



## Synthesis, Characterization and Antimicrobial studies of *o*-Amino-N,N-diethylbenzylamine and its Complexes with Fe(II), Zn(II) and Cu(II) Metals

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*o*-Amino-N,N-diethylbenzylamine and its complexes with Fe(II), Zn(II) and Cu(II) metals were synthesized. Both the ligand and its complexes were characterized on the basis of spectroscopic data. X-Ray diffraction study was done to determine the crystalline nature of complexes. Thermal decomposition was carried out to study the thermal stability of complexes. Antimicrobial activity of ligand and its metal complexes were also recorded against some pathogenic bacteria

**Key Words:** Thermal analysis, Antimicrobial activity, X-Ray diffraction, *o*-Amino-N,N-diethylbenzylamine.

### INTRODUCTION

Almost 24 medicines which are available in market contain N,N-dimethylethylamine group as a side chain which cover most of treatments of different diseases. Some important medicines are antibiotics, antihelminthics, CNS drugs, anti-fungal and medicines used in neoplastic chemotherapy<sup>1</sup>.

The complexes which have been prepared during last decades providing a stimulating area of research and have interesting uses such as in synthesis of epoxy resins<sup>2</sup>, catalysis<sup>3,4</sup> and used to study the immune and citotoxic response induced by two organopalladated complexes: [ $\text{Pd}(\text{N},\text{C-dmba})\text{]}_2(\mu\text{-NCS})_2$  (**1**), [ $\text{Pd}(\text{Cdmba})(\text{NCS})(\text{dppp})$ ] (**2**) were investigated in mice bearing Ehrlich ascites tumour<sup>5</sup>. N,N-dimethylnaphthylamine<sup>6</sup>, 2-bromo N,N-dimethylbenzylamine-  $\text{Mo}(\text{CO})_5$  and 2-bromo N,N-dimethylbenzylamine-  $\text{W}(\text{CO})_5$ <sup>7</sup>, [ $\text{Pd}(\text{dmba})(\text{X})(\text{tu})$ ] complexes<sup>8</sup>, *o*-hydroxy N,N'-dimethylbenzylamine complexes of uranium(VI) and thorium(IV)<sup>9</sup>, and *o*-amino-N-ethyl-N-phenylbenzylamine and its complexes with zinc(II), cadmium(II) and mercury(II)<sup>10</sup> were characterized on the basis of physical properties, elemental analysis data, infrared and nuclear magnetic resonance spectroscopy.

Different N,N-dialkylbenzylamine complexes have been synthesized and characterized. Mononuclear compounds [ $\text{Pd}(\text{C}_2\text{N-dmba})(\text{Cl})(\text{tu})$ ] (**1**) and [ $\text{Pd}(\text{C}_2\text{N-dmba})(\text{Br})(\text{tu})$ ] (**2**), were synthesized and characterized by elemental analysis and infrared (IR), <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} NMR spectroscopies and were also tested against *Mycobacterium tuberculosis*<sup>11</sup>. Antibacterial activity of *o*-cyano-N,N-dipropylbenzylamine and

its complexes with Fe(II), Ni(II) and Co(II)<sup>12</sup>, N,N-dimethylbenzylamine complexes with Zn(II), Cd(II), Hg(II)<sup>13</sup>, *o*-nitro-N,N-dimethylbenzylamine complexes with Cu(II), Ag(I), Au(III)<sup>13</sup>, N,N-dimethylnaphthylamine and its complexes with IB and IIB metals<sup>14</sup>, N,N-dicyclohexylbenzylamine and its complexes with IB and IIB metals<sup>15</sup>, *o*-carboxy-N,N-dibutylbenzylamine and its complexes with IIB metals<sup>16</sup>, *o*-carboxy-N-cyclohexyl-N-methylbenzylamine and its complexes with Zn(II), Cd(II) and Hg(II)<sup>16</sup>, were recorded.

Single crystal X-ray diffraction analyses of N,N-dimethylbenzylamine complexes with IB<sup>16</sup> and IIB metal<sup>16</sup>, *o*-cyano-N,N-dimethylbenzylamine complexes with Cu(II), Ag(I), Au(III)<sup>17</sup>, *o*-cyano-N,N-dimethylbenzylamine complexes with Zn(II), Cd(II) and Hg(II)<sup>17</sup> established the structures of these complexes.

Differential thermal and thermogravimetric analyses of N,N-dimethylbenzylamine complexes with Zn(II), Cd(II) and Hg(II), Ag(I), Au(III)<sup>18</sup>, N,N-dicyclohexylbenzylamine<sup>18</sup> [ $\text{Pd}(\text{dmba})(\text{X})_2$ ] [X = NCO (**2**), SCN (**3**), CN (**4**)]<sup>19</sup> *o*-carboxy N,N'-dimethylbenzylamine uranium(VI) and thorium(IV) complexes, *o*-C<sub>6</sub>H<sub>4</sub>COOHCH<sub>2</sub>N-(CH<sub>3</sub>)<sub>2</sub>UO<sub>2</sub>SO<sub>4</sub> (**I**), *o*-C<sub>6</sub>H<sub>4</sub>COOHCH<sub>2</sub>N(CH<sub>3</sub>)<sub>2</sub>UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub> (**II**), *o*-C<sub>6</sub>H<sub>4</sub>COOHCH<sub>2</sub>N(CH<sub>3</sub>)<sub>2</sub>UO<sub>2</sub>Cl<sub>2</sub> (**III**) and *o*-C<sub>6</sub>H<sub>4</sub>CO-OHCH<sub>2</sub>N(CH<sub>3</sub>)<sub>2</sub>Th(NO<sub>3</sub>)<sub>4</sub> (**IV**)<sup>20</sup>, *o*-hydroxy N,N'-dimethylbenzylamine complexes of the formulae, [LMO<sub>2</sub>X<sub>2</sub>], [LMO<sub>2</sub>SO<sub>4</sub>] and [LTh(NO<sub>3</sub>)<sub>4</sub>], where M = U, L = *o*-C<sub>6</sub>H<sub>4</sub>OHCH<sub>2</sub>N(CH<sub>3</sub>)<sub>2</sub> and X = Cl<sub>2</sub>, NO<sub>3</sub><sup>-</sup><sup>21</sup> had been carried out to explore their degradation patterns.

The mass spectra of benzylamine, N-ethylbenzylamine, N,N-dimethylbenzylamine, *p*-methoxybenzylamine and

4-hydroxy-3-methoxybenzylamine and their acetamide and trimethyl-silyl (TMS) derivatives have been recorded<sup>22</sup>.

## EXPERIMENTAL

E. Merck grade N-bromosuccinimide, benzoyl peroxide, sodium bicarbonate, carbon tetrachloride, *o*-toluidene, diethylamine, *n*-hexane, silica gel G (type 60), ferrous sulphate, zinc sulphate, copper sulphate and alcohol were used in experimental work. All chemicals were used without further purification.

*o*-Amino-N,N-diethylbenzylamine was prepared by treating *o*-toluidene with N-bromosuccinimide in CCl<sub>4</sub> using benzoyl peroxide as catalyst under reflux for 8 h followed by the addition of diethylamine. The ligand was separated by passing through the column packed with Silica gel G (type 60).

$\lambda_{\max}$  of the ligand was taken in CCl<sub>4</sub> on Hitachi UV-visible spectrophotometer, model U-2800 in CCl<sub>4</sub>. Refractive index of ligand was determined by using Abbe's refractometer. Stalagmometer was used to determine the surface tension and viscometer was used to determine the viscosity of ligand. Boiling point and miscibility of ligand was observed.

The complexes of the ligand with Fe(II), Zn(II) and Cu(II) metals were synthesized in ethanol:water mixture (2:1). The products were filtered and washed with distilled water, ethanol, ether and *n*-hexane and dried to get solid compounds. The melting points or decomposition points were determined on electrothermal melting point device model No. 1002, USA by keeping the heating rate programmed at 30 °C/min. The miscibility of the ligand was noted by using common organic solvents.

FTIR absorption spectra of the ligand and metal complexes were recorded on FTIR spectrophotometer MIDAC corporation USA model M-2000 series in spectral range 4500-500 cm<sup>-1</sup>. X-Ray diffractograms of complexes were carried out on Bruker D8 discover X-ray diffractometer.

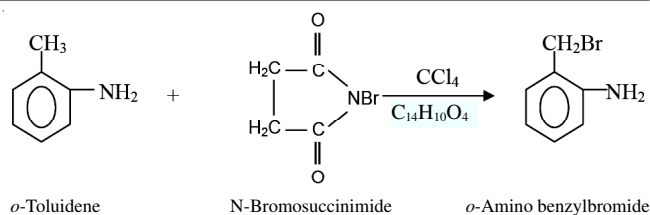
Thermogravimetric and differential thermal analysis (TG/DTA) of all complexes was done in air on a model STD Q 600 in the temperature range of 1-1000 °C and at heating rate 10 °C min<sup>-1</sup> using calcined alumina ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>) as reference material.

Antimicrobial activities of ligand and their complexes were determined by agar diffusion method against *Bacillus subtilis*, *Escherichia coli*, *Micrococcus luteus*, *Staphylococcus aureus* and *Aspergillus flavus*. The antimicrobial activities of the ligand and its complexes were compared with one another and with streptomycin, which was used as a positive control. An emulsion of 5 % gumacacia was used as a negative control, which shows no antimicrobial activity<sup>23</sup>.

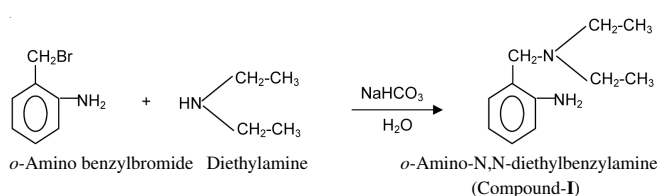
### Synthesis of the ligand *o*-amino-N,N-diethylbenzylamine

**Step-1: Bromination of *o*-toluidene:** Bromination was carried out by method of Farooq *et al.*<sup>10</sup> as outlined below. *o*-Toluidene (0.5 mol), N-bromosuccinimide (0.5 mol) and benzoyl peroxide (1.0 g) in CCl<sub>4</sub> (250 cm<sup>3</sup>) were charged in quickfit flask equipped with water condenser and reaction mixture was heated under reflux for 8 h until all the solid started floating on the liquid surface. The mixture was allowed to cool down to room temperature and the regenerated succinimide was filtered off.

**Step-2: Synthesis:** *o*-Amino benzylbromide (filtrate from step 1) sodium bicarbonate (0.5 mol) and distilled water were



taken in a quickfit flask (500 cm<sup>3</sup>) equipped with water condenser. Diethylamine (0.5 mol) was added dropwise to the reaction mixture through condenser with the help of separating funnel. The mixture was refluxed for 4 h. The mixture was allowed to cool and filtered off. The filtrate was then transferred to a separating funnel where the upper organic layer was collected and passed through a column packed with Silica gel G (type 60). Then *n*-hexane: ethanol (3:2) fraction was collected which on evaporation give brownish purple oily liquid (compound 1), soluble in almost all organic solvents.



### Formation of complexes of *o*-amino-N,N-diethylbenzylamine

**Reaction of *o*-amino-N,N-diethylbenzylamine with ferrous sulphate (FeSO<sub>4</sub>·7H<sub>2</sub>O):** *o*-Amino-N,N-diethylbenzylamine (6.0 mmol) dissolved in ethanol (30 cm<sup>3</sup>) was added to ferrous sulphate (3.0 mmol) dissolved in warm water (15.0 cm<sup>3</sup>). The reaction mixture was stirred for 3 h. A brown solid settled which was filtered and washed with distilled water, ethanol, ether and *n*-hexane and dried to get brown complex (II) having decomposition point 340 °C.

**Reaction of *o*-amino-N,N-diethylbenzylamine with zinc sulphate (ZnSO<sub>4</sub>·7H<sub>2</sub>O):** *o*-Amino-N,N-diethylbenzylamine (6.0 mmol) dissolved in ethanol (30 cm<sup>3</sup>) was added to zinc sulphate (3.0 mmol) dissolved in distilled water (15.0 cm<sup>3</sup>). The reaction mixture was stirred for 3 h. A white solid settled which was filtered and washed with distilled water, ethanol, ether and *n*-hexane and dried to get white complex (III) having decomposition point 330 °C.

**Reaction of *o*-amino-N,N-diethylbenzylamine with copper sulphate (CuSO<sub>4</sub>·5H<sub>2</sub>O):** *o*-Amino-N,N-diethylbenzylamine (6.0 mmol) dissolved in ethanol (30 cm<sup>3</sup>) was added to copper sulphate (3.0 mmol) dissolved in distilled water (15.0 cm<sup>3</sup>). The reaction mixture was stirred for 3 h. A green solid settled which was filtered and washed with distilled water, ethanol, ether and *n*-hexane and dried to get dark green complex (IV) having decomposition point 290 °C.

## RESULTS AND DISCUSSION

*o*-Amino-N,N-diethylbenzylamine was prepared starting from *o*-toluidene and N-bromosuccinimide using CCl<sub>4</sub> as solvent and benzoyl peroxide as catalyst followed by addition of N,N-diethylamine in the presence of NaHCO<sub>3</sub> in distilled water under reflux. The reaction mixture after filtration was purified by column chromatography using silica gel. *n*-Hexane:ether

(3:2) eluted a reddish brown band which on evaporation gave dark brown oily liquid.

The refractive index of the ligand was determined by using Abbe's refractometer, to measure, how much the ligand will slow down the light wave, when it passes through it. The value of refractive index was found to be 1.68. Surface tension of the ligand was determined by using stalagmometer, in order to determine the resistance the ligand exhibits to surface penetration<sup>24</sup>. The value of surface tension was 45.86 dynes/cm at 34 °C. Viscosity of the ligand was obtained with the help of viscometer, in order to determine the resistance that ligand offers to its flow. The value of viscosity was found to be 40.32 g/cm s at 30 °C. Boiling point of ligand was 226 °C. Ligand was miscible in all organic solvents.

The complexes of *o*-amino-N,N-diethylbenzylamine with Fe(II), Zn(II) and Cu(II) were synthesized in ethanol:water mixture (2:1). The products were filtered and washed with distilled water, ethanol, ether and *n*-hexane and dried to get solid compounds. The melting points or decomposition points were noted which were found to be 340, 330 and 290 °C for complex (II), complex (III) and complex (IV), respectively. Brown, white and blue colours were noted, respectively for complex (II), complex (III) and complex (IV).

Infrared spectrum of *o*-amino-N,N-diethylbenzylamine shows the absorption band due to C-H bending at 620 cm<sup>-1</sup>. Aromatic out of plane C-H bending vibration appeared at 751, 813, 867 and 946 cm<sup>-1</sup>. Aromatic in plane C-H bending appeared at 1038, 1116, 1148, 1198 and 1389 cm<sup>-1</sup>. The absorption band due to aromatic stretching and -CH<sub>2</sub> symmetric bending is indicated at 1468 cm<sup>-1</sup>. The absorption bands due to out of plane N-H bending and in plane N-H bending appeared at 684 and 1622 cm<sup>-1</sup>, respectively. C-H stretching bands for methyl and methylene groups are indicated at 2730, 2872, 2930, 2958 and 3027 cm<sup>-1</sup>. The absorption bands which appeared at 3382, 3431 and 3465 cm<sup>-1</sup> are due to N-H stretching vibrations<sup>34</sup>. In case of metal complexes absorption bands appeared below 600 cm<sup>-1</sup> due to M-N bond<sup>35</sup>. The lowering in the position of absorption bands also depicts the formation of complexes. The FTIR data of the ligand and its complexes is given in the Table-1.

The crystal structures of metal complexes were determined by X-ray diffraction spectroscopy. All the complexes were found to be crystalline. The XRD data of complexes is given in Table-2. The XRD patterns for the complexes of Fe(II), Zn(II) and Cu(II) metals are shown in Figs. 1-3, respectively.

The thermogram of Fe(II) complex indicated that it was stable upto 60 °C. Thermal decomposition took place in the temperature range of 70-560 °C. First TG loss was observed in 70-130 °C with the loss of *o*-amino benzaldehyde C<sub>7</sub>H<sub>7</sub>NO (theo. 35 %, exp., 33 %) followed by an exotherm at 100 °C. Second TG loss was in the temperature range of 160-395 °C with the loss of diethylamine C<sub>4</sub>H<sub>11</sub>N (theo. 21 %, exp., 18 %) followed by an exotherm at 290 °C. Third TG loss was in the temperature range of 440-560 °C with the loss of SO<sub>4</sub> in the form of SO<sub>2</sub> + O<sub>2</sub> (theo. 28 %, exp., 27 %) followed by an exotherm at 500 °C. The residue left was FeO which was confirmed through AAS. The TGA/DTA pattern for the complexes of Fe(II) is shown in Fig. 4 and TGA/DTA data of Fe(II) complex is given in Table-3.

TABLE-1 FOURIER TRANSFORMED INFRARED DATA				
No.	Comp.	FTIR absorption bands $\bar{\nu}$ (cm <sup>-1</sup> )		
1	Ligand (I)	3465 (w)	3431 (m)	3382 (m)
		3027 (m)	2958 (m)	2930 (w)
		2872 (s)	2730 (w)	2360 (m)
		1724 (s)	1622 (s)	1468 (s)
		1389 (s)	1198 (w)	1148 (m)
		1116 (m)	1038 (s)	946 (m)
		867 (s)	813 (s)	751 (s)
		684 (m)	620 (w)	
2	Complex (II)	703 (s)	831 (s)	1005 (m)
		1114 (m)	1403 (w)	1631 (w)
		2618 (w)	3396 (w)	4465 (s)
3	Complex (III)	514 (w)	609 (s)	792 (m)
		993 (m)	1122 (s)	1401 (s)
		3510 (m)		
4	Complex (IV)	616 (m)	673 (m)	790 (s)
		1094 (m)	1400 (s)	1633 (w)
		3125 (w)	3546 (s)	

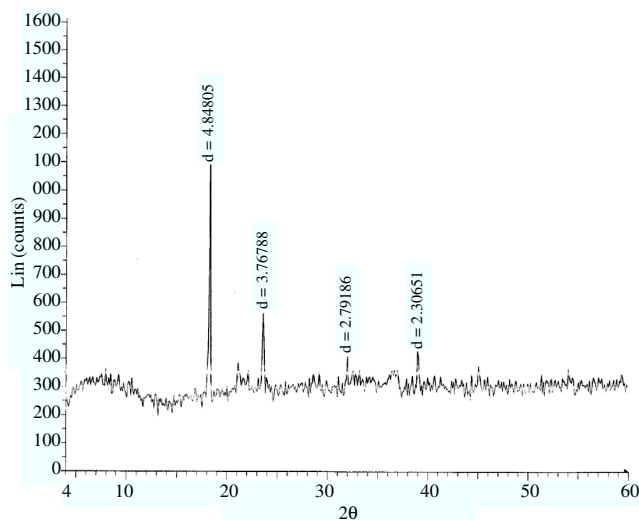


Fig. 1. XRD Pattern of complex (II)

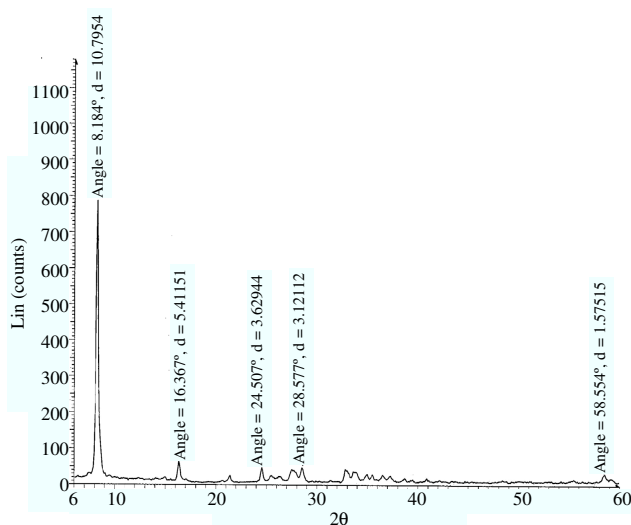


Fig. 2. XRD pattern of complex (III)

**TABLE-2**  
**X-RAY DIFFRACTION ANALYSIS OF COMPLEXES**

No. of observations	Comp. No.	2θ (°)	d-spacing	Intensity line (counts)
1	Complex (II)	17.5	4.848	1090
		23.8	3.767	570
		32.0	2.791	420
		39.0	2.306	440
2	Complex (III)	8.5	10.795	7900
		16.5	5.411	700
		24.5	3.629	400
		28.5	3.121	500
		58.5	1.575	450
3	Complex (IV)	8.5	10.684	1600
		16.5	5.355	1300
		17.2	5.127	460
		20.0	4.418	520
		21.5	4.133	430
		22.8	3.926	450
		24.9	3.591	750
		26.6	3.352	470
		33.4	2.698	650
		34.0	2.641	530
		34.8	2.576	350
		37.2	2.417	580
		38.2	2.356	490
		42.0	2.153	400
50.8	1.797	350		
52.6	1.730	440		

The thermogram of Zn(II) complex indicated that it was stable upto 30 °C. Thermal decomposition took place in the temperature range of 30-920 °C. First TG loss was observed in the temperature range of 30-210 °C with the loss of *o*-amino benzaldehyde C<sub>7</sub>H<sub>7</sub>NO (theo. 34 %, exp., 30 %) followed by an exotherm at 90 °C. Second TG loss was in the temperature range of 265-400 °C with the loss of diethylamine C<sub>4</sub>H<sub>11</sub>N (theo. 21 %, exp., 19 %) followed by an exotherm at 265 °C. Third TG loss for Zn(II) complex occurred in the temperature range of 700-920 °C with the loss of SO<sub>4</sub> in the form of SO<sub>2</sub> + O<sub>2</sub> (theo. 28 %, exp., 26 %) followed by an exotherm at 750 °C. The residue left was ZnO which was confirmed through AAS. The TGA/DTA pattern for the complexes of Zn(II) is shown in Fig. 5 and TGA/DTA data of Zn(II) complex is given in Table-3.

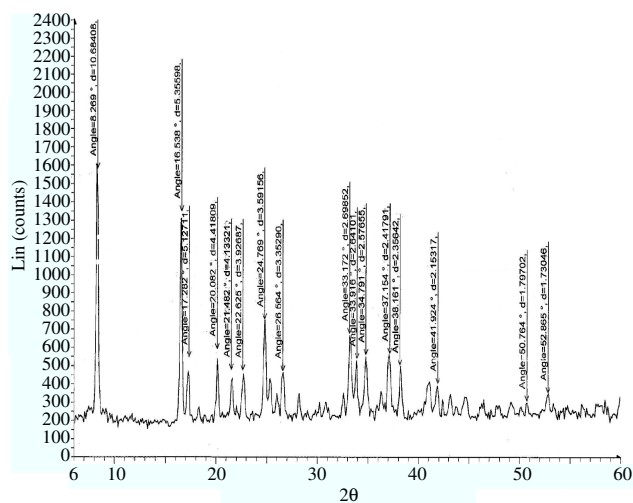


Fig. 3. XRD pattern of complex (IV)

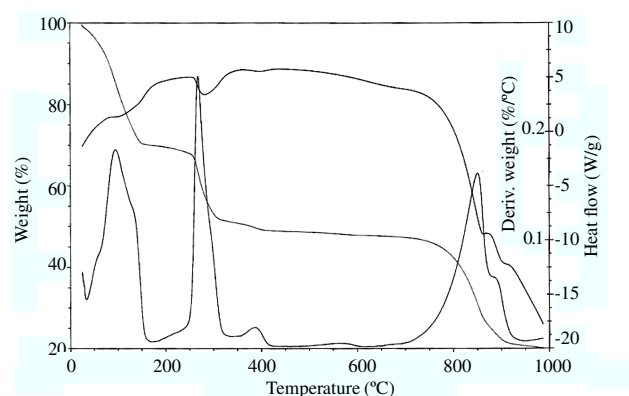


Fig. 5. TGA/DTA pattern of complex (III)

The thermogram of Cu(II) complex indicated that it was stable upto 120 °C. Thermal decomposition took place in the temperature range of 120-480 °C. First TG loss was observed in the temperature range of 120-340 °C with the loss of ligand (theo. 52 %, exp., 49 %) followed by an exotherm at 310 °C. Second TG loss was in the temperature range of 375-480 °C with the loss of SO<sub>4</sub> in the form of SO<sub>2</sub> + O<sub>2</sub> (theo. 28 %, exp., 27 %) followed by an exotherm at 390 °C. Another DTA peak was observed at 160 °C without TG loss due to melting of the compound. The TG/DTA data of complexes is given in Table-3. The TG/DTA patterns for the Cu(II) complex is shown in Fig. 6.

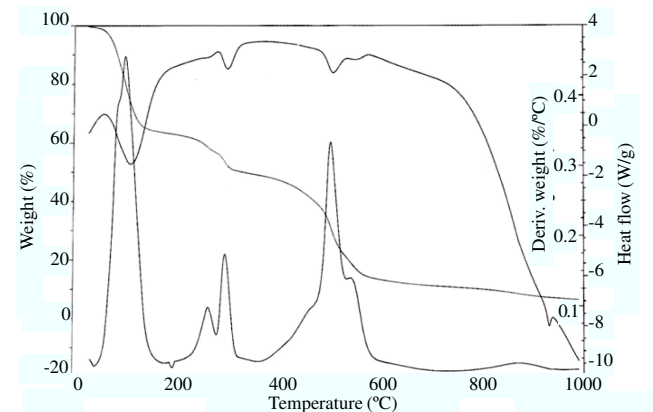


Fig. 4. TGA/DTA pattern of complex (II)

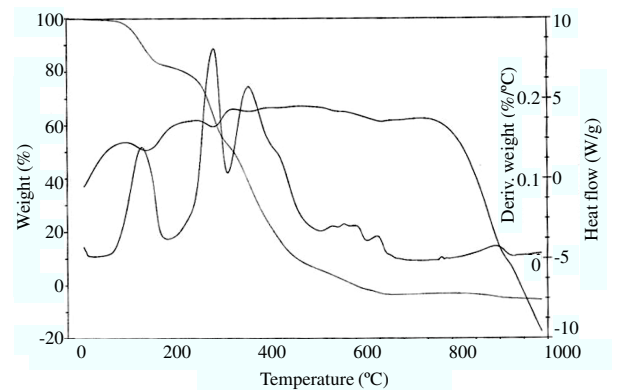


Fig. 6. TGA/DTA pattern of complex (IV)



TABLE-3  
THERMOGRAVIMETRIC AND DIFFERENTIAL THERMAL ANALYSIS (TGA/DTA) OF COMPLEXES

No. of observation	Compound No.	Temperature range (°C)	Weight loss (%) Exp. (Theo).	Decomposition product	Exo/Endo at (°C)
1	Complex(II)	70-130	33 (35)	C <sub>7</sub> H <sub>7</sub> NO	100 exo
		160-395	18 (21)	C <sub>4</sub> H <sub>11</sub> N	290 exo
		440-560	27 (28)	SO <sub>2</sub> + O <sub>2</sub>	500 exo
	Residue	1000		FeO	
2	Complex(III)	30-210	30 (34)	C <sub>7</sub> H <sub>7</sub> NO	90 exo
		265-400	19 (21)	C <sub>4</sub> H <sub>11</sub> N	265 exo
		700-920	26 (28)	SO <sub>2</sub> + O <sub>2</sub>	750 exo
	Residue	1000		ZnO	
3	Complex(IV)	120-340	49 (52)	Ligand	310 exo
		375-480	27 (28)	SO <sub>2</sub> + O <sub>2</sub>	390 exo
			Residue	1000	

Antimicrobial activities of the ligand and its complexes were investigated against some bacteria such as *Bacillus subtilis*, *Escherichia coli*, *Micrococcus luteus*, *Staphylococcus aureus* and *Aspergillus flavus* by diffusion plate method. Ligand showed no antimicrobial activity. It has been observed that antimicrobial activity of copper complex (IV) is greater as compared to zinc complex (III) which is greater than iron complex (II). The antimicrobial activity of complexes is in the following order

Cu complex > Zn complex > Fe complex

The values for antimicrobial activity are shown in the Table-4.

TABLE-4  
ANTIBACTERIAL ACTIVITY EXHIBITED  
BY THE COMPOUNDS

S. No.	Compound	Conc. (mg/mL)	Inhibition zone diameter (nm)				
			EC	SA	ML	BS	AF
1	(C <sub>11</sub> H <sub>18</sub> N <sub>2</sub> )FeSO <sub>4</sub>	10.00	9	10	9	8	10
		25.00	10	10	11	10	9
		50.00	11	10	12	11	11
2	(C <sub>11</sub> H <sub>18</sub> N <sub>2</sub> )ZnSO <sub>4</sub>	10.00	9	9	10	9	10
		25.00	12	10	1	10	10
		50.00	11	12	11	9	11
3	(C <sub>11</sub> H <sub>18</sub> N <sub>2</sub> )CuSO <sub>4</sub>	10.00	10	11	11	10	9
		25.00	12	13	10	12	10
		50.00	13	12	11	13	11
4	Streptomycin	10.00	17	15	18	18	–
5	Control	0.12	–	–	–	–	–

EC: *Escherichia coli*, SA: *Staphylococcus aureus*, ML: *Micrococcus luteus*, BS: *Bacillus subtilis*, AF: *Aspergillus flavus*.

## REFERENCES

- S.A. Lawrence, Amine Synthesis, Properties and Applications, University press, New York, p. 269 (2004).
- B. Stahlbom and B. Akesson, *Int. Arch. Occup. Environ. Health*, **67**, 159 (1995).
- M.F. Mohamed, A.A. Neverov and R.S. Brown, *Inorg. Chem.*, **48**, 1183 (2009).
- Z.L. Lu, A.A. Neverov and R.S. Brown, *Org. Biomol. Chem.*, **3**, 3379 (2005).
- M.C. Rocha, A.M. Santana, S.R. Ananias, E.T. Almeida and A.E. Mauro, *J. Braz. Chem. Soc.*, **18**, 8 (2007).
- N.D. Ghavale, S. Dey and V.K. Jain, *Inorg. Chim. Acta*, **361**, 2462 (2008).
- T. Mahmud, J. Iqbal, M. Imran and V. Mckee, *J. Appl. Sci.*, **7**, 1347 (2007).
- A.C. Moro, A.E. Mauro and S.R. Ananias, *Eclat. Quim.*, **29**, 57 (2004).
- S. Khan, J. Ch. Anwar and M. Arshad, *Radiochim. Acta*, **92**, 501 (2004).
- H.M. Farooq and M.Z. Iqbal, F. Mazher and B.B. Ali, *Acta Sci.*, **9**, 163 (1999).
- A.C. Moroa, A.E. Mauro, A.V.G. Nettoa, S.R. Ananias, M.B. Quillesb, I.Z. Carlosb, F.R. Pavanc, C.Q.F. Leitec and M. Hornerd, *J. Med. Chem.*, **44**, 4611 (2009).
- F. Mazher and M.Z. Iqbal, *J. Pak. Chem. Soc.*, **26**, 127 (2004).
- H.M. Farooq and M.Z. Iqbal, *Acta Sci.*, **4**, 25, 95 (1994).
- H.M. Farooq and M.Z. Iqbal, *J. Sci. Res.*, **25**, 121 (1997).
- H.M. Farooq and M.Z. Iqbal, *J. Sci. Res.*, **27**, 87 (1999).
- H.M. Farooq and M.Z. Iqbal, *Acta Sci.*, **10**, 59, 205, 213, 217, 253 (2000).
- H.M. Farooq and M.Z. Iqbal, *Acta Sci.*, **11**, 25, 29 (2001).
- H.M. Farooq and M.Z. Iqbal, *J. Anal. Appl. Pyrol.*, **52**, 139 (1999).
- V.A.D. Neto, A.E. Mauro, A.C.F. Caires, S.R. Ananias and E.T.D. Almeida, *Polyhedron*, **18**, 413 (1998).
- S. Khan and J. Ch. Anwar, *Radiochim. Acta*, **94**, 23 (2006).
- S. Khan, J. Ch. Anwar, M. Arshad and K. Masud, *Radiochim. Acta*, **94**, 853 (2006).
- M.W. Couch and C.M. Williams, *Org. Mass Spectro.*, **6**, 21 (2005).
- F. Mazher and M.Z. Iqbal, *J. Chem. Soc.*, **32**, 218 (2008).
- S.H. Maron and C.F. Prutton, Principles of Physical Chemistry, Macmillan, New York, edn. 4, p. 801 (1968).
- J. Mohan and N. K. Mehra, Organic Analytical Chemistry, Noros Publishing House Pvt. Ltd., pp. 212-231 (2003).
- I. Nakagawa, R.B. Penland, S. Mizushima, T.J. Lane and J.V. Quagliano, *Spectrochim. Acta*, **9**, 199 (1957).