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Synthesis, Spectral and Thermal Studies on Cobalt(II), Copper(II), Nickel(II) and Zinc(II) Chelates with ptolylsalicylaldimine and Some Amino Acids

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## SYNTHESIS, SPECTRAL AND THERMAL STUDIES ON COBALT(II), COPPER(II), NICKEL(II) AND ZINC(II) CHELATES WITH *p*-TOLYLSALICYLALDIMINE AND SOME AMINO ACIDS

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#### ABSTRACT

New cobalt(II), copper(II), nickel(II) and zinc(II) complexes of the *p*-tolylsalicylaldimine Schiff base (sal-TH) and some amino acids have The complexes have the general formula prepared. been [M(sal-T)(aa)].nH<sub>2</sub>O where M = Co(II), Cu(II), Ni(II) and Zn(II); sal-T = monoanion of the Schiff base *p*-tolylsalicylaldimine; aa = monoanion of the amino acids phenylalanine (Phala), tyrosine (tyr) or methionine (met); n = 0 for M = Co(II), Cu(II) or Ni(II) and n = 2 for M = Zn(II). The complexes have been characterized and studied by elemental analyses, conductivity, IR and UV-VIS spectroscopy as well as thermogravimetry. The Cu(II) complexes have been proposed to possess a square-planar structure while the Co(II), Ni(II) and Zn(II) complexes have an octahedral structure attained by mutual intermolecular interactions among the amino acid-Schiff base molecules. The thermal adjacent mixed

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decomposition reaction order and activation energy have been computed by means of the Coats-Redfern and Horowitz-Metzger methods. The biological activity of selected complexes against some bacteria and fungi have been tested.

## **INTRODUCTION**

There are many publications dealing with metal chelates formed by Schiff bases<sup>1-7</sup>. The Schiff bases derived from salicylaldehyde and different amines are considered to be suitable models for  $B_6$  vitamins<sup>8</sup>. They form stable complexes with metal ions.

Ternary complexes containing an amino acid as a second ligand are of significance as they are regarded as potential models for enzyme metal ion substrate complexes<sup>9</sup>; they have considerable biological activity<sup>10-12</sup>. The purpose of this study is to describe and discuss the interactions of cobalt(II), copper(II), nickel(II) and zinc(II) with a Schiff base sal-TH derived from salicylaldehyde and *p*-toluidine and amino acids as the second ligand. The amino acid used were phenylalanine (phala), tyrosine (tyr) or methionine (met) (Fig. 1). The theraml properties of the solid complexes are discussed.

### **RESULTS AND DISCUSSION**

*p*-Tolylsalicylaldimine (sal-TH) reacts with different metal acetates and amino acids to form mixed-ligand complexes in the molar ratio 1:1:1 according to the following equation:

sal-TH  $\neg$  M(CH<sub>3</sub>COO)<sub>2</sub> + RNH<sub>2</sub>COOH  $\frac{2\text{NaOH}}{+\text{nH}_2\text{O}}$ [M(sal-T)(RNH<sub>2</sub>COO)].nH<sub>2</sub>O + 2CH<sub>3</sub>COONa





Phenylalanine (phala)

p-Tolylsalicylaldimine Schiff base (sal-TH)



Tyrosine (tyr)

CH<sub>3</sub>SCH<sub>2</sub>CH<sub>2</sub>CHNH<sub>2</sub>COOH

methionine (met)

Fig. 1. Structure of the Ligands

where, sal-TH = p-tolylsalicylaldimine; M = cobalt(II), copper(II). nickel(II) or zinc(II); R = PhCH<sub>2</sub>CH, OH-PhCH<sub>2</sub>CH or CH<sub>3</sub>SCH<sub>2</sub>CH<sub>2</sub>CH; n = 0 for M = Co(II), Cu(II) or Ni(II) and n = 2 for Zn(II) complex.

The prepared complexes are listed in Table I together with their colour, decomposition temperature and analytical data. The complexes are soluble in most of organic solvents. The electrical conductivity of the complexes in ethanol (6.7-10.8  $ohm^{-1} cm^2 mol^{-1}$ ) illustrates that the complexes are non electrolytes.

### IR Spectra

The coordination of the *p*-tolylsalicylaldiminate moiety is manifested by the appearance of a band in the region 1600-1630 cm<sup>-1</sup> which indicates bonding of the azomethine nitrogen to the metal center<sup>13</sup>. The IR spectra also display a band in the region 1200-1245 cm<sup>-1</sup> attributable to  $\upsilon$ (C-O).

Table I. Analytical Data, Percent Yields, Colours and Melting Points of the Complexes.

Compound	Mol.	Colour		Found (C	alcd.%)		Yield.	M.p.
(Empirical formula)	Wt.		J	Н	z	M	%	Ŝ
[Co(sal-T)(tyr)]	449.35	brown	61.80	4.97	6.31	12.99	65	240
(C <sub>23</sub> H <sub>22</sub> N <sub>2</sub> O <sub>4</sub> C <sub>0</sub> )			(61.47)	(4.94)	(6.24)	(13.11)		
[Co(sal-T)(phala)]	433.36	brown	63.45	5.09	6.50	13.71	67	205
(C <sub>23</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> C <sub>0</sub> )			(63.74)	(5.12)	(6.46)	(13.60)		
[Cu(sal-T)(phala)]	437.97	pale	63.12	4.99	6.46	14.41	88	176
(C <sub>23</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> Cu)		blue	(63.07)	(5.06)	(6.40)	(14.51)		
[Cu(sal-T)(met)]	421.99	green	54.15	5.30	6.70	14.98	85	180
(C <sub>19</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> SCu)			(54.08)	(5.25)	(6.64)	(15.06)		
[Ni(sal-T)(phala)]	433.14	yellowish	63.88	5.22	6.50	13.60	55	195
(C <sub>23</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> Ni)		green	(63.78)	(5.12)	(6.47)	(13.55)		
[Zn(sal-T)(phala)].2H20	475.83	yellowish	57.99	5.49	5.82	ı	60	232
(C <sub>23</sub> H <sub>26</sub> N <sub>2</sub> O <sub>5</sub> Zn)		white	(58.06)	(5.51)	(5.89)			

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This band is shifted to higher frequency in the complexes indicating coordination of the phenolic oxygen to the metal ion<sup>14,15</sup>.

For the amino acid moiety, the bands observed in the regions 1400-1450 cm<sup>-1</sup> and 1520-1530 cm<sup>-1</sup> are assignable to  $\upsilon_s(COO)$  and  $\upsilon_{as}(COO)$ , respectively. The band in the range 3200-3300 cm<sup>-1</sup> is associated with  $\upsilon(NH_2)$ . The frequencies of the carboxylate and the amino groups point to the bidentate behaviour of the amino acids in the complexes. The bands appearing in the regions 415-425 cm<sup>-1</sup> and 480-498 cm<sup>-1</sup> may be assigned to stretching frequencies of M-N and M-O, respectively.  $\upsilon(OH)$  of water is seen at 3450 cm<sup>-1</sup> for the Zn(II) complex, a position known for water of crystallization<sup>16</sup>. The absence of wagging, twisting and rocking vibrational modes, activated by coordination, is also evidence that coordinated water is absent<sup>17</sup>. The infrared spectral data for the complexes are indicated in Table II.

## Electronic Spectra

The electronic spectral data of the complexes are summarized in Table III. The spectra of the complexes were measured in ethanol solutions. The Co(II) complexes display a d-d band in the region 15,504-15,625 cm<sup>-1</sup>,  ${}^{4}T_{1g} \rightarrow {}^{4}T_{1g}$  (p); that of the Ni(II) complex is at 15,552 cm<sup>-1</sup> ( ${}^{3}A_{2g} \rightarrow {}^{3}T_{1g}$ ) and for the Cu(II) complexes this band appears in the region 15,037-15,151 cm<sup>-1</sup> ( $T_{2g} \rightarrow E_{g}$ ). Bands occuring in the region 26,882-29,154 cm<sup>-1</sup> for all complexes are ascribed to charge transfer transition (L $\rightarrow$ M). The absorption bands which are located in the region 32,258-38,610 cm<sup>-1</sup> may be assigned to intraligand transitions.

Based on the above results a square-planar structure is assigned to the Cu(II) complexes while an octahedral structure is proposed for the

Table II. IR Spectra of the Complexes (cm<sup>-1</sup>).

Compound	υ(C-O)	ν(C-N)	∿₅(COO)	υ <sub>as</sub> (COO)	υ(NH2)	u(OH)	ი(M-O)	N-W)n
[Co(sal-T)(tyr)]	1225 m	1600 s	1440 s	1530 m	3250 m	1	498 w	415 w
[Co(sal-T)(phala)]	1245 s	1600 s	1440 s	1520 m	3250 m	1	498 w	415 w
[Cu(sal-T)(phala)]	1200 m	1610 s	1450 s	1530 m	3250 m	L	495 w	420 w
[Cu(sal-T)(met)]	1200 s	1620 s	1400 s	1520 s	3200 m	T	480 w	420 w
[Ni(sal-T)(phala)]	1200 w	1630 s	1400 m	1530 m	3200 m		495 w	420 w
[Zn(sal-T)(phala)].2H2O	1225 s	1620 s	1440 s	1530 s	3300 m	3450 m	495 w	425 w

Key: s (strong), m (medium), w (weak)

Table III. Electronic Spectral Data of the Complexes (cm<sup>-1</sup>) ( $\epsilon_{max}$ , M<sup>-1</sup> cm<sup>-1</sup>).

Compound	p-p	Charge transfer	Intraligand transition
[Co(sal-T)(tyr)]	15,504 (190)	28,571 (600)	32,258 (11240) - 37,878 (21872)
[Co(sal-T)(phala)]	15,625 (231)	27,174 (562)	32,787 (11239) - 38,314 (21800)
[Cu(sal-T)(phala)]	15,037 (240)	29,154 (912)	33,333 (11105) - 36,630 (20072)
[Cu(sal-T)(met)]	15,151 (221)	26,882 (789)	33,898 (11002) - 37,453 (20765)
[Ni(sal-T)(phala)]	15,552 (433)	28,169 (1001)	32,787 (11650) - 38,610 (211420)
[Zn(sal-T)(phala)]_2H <sub>2</sub> O		27,322 (620)	33,003 (11909) - 36,496 (11790)



Fig. 2. Suggested Molecular Structures of the Complexes

Co(II), Ni(II), and Zn(II) complexes. The six-coordination geometry in the latter complexes may be attained through molecular association. The suggested structures of the complexes are shown in Fig. 2.

## Thermal Behaviour

Thermogravimetric studies of all complexes were carried out in air with a heating rate of  $8^{\circ}$  C min<sup>-1</sup>. The TG and DTG curves of [Co(sal-T)(phala)] show a first step at 208° C which consists of a weight loss of 38.29% which is in good agreement with the theoretical weight loss corresponding to the elimination of the amino acid moiety (38.12%). The rest of the complex decomposes in a second rapid step at 335° C with the formation of CoO as the end product.

The complexes [Cu(sal-T)(phala)], [Cu(sal-T)(met)] and (Ni(sal-T)-(phala)] decompose in two steps. The TG curves of the complexes display weight losses in the first step of 58.90% (265° C), 57.50% (261° C) and



Fig. 3. Suggested Decomposition Mechanism of the Complexes

59.50% (297° C), respectively. These are in good agreement with the loss of  $\cdot$   $\bigcirc$  - CH<sub>3</sub> and the corresponding amino acid (calc. values: 58.52, 56.93 and 59.18%). Dimerization of  $\cdot$   $\bigcirc$  - CH<sub>3</sub> may occur to give the decomposition product H<sub>3</sub>C- $\bigcirc$  - CH<sub>3</sub> (Fig. 3). It is proposed that a proton transfers from the azomethine group to the coordinated amino acid anion. This proton transfer during the thermal decomposition is reported in the literature<sup>18</sup>. The remainder of the complexes decompose in a second step at 490, 465 and 532° C respectively, with the formation of MO as the end products.

Water of crystallization is liberated in the first stage of decomposition of the [Zn(sal-T)(phala)].2H<sub>2</sub>O complex. The mass loss is consistent with loss of two water molecules (found: 7.25%, calc.: 7.57%). The decomposition temperature (100° C) of this step is in agreement with that known for water of crystallization. Attempts to correlate the weight losses

Compound	Step	Ti	Tm	T <sub>f</sub>	Weight loss% Found (Calc.)
[Co(sal-T)(phala)]	1 <u>st</u>	160	208	292	38.29 (38.12)
	2 <u>nd</u>	300	335	401	48.75 (48.52)
[Cu(sal-T)(phala)]	1 <u>st</u>	177	265	335	58.90 (58.52)
	2 <u>nd</u>	451	490	554	26.75 (26.97)
[Cu(sal-T)(met)]	1 <u>st</u>	187	261	326	57.50 (56.93)
	2 <u>nd</u>	419	465	506	27.77 (27.99)
[Ni(sal-T)(phala)]	1 <u>st</u>	200	297	400	59.50 (59.18)
	2 <u>nd</u>	490	532	565	27.05 (27.27)
[Zn(sal-T)(phala)] 2H2O	l <u>st</u>	85	100	120	7.25 (7.57)

Table IV. Thermal Data of the Complexes<sup>a</sup>.

 ${}^{a}T_{i}$  = initial temperature,  $T_{m}$  = maximum temperature and  $T_{f}$  = final temperature.

of the subsequent steps with theoretical mass losses for this complex were not successful due to overlap. The decomposition is completed of around  $600^{\circ}$  C and the overall mass loss (83.25%) is in accord with the theoretical value (82.90%) of ZnO formation as the final product. The TG curve of [Co(sal-T)(try)] shows overlapping of the decomposition stages, which precludes the identification of the decomposition products of this complex. The final product is CoO. The thermal data of the complexes are shown in Table IV.

## Kinetic Data of the Complexes

The kinetic data of each decomposition step are evaluated using Coats-Redfern and Horowitz-Metzger methods<sup>19,20</sup>.

(a) Coats-Redfern

$$\ln\left[\frac{1-(1-\alpha)^{1-n}}{(1-n)T^{2}}\right] = M/T + B \text{ for } n \neq 1$$

$$\ln\left[\frac{-\ln(1-\alpha)}{T^{2}}\right] = M/T + B \text{ for } n = 1$$
(1)

where M = -E/R and  $B = \ln ZR/\phi E$  where E, R, Z and  $\phi$  are the activation energy, gas constant, pre-exponential factor and heating rate, respectively.

(b) The Horowitz-Metzger equation

$$\ln\left[\frac{1-(1-\alpha)^{1-n}}{1-n}\right] = \ln\frac{ZRT_{s}^{2}}{\phi E} - \frac{E}{RT_{s}} + \frac{E\theta}{RT_{s}^{2}} \text{ for } n \neq 1 \quad (3)$$
$$\ln\left[-\ln(1-\alpha)\right] = \frac{E\theta}{RT_{s}^{2}} \text{ for } n = 1 \quad (4)$$

where  $\theta = T - T_s$  is the temperature at the DTG peak.

The correlation coefficient r is computed using the least square method for equations (1), (2), (3) and (4). Linear curves were drawn for different values of n ranging from 0 to 2 in increments of 0.2. The value of n which gave the best fit was choosen as the order parameter for the decomposition stage of interest. The kinetic parameters were calculated from the plots of the left hand side of equations (1) and (2) against 1/T and against  $\theta$  for equations (3) and (4) (Figs. 4 and 5).



Fig. 4. Coats-Redefern Plots for the Two Decomposition Steps of [Ni(sal-T)(phala)] a) First Step, b) Second Step; where  $Y = \ln [1-(1-\alpha)^{1-n}/(1-n)T^2]$  for  $n \neq 1$  or  $Y = \ln [-\ln(1-\alpha)/T^2]$  for n = 1.



Fig. 5. Horowitz-Metzger Plots for the Two Decomposition Steps of [Cu(sal-T)(phala)] a) First Step, b) Second Step; where  $Y = \ln [1-(1-\alpha)^{1-n}/(1-n)T^2]$  for  $n \neq 1$  or  $Y = \ln [-\ln(1-\alpha)]$ for n = 1.

From the observation that the activation energies of the first and second steps of the complexes [Cu(sal-T)(phala)] and [Cu(sal-T)(met)] are only slightly different (Table V), it was assumed that they have the same thermal stability and their decomposition takes place through the same mechanism<sup>21</sup>. [Ni(sal-T)(phala)] seems to be more stable than the above two complexes; this may be attributed to probable strong bonds of Ni(II) towards the Schiff base and the amino acid moieties. The difference of the structure between the Ni(II) complex and the Cu(II) complexes may account for their variable stabilities.

## Thermal Studies of Irradiated Complexes

The decomposition of some <sup>60</sup>Co-gamma irradiated complexes have been studied. The complexes studied were [Cu(sal-T)(phala)], [Cu(sal-T)(met)] and [Ni(sal-T)(phala)]. There is no notable effect in the infrared spectra of the investigated complexes for the doses  $1\times10^5$  and  $3\times10^5$  Gy. But for the dose  $6\times10^5$  Gy a decrease in the intensities is observed. especially for the metal nitrogen band in the 415-425 cm<sup>-1</sup> region. Further, neither a change in the frequency of the absorption bands nor the appearance of new bands have been noticed as a result of  $\gamma$ irradiation (Fig. 6).

The TG curves of the investigated complexes before and after  $\gamma$ -irradiation show the same decomposition pattern with lowering in the decomposition temperature, especially for the Cu(II) complexes (Fig. 7 and Table VI). A decrease in the activation energy is also observed (Table VI). This may be attributed to the creation of a high concentration of electron-hole pairs by radiation which may result in the formation of

			Coats-Re	dfern	Но	prowitz-l	Metzger
Complexes	Step	n	r	Е	n	r	Е
				(kJ/mole)			(kJ/mole)
[Co(sal-T)(phala)]	1 <u>st</u>	0.33	0.997	32	0.33	0,990	34
	2 <u>nd</u>	1.00	0.999	169	1.00	0.998	164
[Cu(sal-T)(phala)]	1 <u>st</u>	0.0	0.997	36	0	1.00	38
	2 <u>nd</u>	1.00	0.991	160	1.00	0.998	163
[Cu(sal-T)(met)]	1 <u>st</u>	0.33	0.996	40	0.33	0.999	42
	2 <u>nd</u>	1.00	0.997	158	1.00	0.997	159
[Ni(sal-T)(phala)]	1 <u>st</u>	0	0.993	50	0	0.997	52
	2 <u>nd</u>	0.66	0.999	189	0.66	0.999	192

Table V. Kinetic Parameters of the Thermal Decomposition of the Complexes.

cation vacancies which in turn could decrease the activation energy of the reaction<sup>22</sup>.

## Microbiological Screening

The biological activities of some of the complexes were tested against a number of bacteria and fungi. The used bacteria were *B. cereus* G +ve, *E. coli* G -ve and *P. aeruginosa* G -ve while the tested fungi were *P. oxaliaum*, *A. niger* and *T. roseun*. The culture media were nutrient agar (N.A) supplemented with one gm yeast/litre. The antibacterial and



Fig. 6. IR spectra of Unirradiated (\_\_\_\_) and  $\gamma$ -Irradiated (\_\_\_\_) [Cu(sal-T)(met)]. Irradiation Dose =  $6 \times 10^5$  Gy.



Fig. 7. TG and DTG Thermograms of Unirradiated (\_\_\_\_) and  $\gamma$ -Irradiated (\_\_\_\_) [Cu(sal-T)(phala)]. Irradiation dose =  $6 \times 10^5$  Gy.

Table VI. Thermal Data and Kinetic Parameters of Irradiated Complexes at Irradiation Dose 6x105 Gya.

Aetzger	ш	(kJ/mole)	22	150	27	129	47	185
rowitz-N	۲		666.0	0.996	066.0	1.00	066.0	0.999
Hc	u		0	1.00	0.33	1.00	0	0.66
sdfern	ш	(kJ/mole)	21	149	25	127	46	182
Coats-Re	L		666'0	0.994	0.996	0.998	066.0	0.997
	E		0.0	1.00	0.33	1.00	0	0.66
	Tf	;	321	530	312	491	396	557
	T_m		242	465	244	442	294	525
	Ę,		165	433	180	407	197	486
	Step		1 <u>st</u>	2nd	1 <u>st</u>	2nd	1 <u>st</u>	2 <u>nd</u>
	Complexes		[Cu(sal-T)(phala)]		[Cu(sal-T)(met)]		[Ni(sal-T)(phala)]	

 ${}^{a}Gy = gray$ 

antifungal activity of each compound were evaluated by the classical filter paper technique. For each complex, a weight of 0.01 g was dissolved in 5 mL DMF. The complexes show variable antibacterial and antifungal activities against the used bacteria and fungi (Table VII).

#### <u>EXPERIMENTAL</u>

#### Materials and Methods

All amino acids viz., phenylalanine, methionine and tyrosine were BDH grade.

Chemical analyses for C, H, N were made on a 240 Perkin-Elmer elemental analyser, while Co, Ni and Cu were determined by a Perkin-Elmer 3110 atomic absorption spectrophotometer. The infrared spectra were obtained in the solid state (KBr pellets) on a 470 Shimadzu infrared spectrophotometer. Electronic spectra were recorded on a UV-2101 PC Shimadzu spectrophotometer. The thermogravimetric analyses were determined using Sartorius 200 MP electrobalance converted to a thermobalance by the addition of a small furnace and sample holder. The temperature was measured using a chromal-alumal thermocouple attached to a soar MF-550 digital multimeter, the heating rate was adjusted to be 8° C min<sup>-1</sup>.

For the gamma irradiation, the samples were kept in small glass containers and irradiated for the appropriate irradiation times at ambient temperature using a <sup>60</sup>Co gamma cell having a dose rate of 1.1 Gy/S.

### Synthesis of the Ligand

p-Tolylsalicylaldimine was prepared according to the recommended literature procedure<sup>2</sup>.

Table VII. Microbiological Screening of the Complexes.

		Bacteria			Fungi	
Complex	B. cereus	E. coli	P. aeruginosa	P. oxaliaum	A. niger	T. roseun
	G+ve	G-ve	G-ve			
[Co(sal-T)(tyr)]	1	+	1	+	,	I
[Cu(sal-T)(phala)]	+	+	‡	‡	+	‡
[Cu(sal-T)(met)]	+	‡	+	1	‡	1
[Zn(sal-T)(phala)].2H <sub>2</sub> O	+	+	‡	‡	+	+

The diameter(D) of the inhibition zone (in mm) is expressed as "+" and "-"

"++" D = 15-20 "+" D = 1.15 "-"D = 0

#### Preparation of the Complexes

Preparation of the complexes followed essentially the same procedure. The preparation of (*p*-tolylsalicylaldiminato)(phenylalaninato)nickel(II) is typical.

A quantity of 0.422 g (2.0 mmol) of *p*-tolylsalicyaldimine in 20 mL hot absolute ethanol was added to 0.50 g (2.0 mmol) of nickel acetate tetrahydrate. The solution mixture was treated with 0.33 g (2.0 mmol) phenylalanine in 20 mL 0.1 N aqueous sodium hydroxide solution. A vellowish green solid deposited after refluxing for three hours. It was filtered off, washed with 50% ethanol and dried over  $P_4O_{10}$ .

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