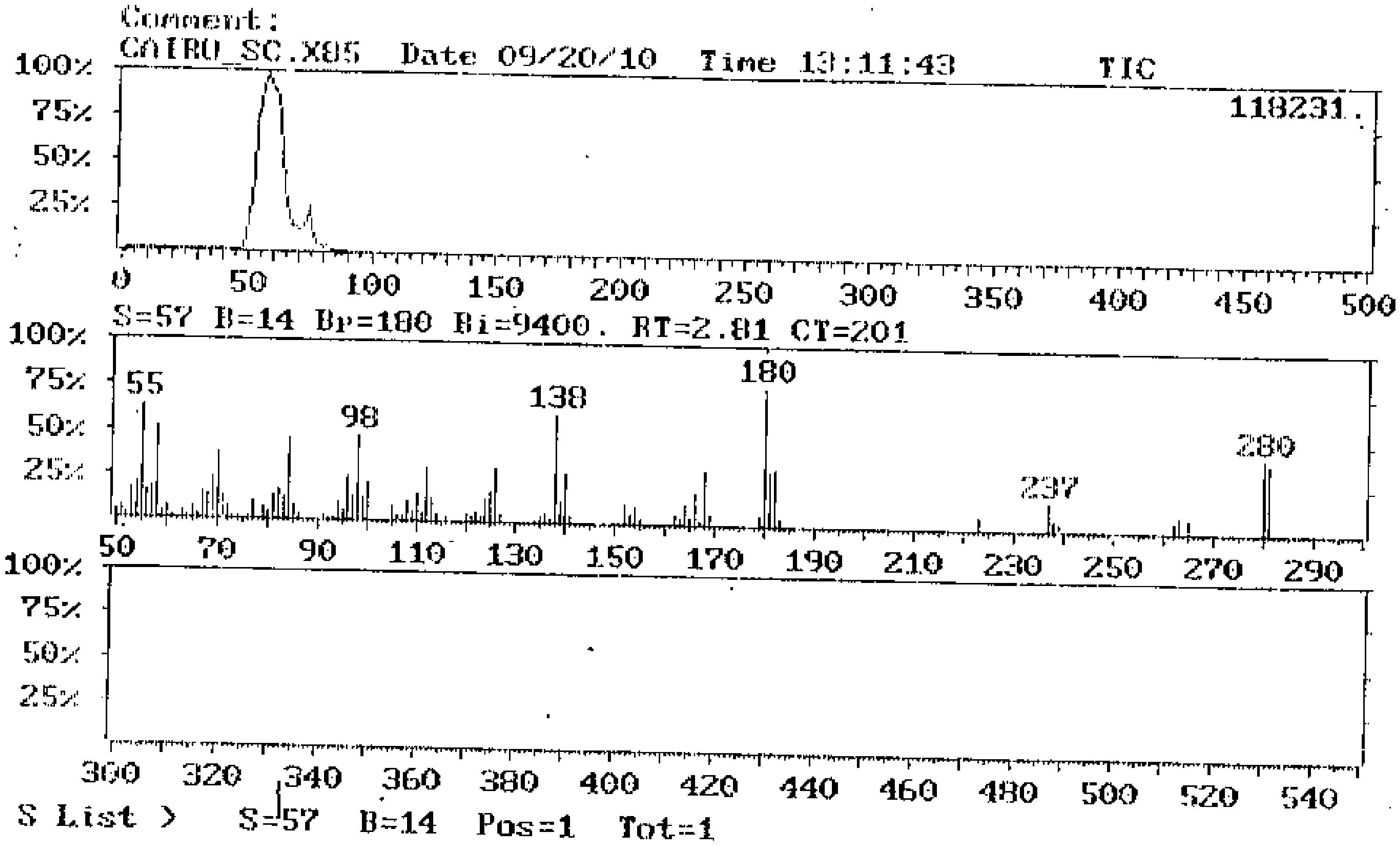


Fig.(13) Mass spectrum of (H_2L^3) ligand

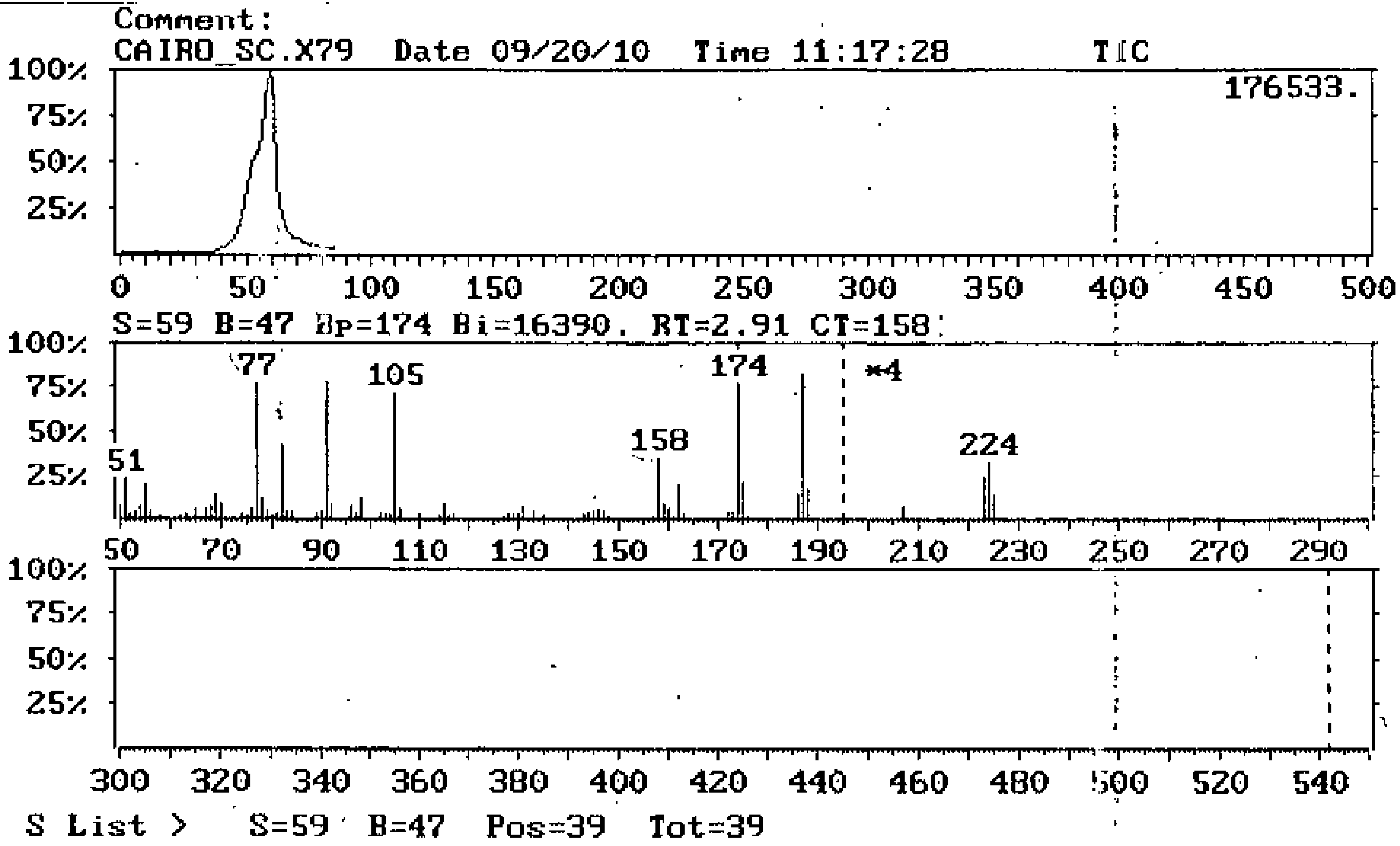


Fig.(14) Mass spectrum of (H_2L^4) ligand

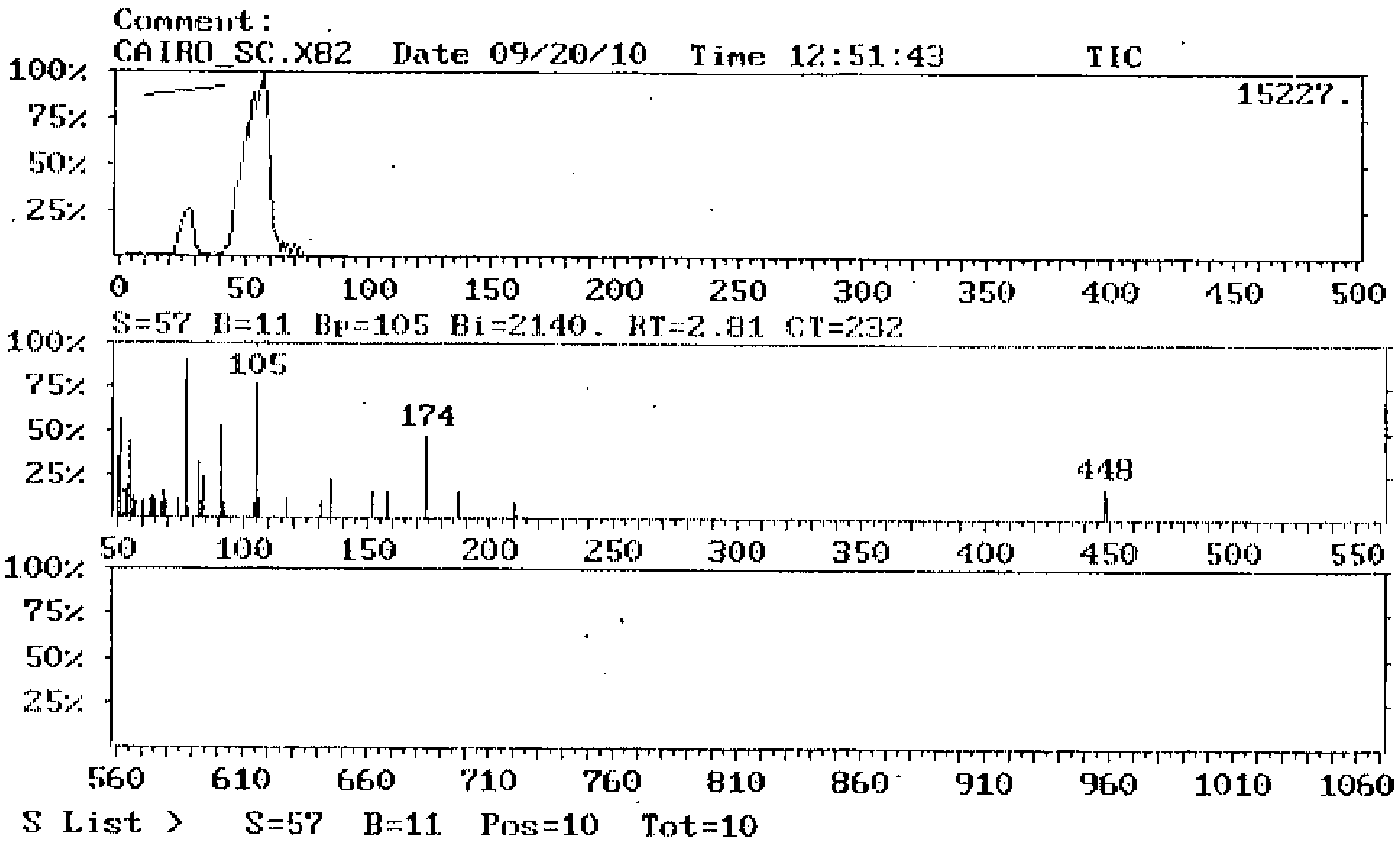


Fig.(15) Mass spectrum of (H_2L^5) ligand

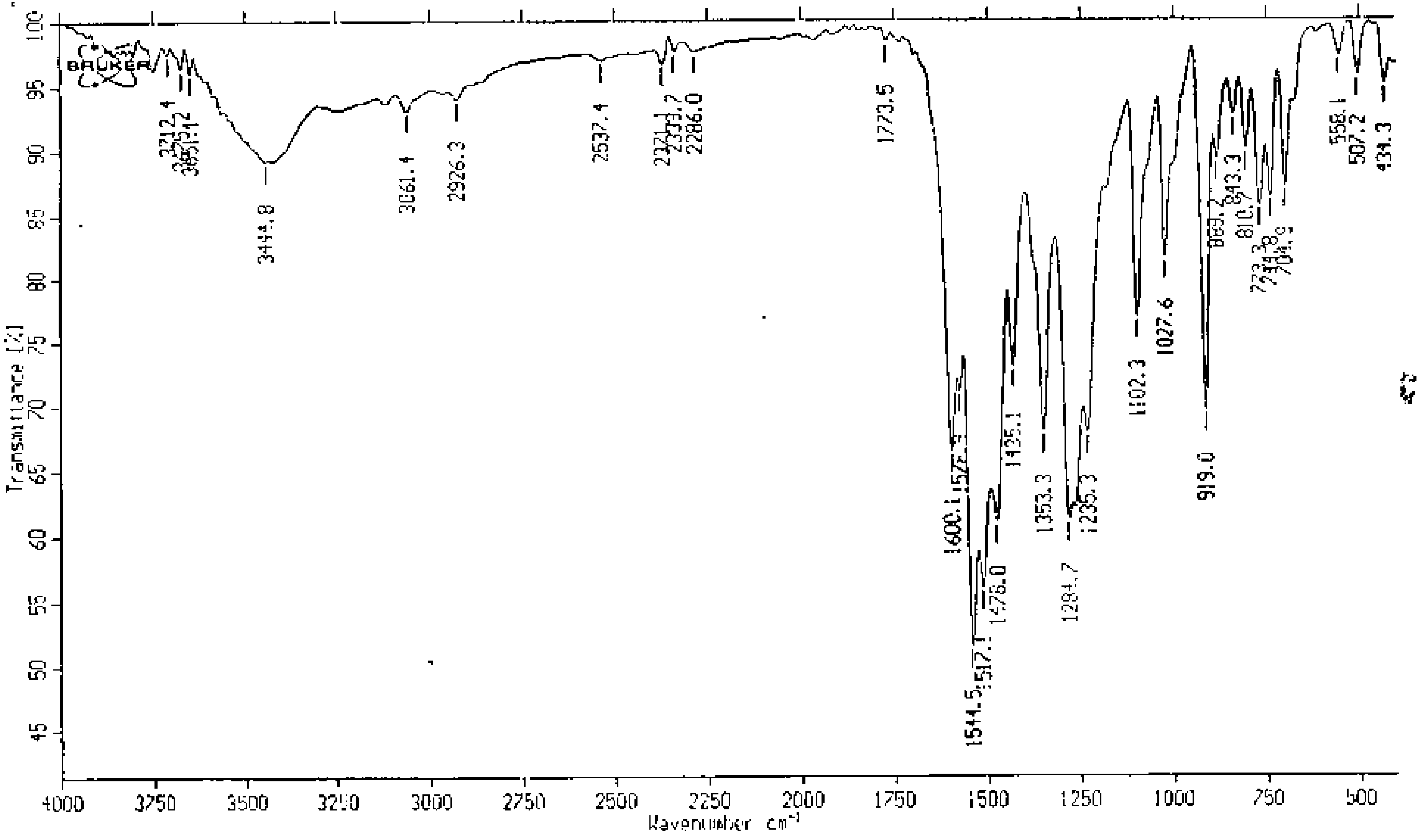


Fig.(16) Infrared spectra of $C_{22}H_{22}N_2O_4U.8H_2O$ Complex

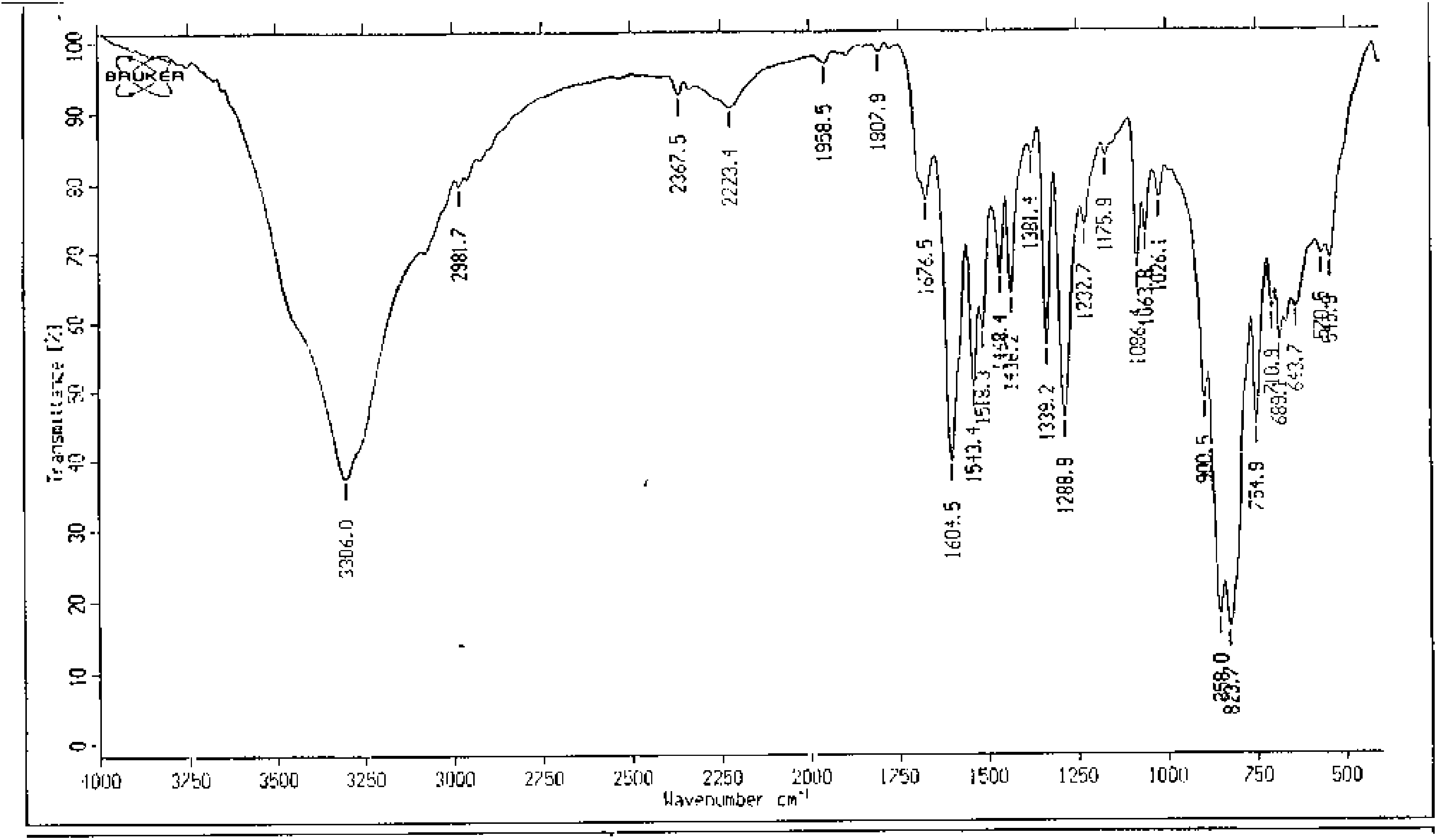


Fig.(17) Infrared spectra of $C_{22}H_{22}N_2O_4Mo.4H_2O$ Complex

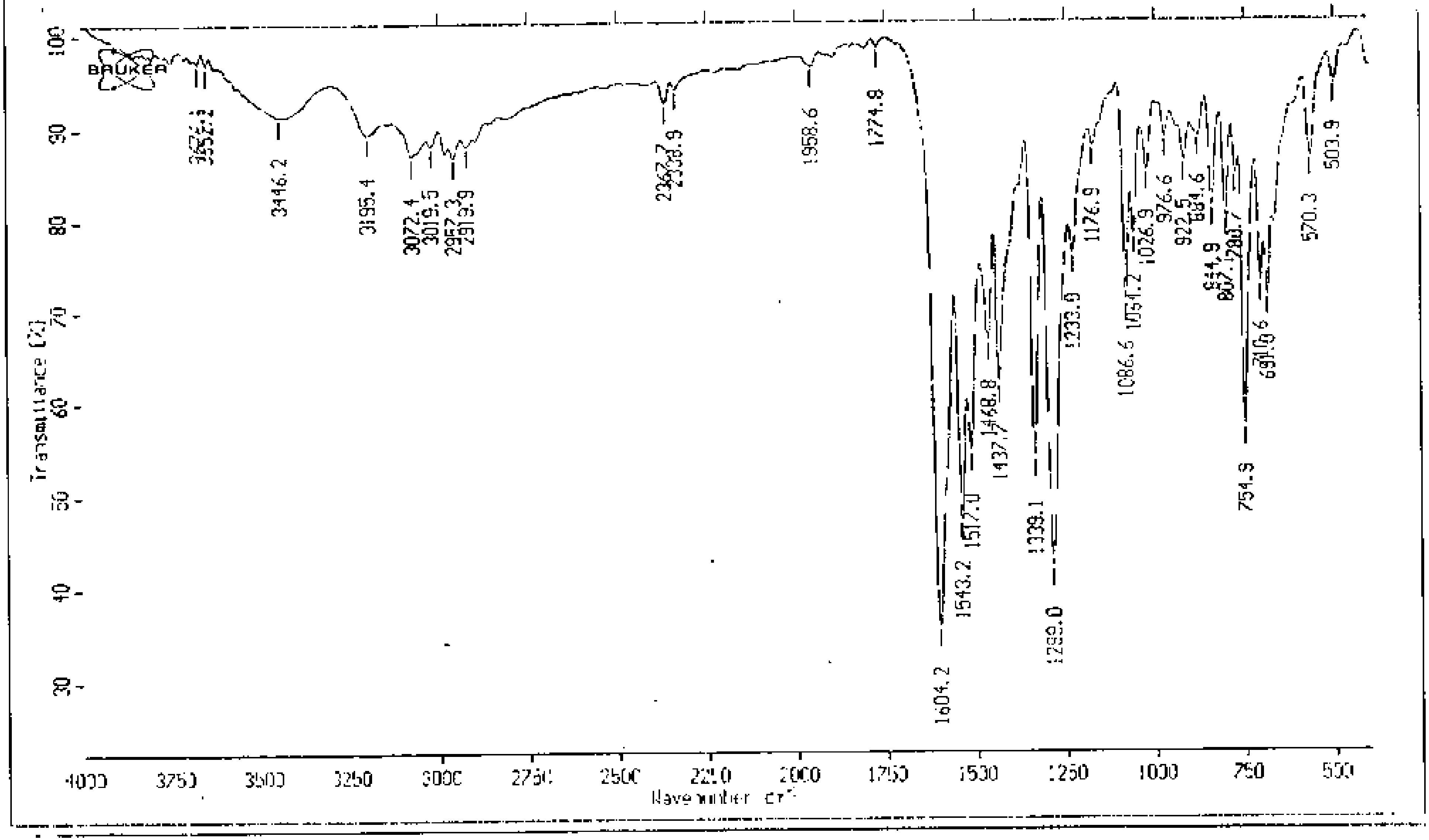


Fig.(18) Infrared spectra of $C_{22}H_{22}N_2O_3V.\ H_2O$ Complex

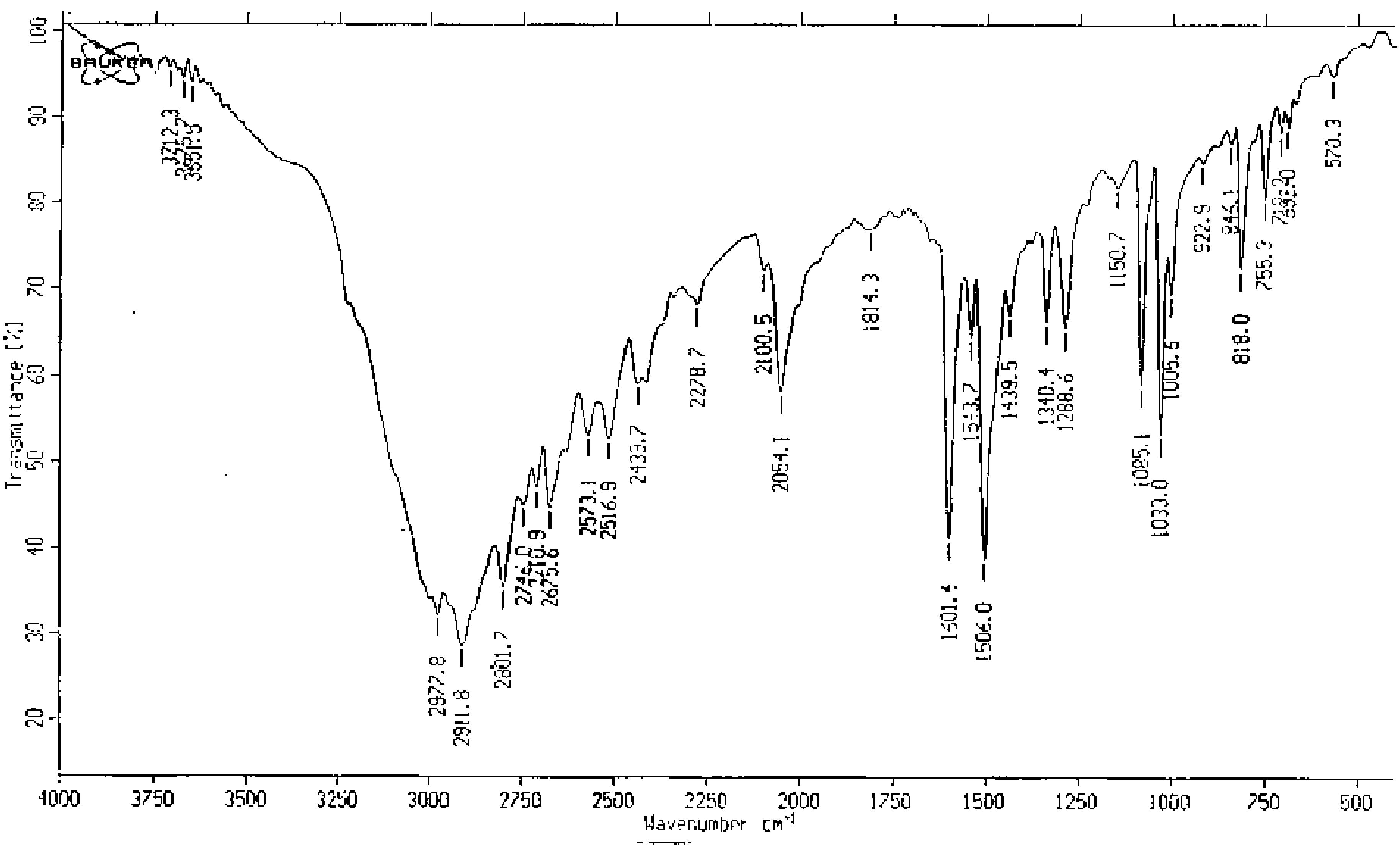


Fig.(19) Infrared spectra of $C_{22}H_{22}N_2O_3Zr.2\ H_2O$ Complex

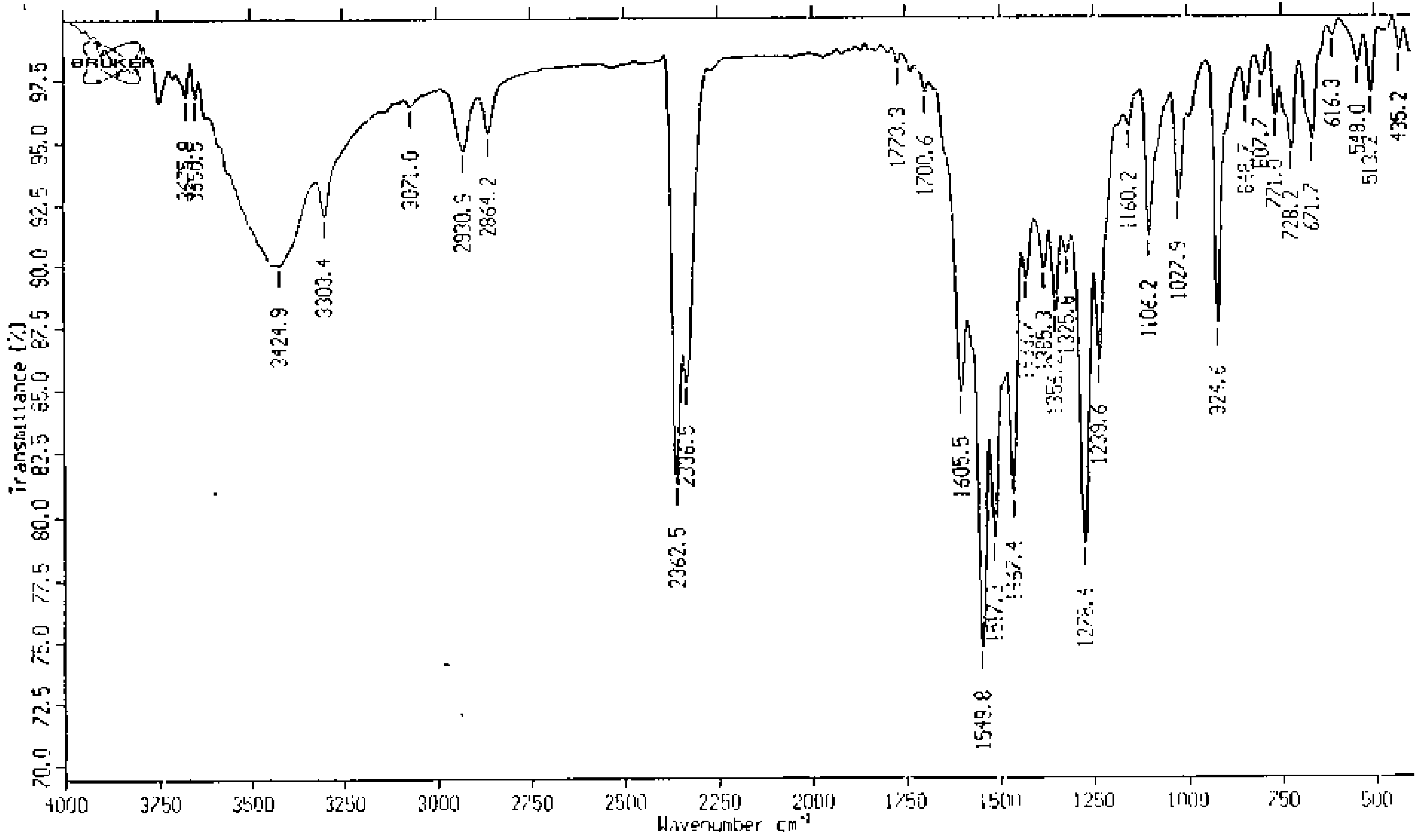


Fig.(20) Infrared spectra of $C_{26}H_{30}N_2O_4U.6\ H_2O$ Complex

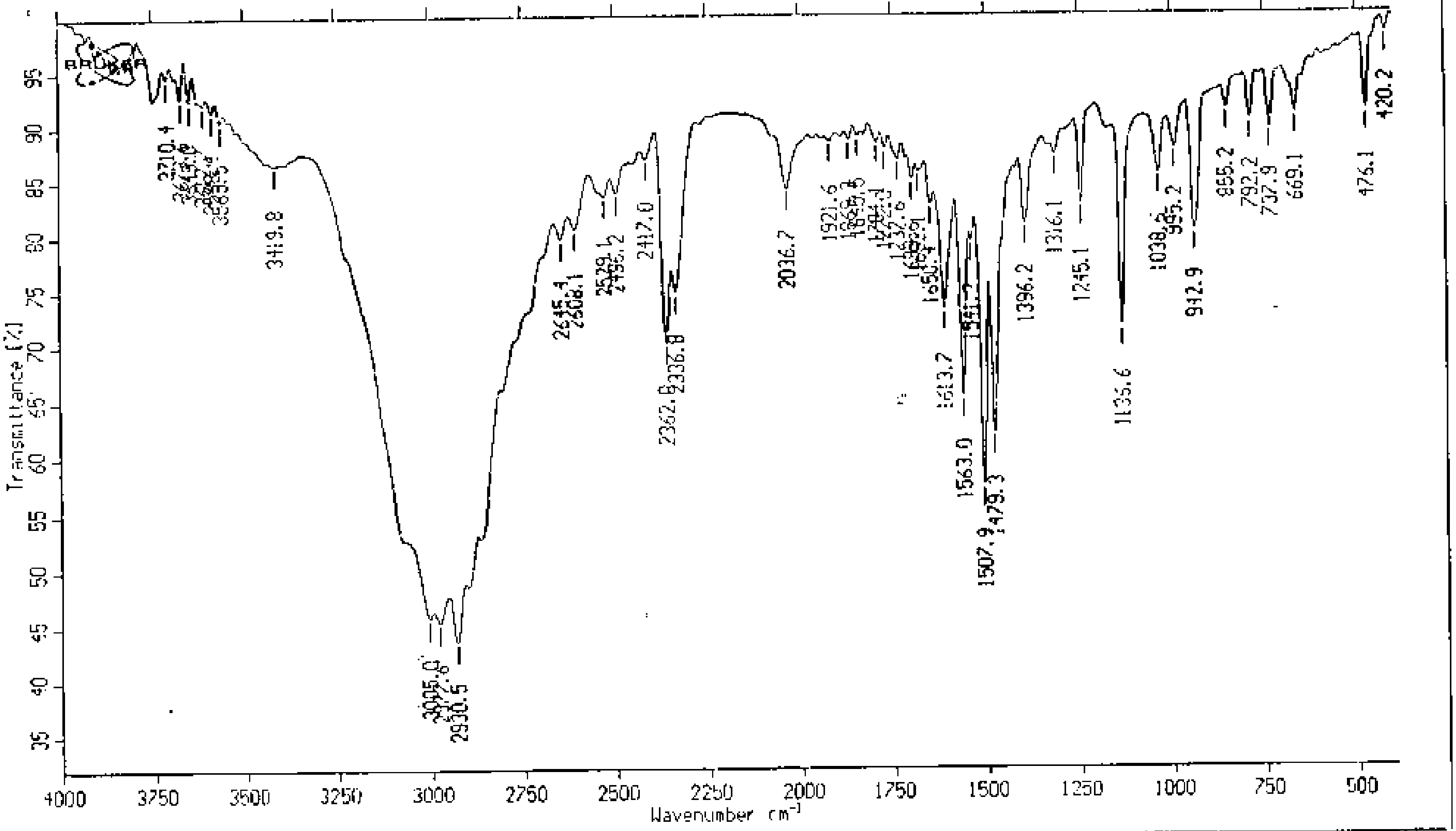


Fig.(21) Infrared spectra of $C_{26}H_{30}N_2O_4Zr.6\ H_2O$ Complex

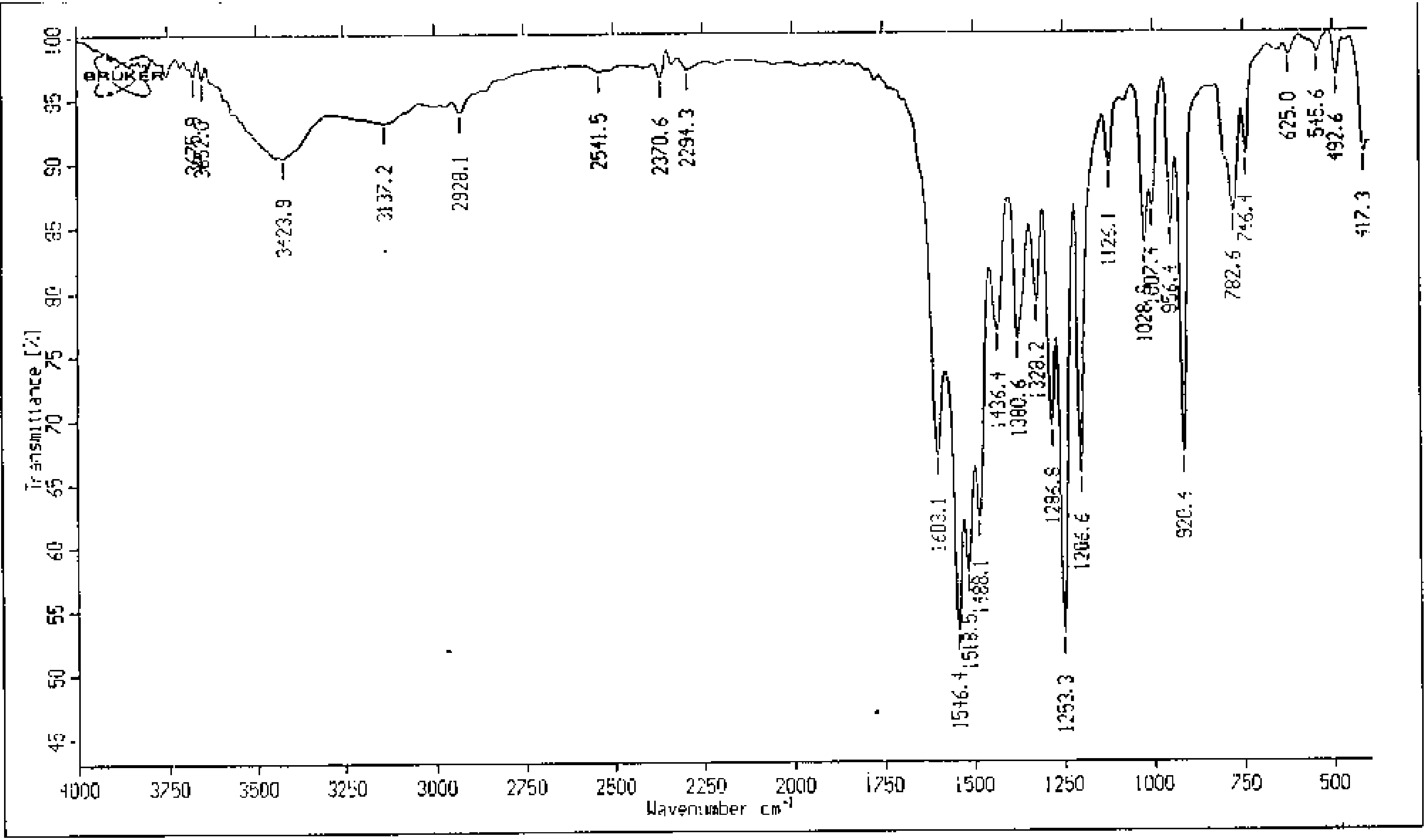


Fig.(22) Infrared spectra of $C_{16}H_{26}N_2O_4U.2\ H_2O$ Complex

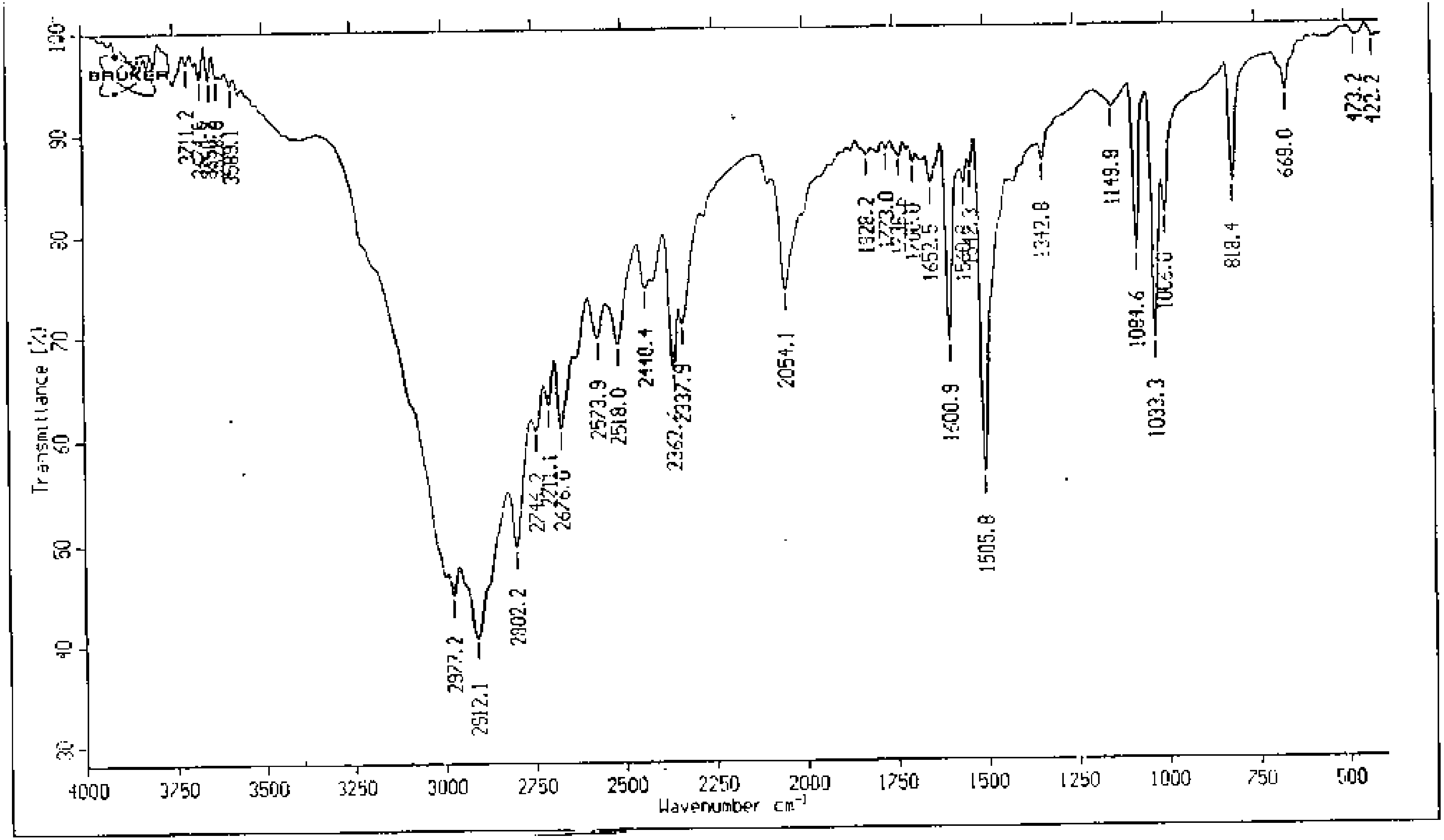


Fig.(23) Infrared spectra of $C_{16}H_{26}N_2O_3Zr.2\ H_2O$ Complex

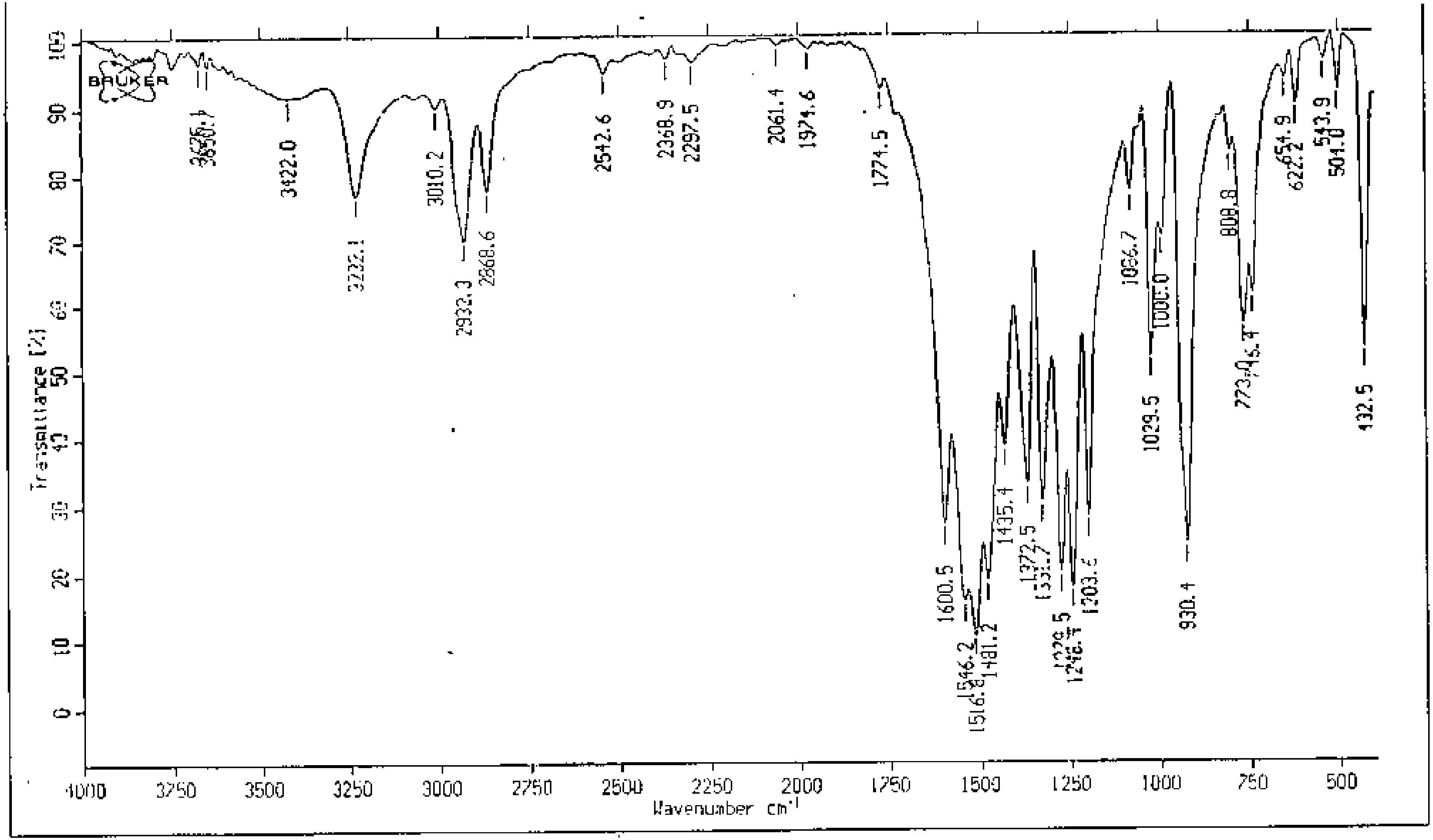


Fig.(24) Infrared spectra of $C_{12}H_{18}N_2O_4U. H_2O$ Complex

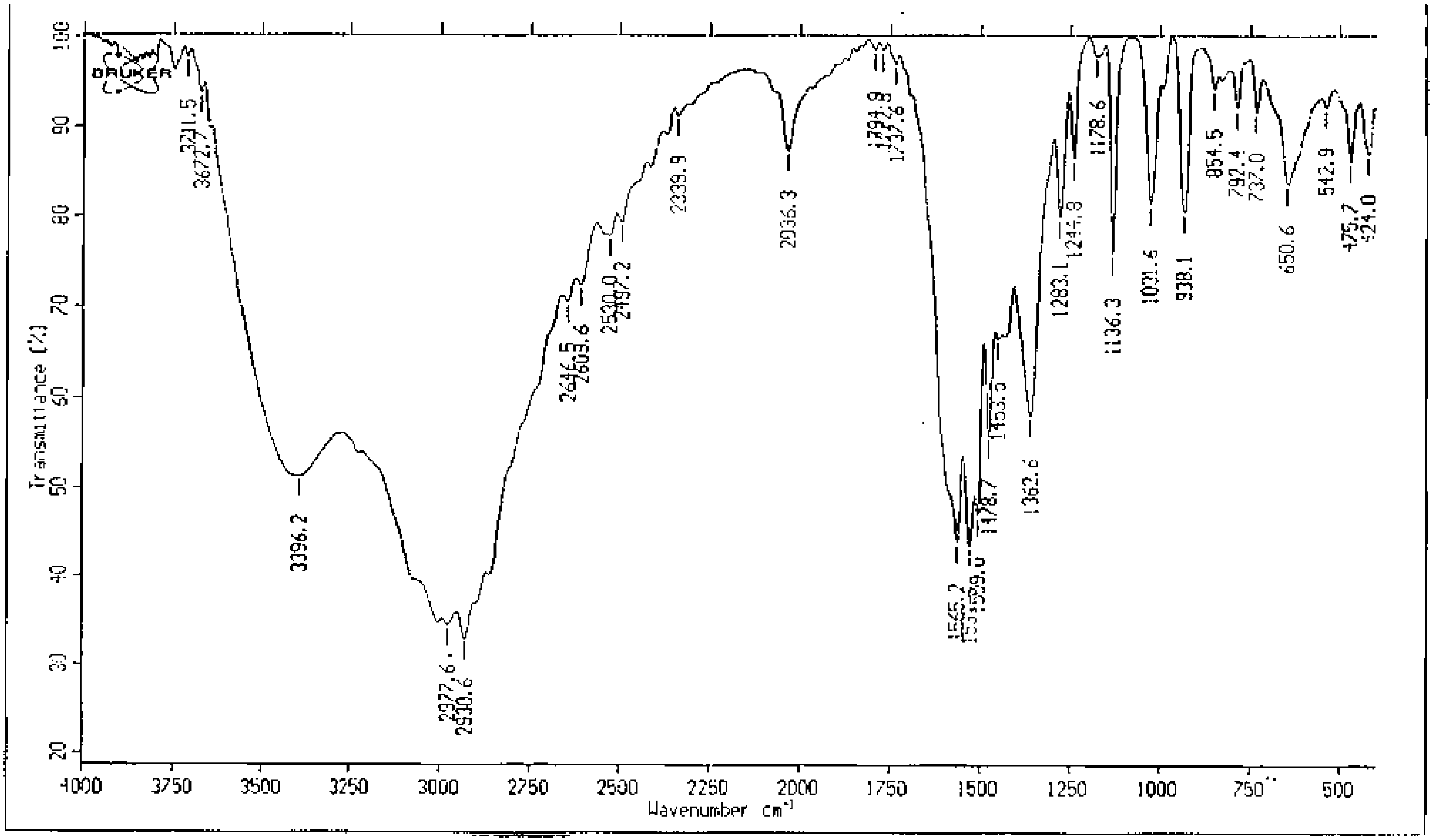


Fig.(25) Infrared spectra of $C_{12}H_{18}N_2O_3V.\ 6H_2O$ Complex

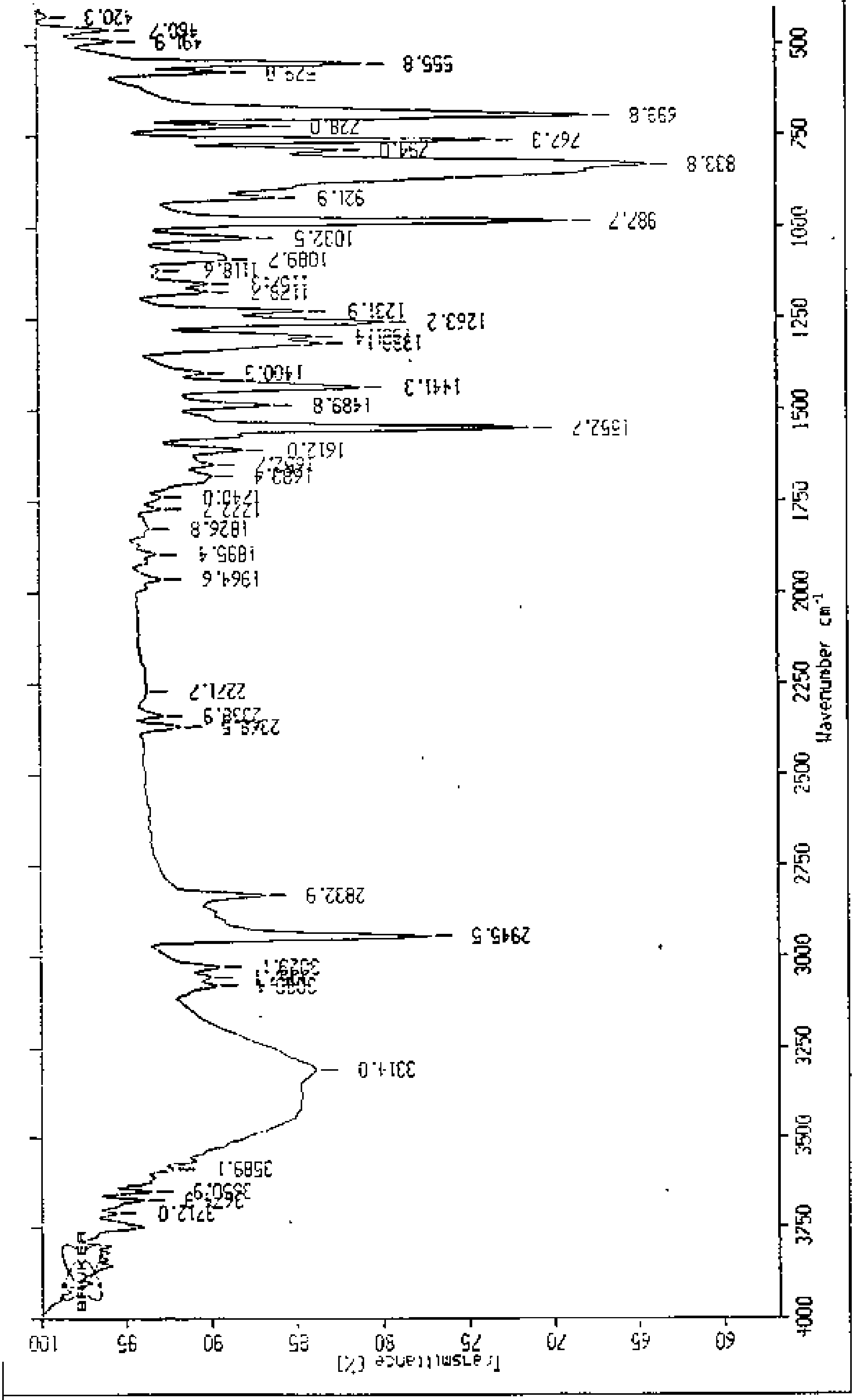
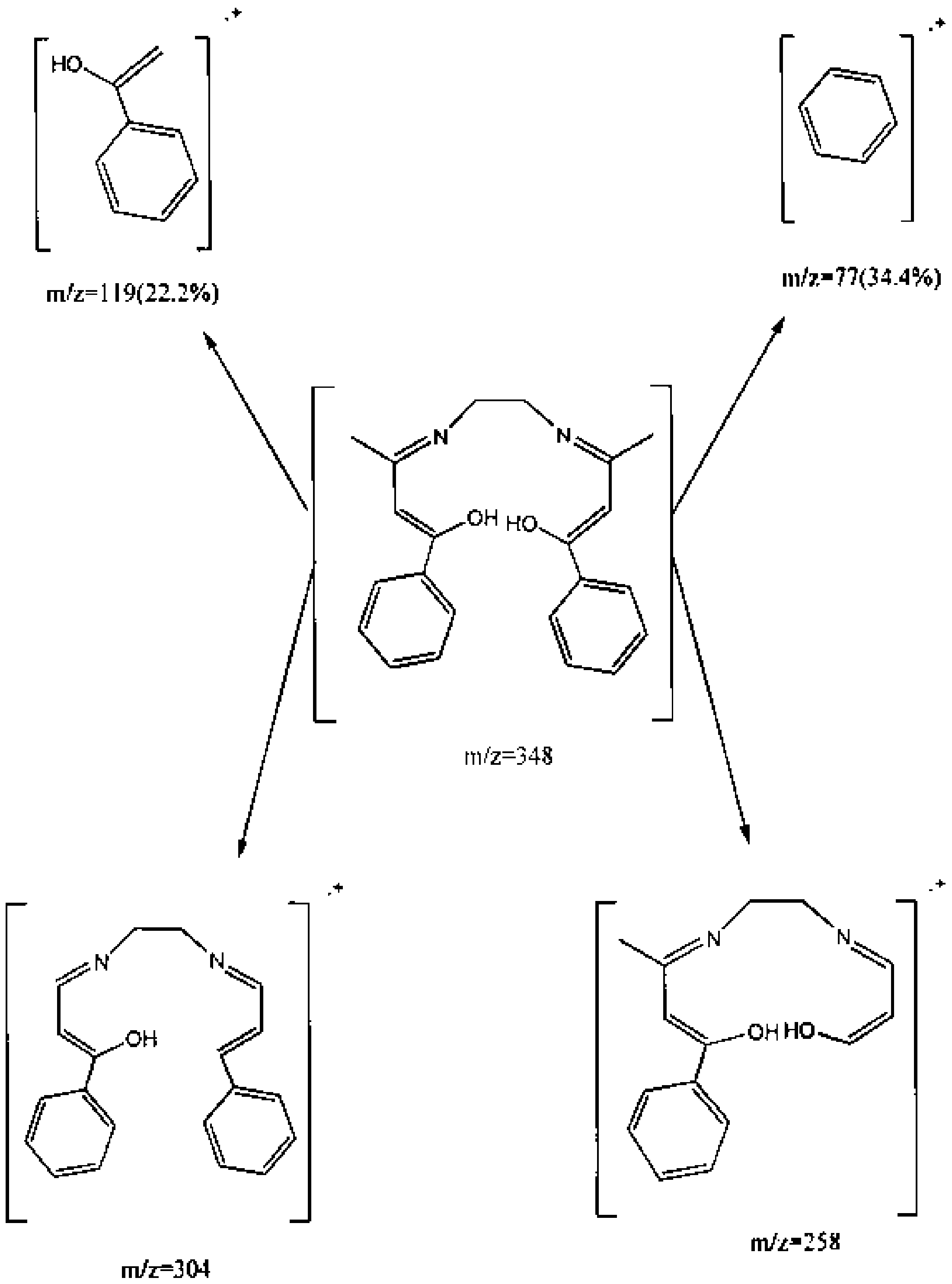


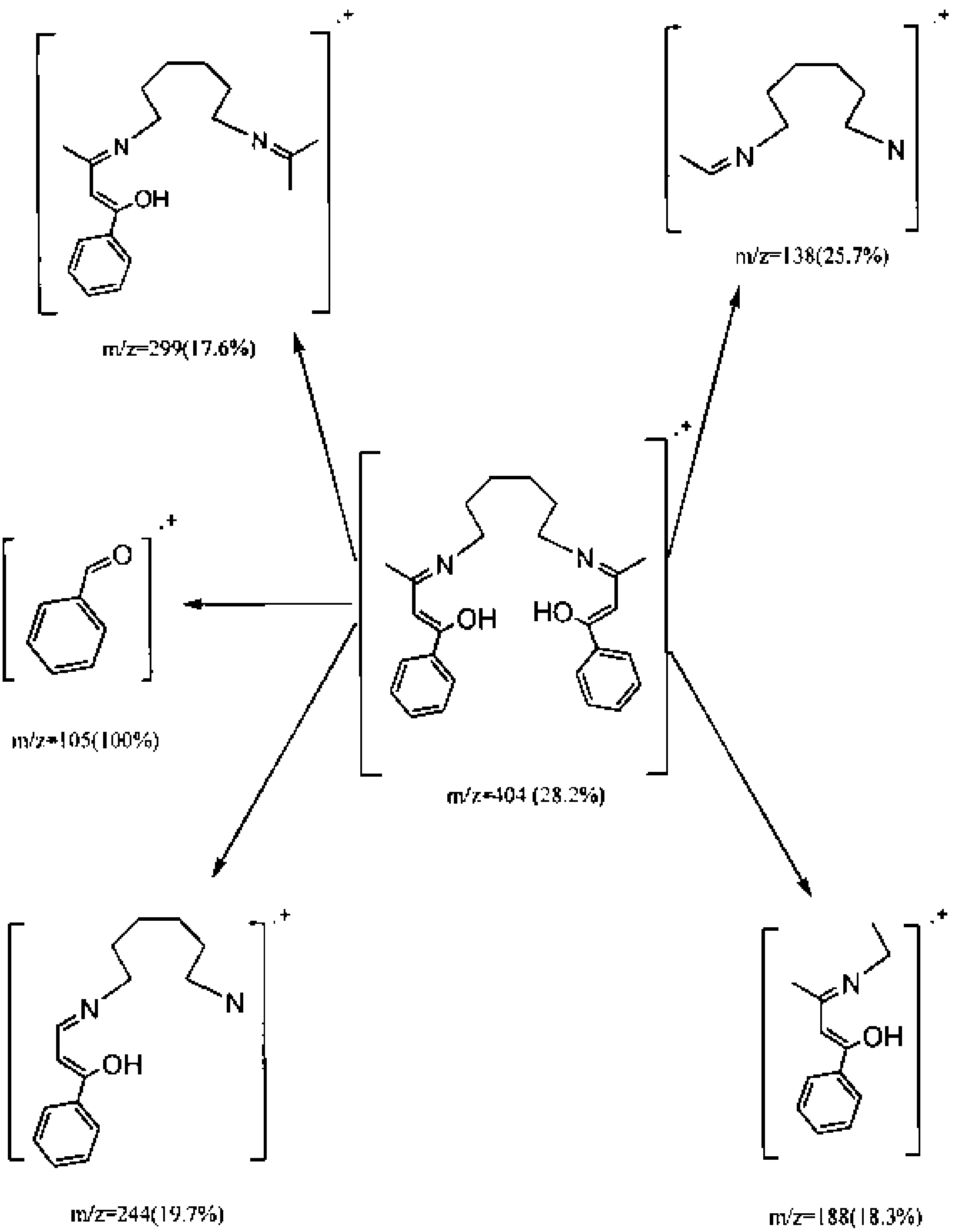
Fig.(26) Infrared spectra of $C_{30}H_{26}N_2O_4Mo.\ H_2O$ Complex

APPENDIX

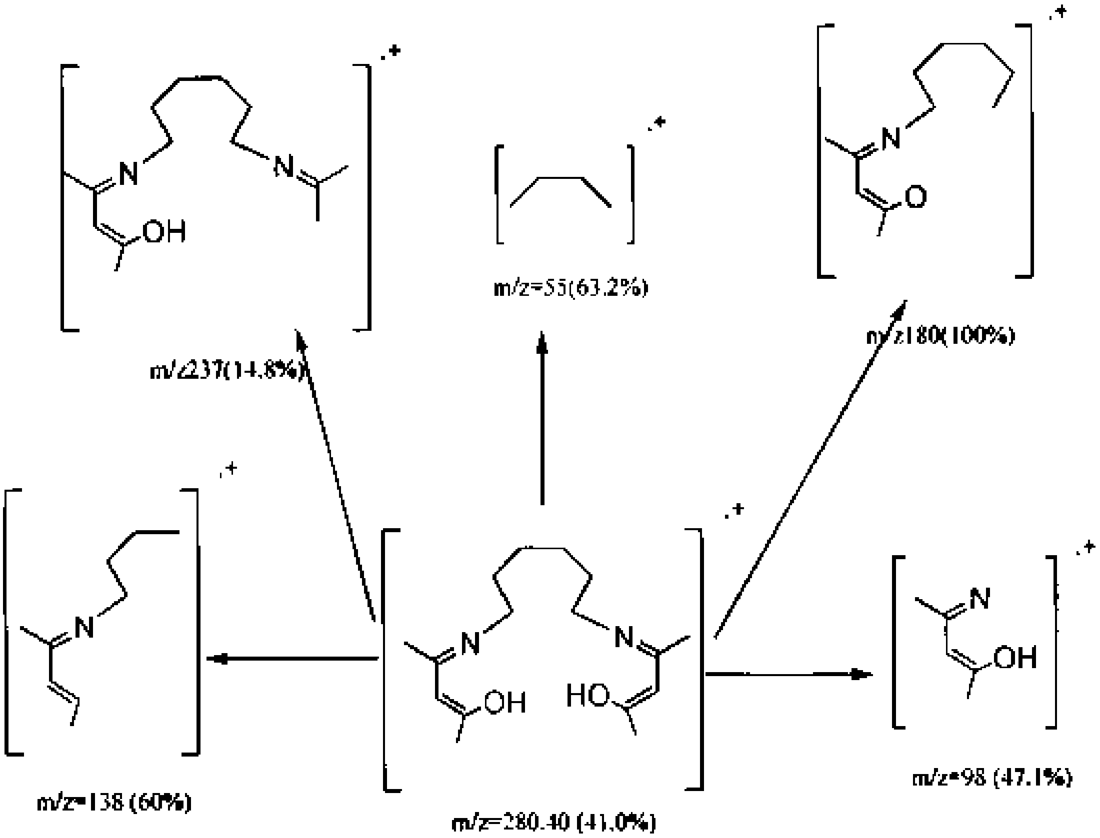
Mass spectrum of Schiff base Ligands



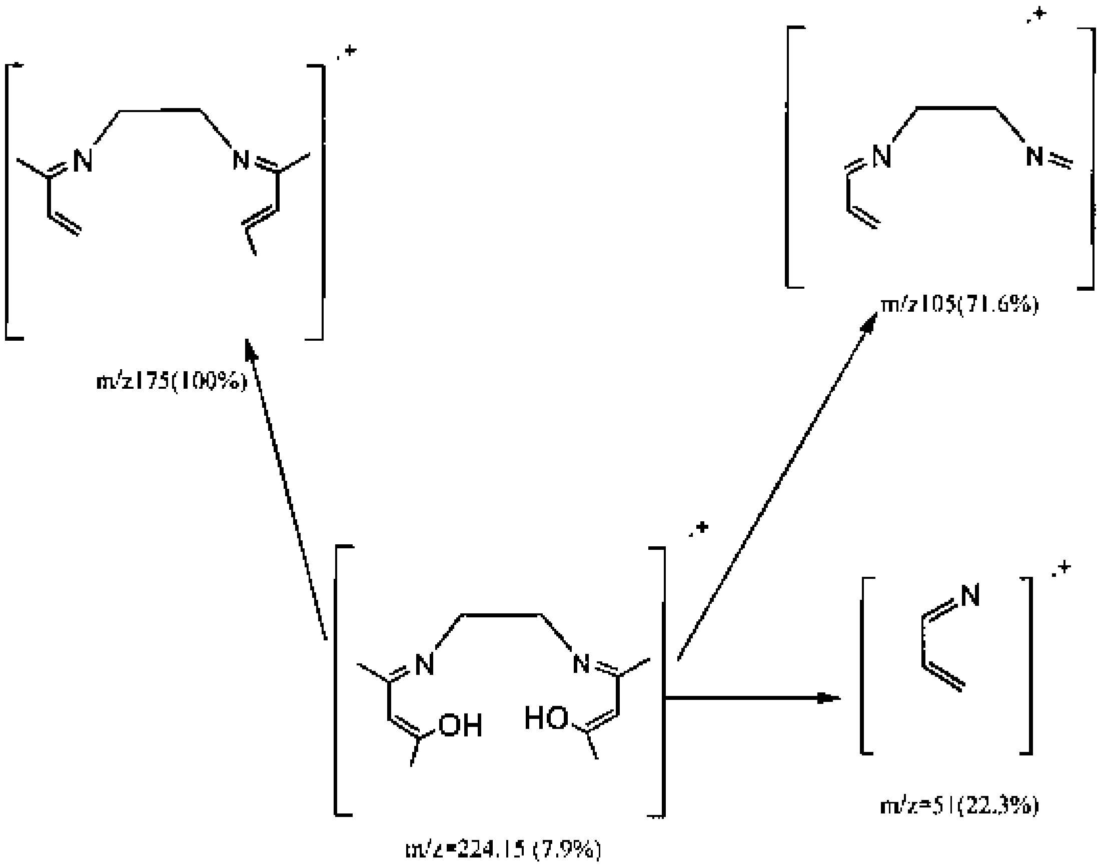
Scheme 1


m/z=299(17.6%)
m/z=138(25.7%)
m/z=105(100%)
m/z=404 (28.2%)
m/z=244(19.7%)
m/z=188(18.3%)
N
OH
HO
O
.+

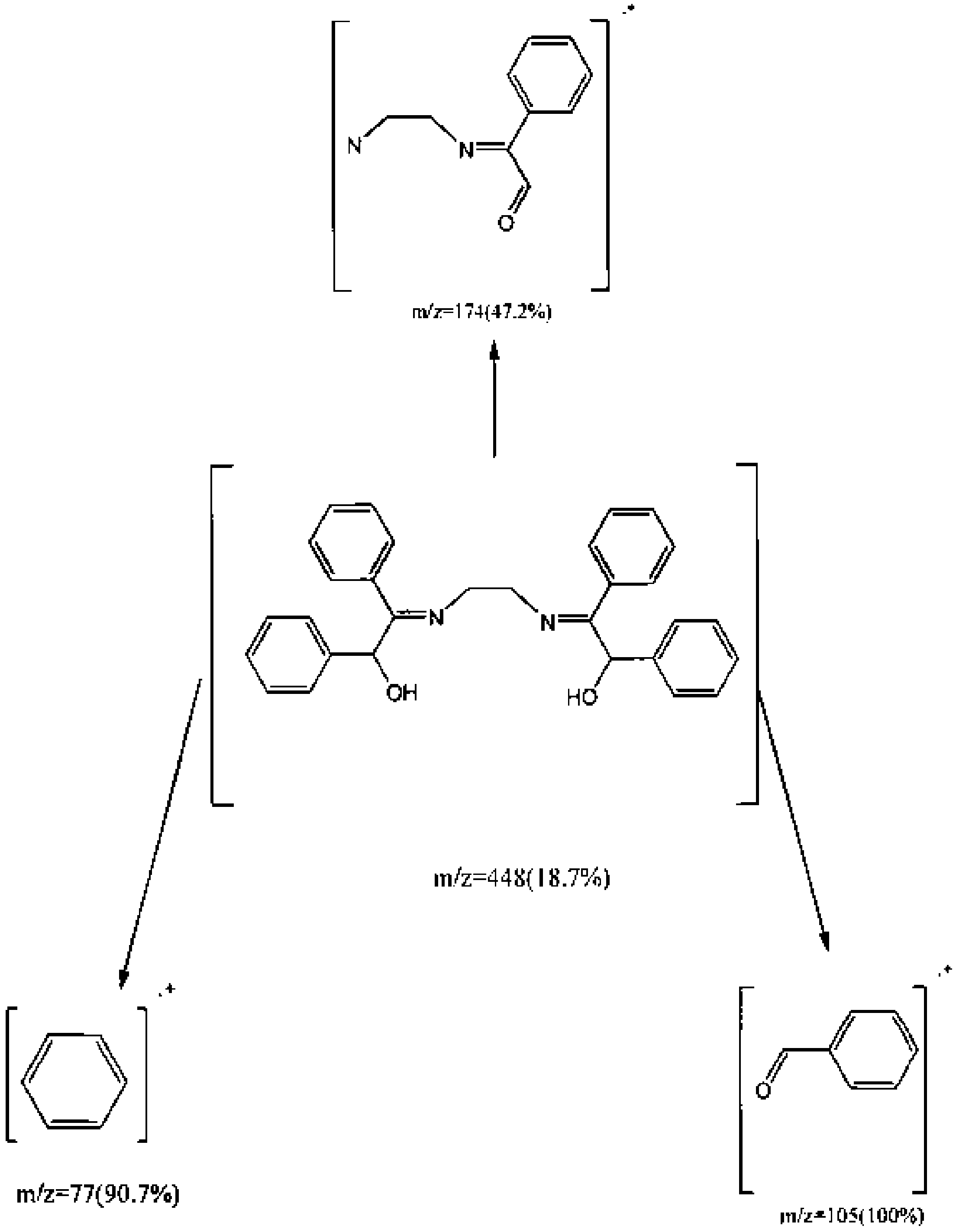
Scheme 2


m/z237(14.8%)
m/z=55(63.2%)
m/z180(100%)
m/z=138 (60%)
m/z=280.40 (41.0%)
m/z=98 (47.1%)
N
OH
HO
O

Scheme 3

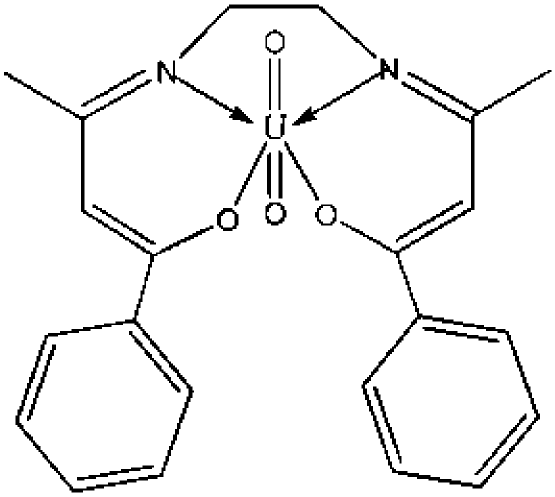


Scheme 4

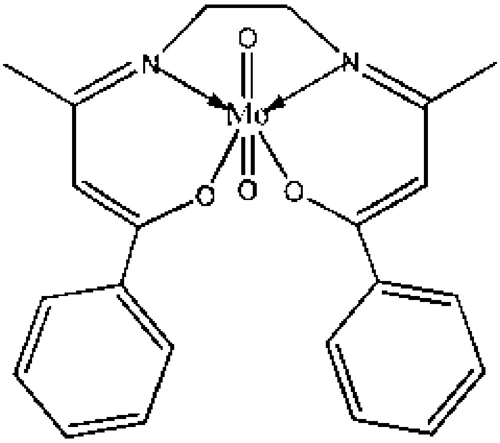


Scheme 5

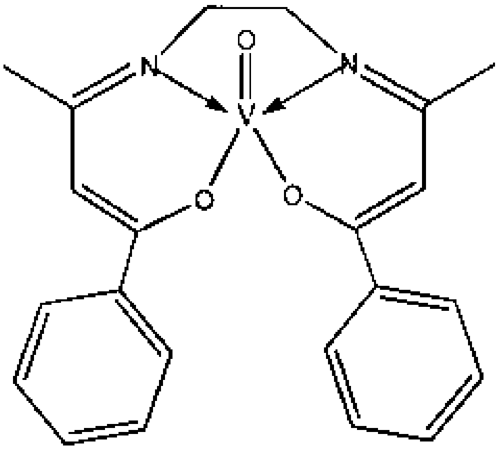
Suggested geometrical structures for the metal complexes under investigation



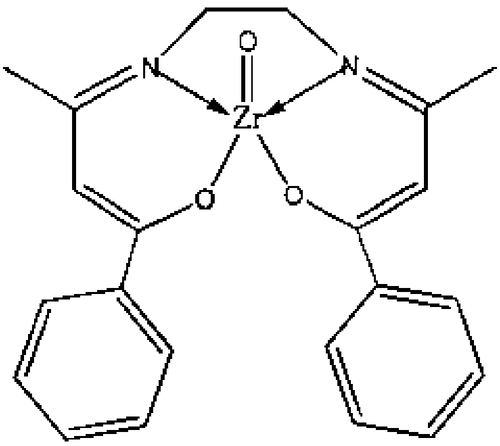
$C_{22}H_{22}N_2O_4U.8H_2O$



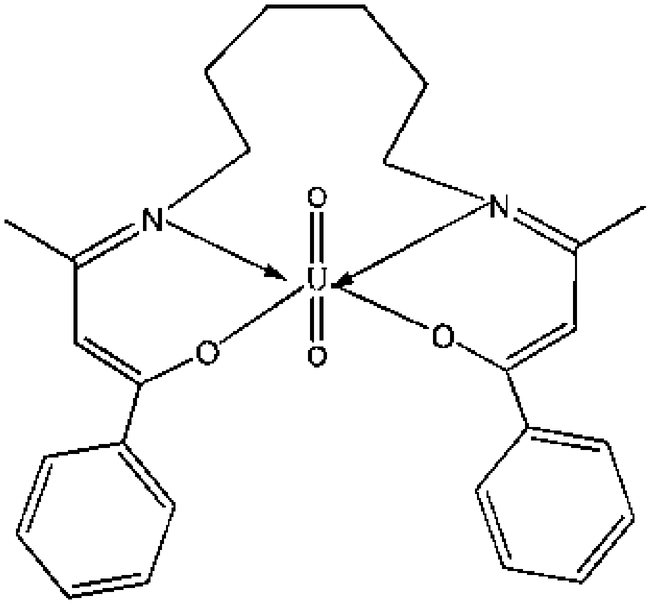
$C_{22}H_{22}N_2O_4Mo.4H_2O$



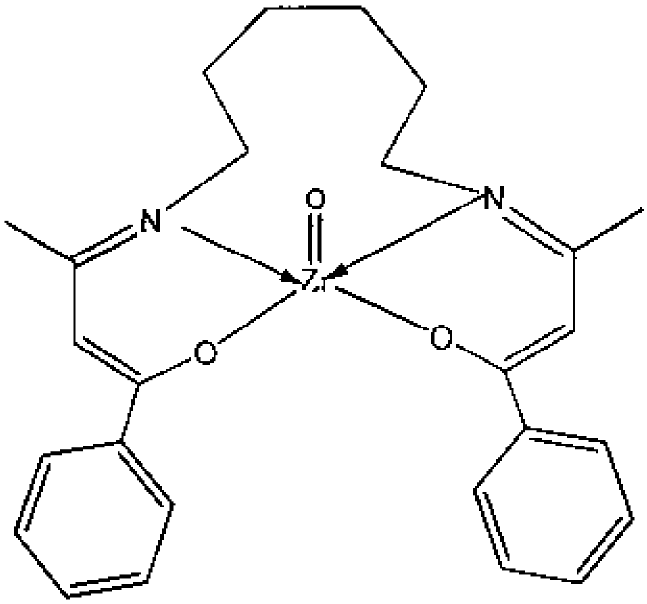
$C_{22}H_{22}N_2O_3V.\ H_2O$



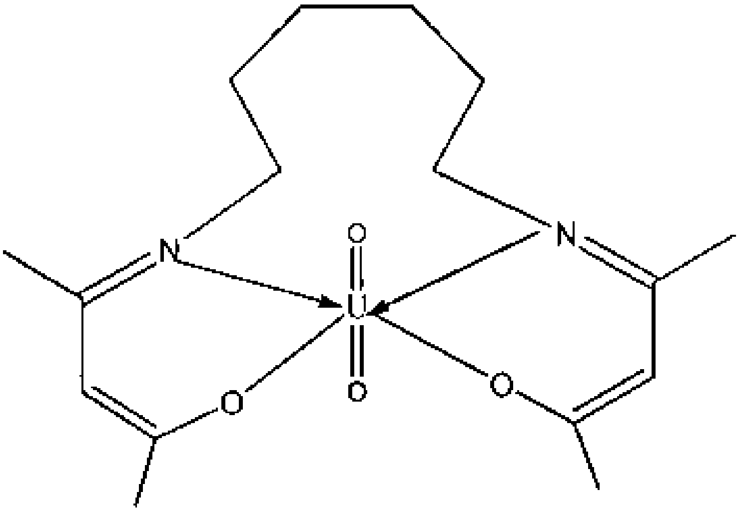
$C_{22}H_{22}N_2O_3Zr.2H_2O$



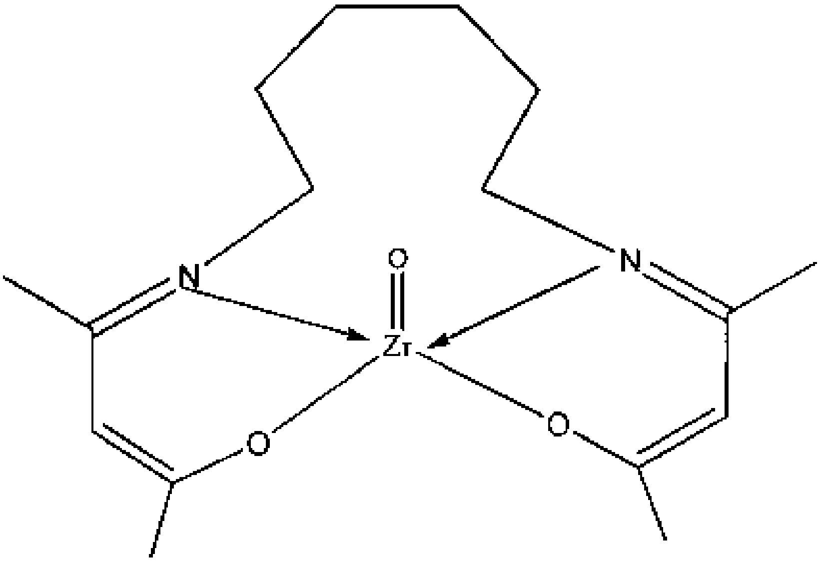
$C_{26}H_{30}N_2O_4U.6H_2O$



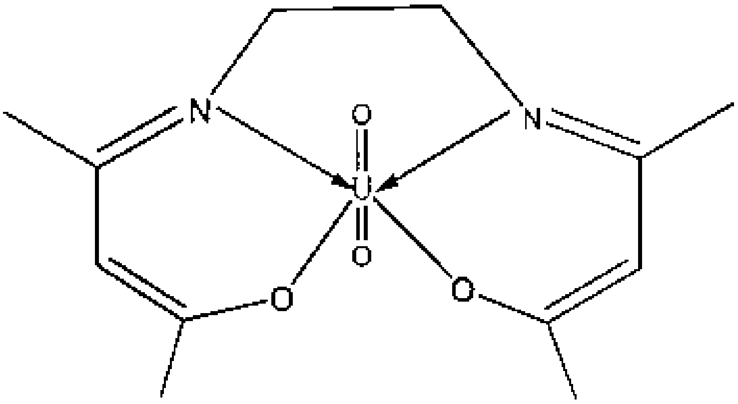
$C_{26}H_{30}N_2O_3Zr.6H_2O$



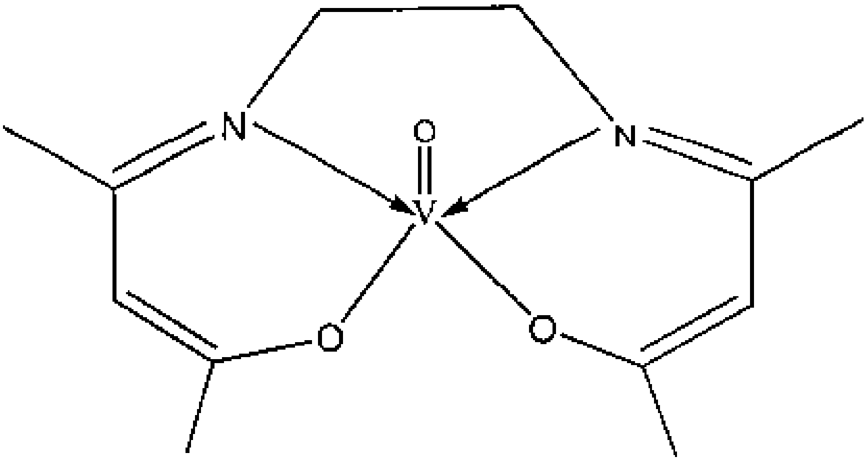
$C_{16}H_{26}N_2O_4U.2H_2O$



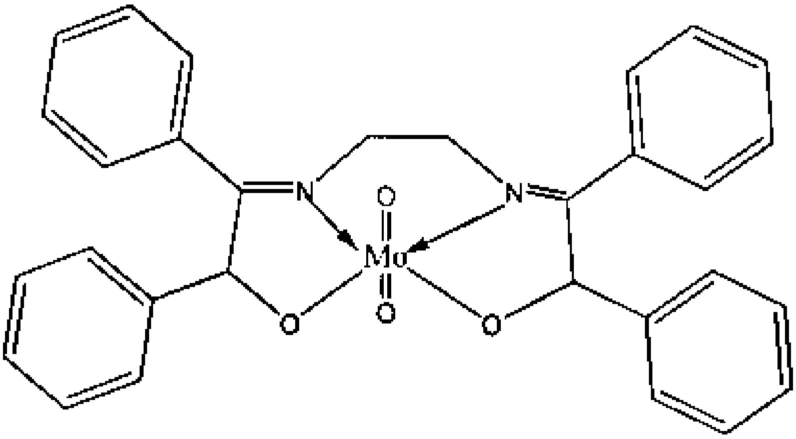
$C_{16}H_{26}N_2O_3Zr.2H_2O$



$C_{12}H_{18}N_2O_4U. H_2O$



$C_{12}H_{18}N_2O_3V. 6H_2O$



$C_{30}H_{26}N_2O_4Mo.H_2O$

Conclusion

The Ethylenediamine and 1,6hexandiamine reacts with Benzoylacetone, acetylacetone, and Benzoin, the reaction produced are the Schiff base ligands H_2L^1= $[C_{22}H_{24}N_2O_2]$, H_2L^2= $[C26H32N_2O_2]$, H_2L^3= $[C_{16}H_{28}N_2O_2]$,H_2L^4= $[C_{12}H_{20}N_2O_2]$ and H_2L^5= $[C_{30}H_{28}N_2O_2]$ have been prepared by direct condensation. The prepared Schiff base ligands have been reacted with some oxy metal ions UO_2^{2+}, VO^{2+}, MoO_2^{2+} and $ZrO^{2+}$and produced the corresponding complexes. The analysis of the Schiff base ligands and their complexes showed that the formation of mononuclear metal complexes.

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الخلاصة

إن الاهتمام الكبير لمعقدات العناصر الانتقالية التي تتضمن تركيب قواعد شيف المحتوية على مجموعة (الازوميثين) (azomethine) بدأ في الظهور بعد اكتشافها من قبل العالم هيوجو شف الصيدلي الألماني حيث أنه درسها بتوسع هي ومعقداتها وذلك بسبب أهمية هذه المركبات الحيوية والصناعية حيث أن ارتباط قواعد شيف مع بعض العناصر الانتقالية يزيد من نشاطها وفاعليتها وقد استخدمت هذه المواد في المجال الطبي لعلاج بعض الأمراض مثل السرطان وغيرها واستخدمت كمضادات حيوية لأنواع مختلفة من البكتيريا والميكروبات والكائنات المجهرية الضارة.

وقد تم في هذا البحث تحضير ودراسة خمس مترابطات (Ligands) من نوع (2N,2O) وذلك بتكثيف بعض الأمينات الاليفاتية مع بعض من الكيتونات الاروماتية وقد تحصلنا على عدد إحدى عشر معقداً لهذه المترابطات بعد إضافة العناصر الانتقالية$(UO2^{+2},VO^{+2},MoO2^{+2},ZrO^{+2})$ لها ودراسة من حيث الشكل والتركيب المتوقع ذلك من خلال إجراء بعض التحاليل عليها.

لقد تم وضع النتائج المتحصل عليها في ست جداول وقد نوقشت النتائج من حيث الشكل والتركيب المتوقع وتم مقارنتها مع بعض الدراسات السابقة.

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جامعة التحدي

إن الدراسة ليست غاية في حد ذاتها
وإنما الغاية هي خلق الإنسان النموذجي الجديد

الإشاري: ش.ع.ت.ش.س.ك 2009/1/408/02 م

التاريخ: ....
الموافق: 2009/3/4 مسيحي

كلية العلوم

قسم الكيمياء

عنوان البحث

**الدراسات التركيبية لبعض مركبات أوكسي المعادن
مع رباعي المطي (2N,2O) لقواعد شيف أوكسي
المعادن مع عضائد الأيمين**

مقدمة من الطالب
عثمان المجدوب عثمان السوسي

* * لجنة المناقشة :

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(مشرفاً)

2 – د.زيدان جاسم خلف 25-3-2009
(ممتحناً داخلياً)

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جامعة التحدي - كلية العلوم

قسم الكيمياء

سرت – ليبيا

الدراسات التركيبية لبعض معقدات أوكسي المعادن مع قواعد شيف رباعي العطاء (2N,2O)

بحث مقدم كجزء من متطلبات استكمال درجة الماجستير في علم الكيمياء

إعداد

عثمان المجذوب عثمان السوسي

تحت إشراف

الدكتور: عبدالحكيم بلقاسم احمد

2009-2008