

## Electronic Spectral Characterisation of Modified Doped Systems of Tm(III) Ion With Various Sulphonanilides in DMSO Medium

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**Abstract:** Tm(III) systems with six sulphonanilides ligands were prepared on modified doped model pattern in DMSO solvent and examined for metal-ligand interaction on the basis of different parameters such as intensity parameters (oscillator strength-P, Judd-Ofelt parameters- $T_2$ ,  $T_4$  and  $T_6$  r.m.s. deviation- $\sigma$ ), bonding parameters (Nephelauxetic ratio- $\beta$ , bonding parameter- $b^{1/2}$ , Sinha's covalency parameter- $\delta\%$ , covalency angular overlap parameter- $\eta$ ), symmetry parameter- $T_4/T_6$ , co-ordination parameter- $T_4/T_2$ , thermodynamic parameter (thermodynamic efficiency of transition-TET, partition function of transition-Q, ratio of partition- $r_p$ ) & Peacock constant- $K'$ . Electronic spectral data suggests covalency in M-L bond.

**Keywords:** Tm(III) ion, Electronic spectral parameters, sulphonanilides.

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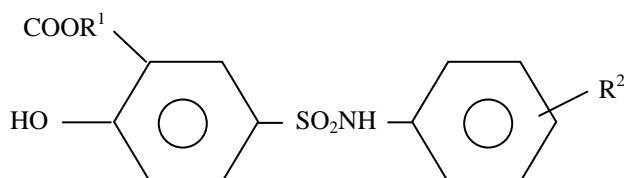
### Introduction

The tri-positive rare earth metal ions show comparatively little tendencies to form complexes with a variety of coordinating agents due to their peculiar electronic configurations. However it has been observed that the f-f spectra of lanthanide ion is affected when ligand environment and solvent is changed<sup>(1-5)</sup>, which suggests the involvement of f-orbital in metal-ligand interaction. In case of f-block lanthanide metal complexes, a limited study has so far been carried out. Systematic study of lanthanide complexes began only after the publication of Judd-Ofelt theory<sup>(6-7)</sup> in 1962. Several lanthanide complexes were prepared with the ligands possessing "O" and "N" as donar atoms and examined for various electronic-spectral parameters resulting from f-f transitions<sup>(1-5,8)</sup>. The complexing ability has been reported poor in case of lanthanide complexes and also the stability of lanthanide complexes is found similar to  $[Ag(NH_3)_2]^+$  type of complex, which makes the isolation of these complexes in solid state difficult. Owing to the poor thermodynamic stability of lanthanide complexes, a modified doped model<sup>(9)</sup> technique will be taken as system in the present electronic-spectral study. So complexes of Tm(III) ions will be synthesized with substituted sulphonanilides for electronic-spectral study.

### Experimental

Six systems were prepared by using standard grade chemicals  $TmCl_3 \cdot 6H_2O$  & ligands  $L_1$  to  $L_6$  (table I) in DMSO medium by using standard method<sup>(10)</sup>. Solution spectra have been taken for Tm(III) systems by standard spectrophotometer in the range 400-820 nm. A simplified representation of sulphonanilides (ligands  $L_1$  to  $L_6$ ) is given in table-I

Nine ( $L_1$  to  $L_6$ ) ligands can be represented as follows:-



**Table –I**  
Representation Of Sulphonanilides

Ligands	Group & their representation	
	R <sup>2</sup>	R <sup>1</sup>
L <sub>1</sub>	o-nitro	H
L <sub>2</sub>	p- nitro	H
L <sub>3</sub>	o-nitro	CH <sub>3</sub>
L <sub>4</sub>	p- nitro	CH <sub>3</sub>
L <sub>5</sub>	o-nitro	C <sub>2</sub> H <sub>5</sub>
L <sub>6</sub>	p- nitro	C <sub>2</sub> H <sub>5</sub>

**Calculation Of Various Parameters**

**(I) Intensity parameters**

**(a) Oscillator strength (P):**

In Tm(III) doped systems, we observed four peaks<sup>(11-12)</sup> due to <sup>1</sup>G<sub>4</sub>, <sup>3</sup>F<sub>2</sub>, <sup>3</sup>F<sub>3</sub> & <sup>3</sup>H<sub>4</sub>. The intensity of an absorption band is measured by Oscillator strength, which is directly proportional to area under the absorption curve.

$$P = 4.315 \times 10^{-9} \int \epsilon \, d\nu \quad \text{Where } \epsilon = \text{molar absorptivity}$$

**(b) r.m.s. deviation (σ):**

From the calculated and observed values of Oscillator strength, the values of r.m.s. deviation for systems have been computed by following equation -

$$\sigma = \left[ \frac{\sum (P_{\text{cal}} - P_{\text{obs}})^2}{N} \right]^{1/2}$$

**(c) Judd-Ofelt parameters-T<sub>2</sub>, T<sub>4</sub> and T<sub>6</sub>:**

The interaction between M-L are predominantly measured by three main parameter known as Judd-Ofelt parameters<sup>(6-7)</sup> (T<sub>2</sub>, T<sub>4</sub> and T<sub>6</sub>).

**(II) Symmetry parameter<sup>(13-14)</sup>**

Judd-Ofelt parameter ratio T<sub>4</sub>/T<sub>6</sub> is called Symmetry-parameter.

**(III) Co-ordination parameter<sup>(13-14)</sup>**

Judd-Ofelt parameter ratio T<sub>4</sub>/T<sub>2</sub> is called Co-ordination parameter.

**(IV) Hypersensitive transitions<sup>(15-16)</sup>**

The bands having oscillator strength ~10<sup>-5</sup> and found much sensitive to the ligands & solvents are called hypersensitive transitions. For Tm(III): <sup>3</sup>H<sub>6</sub> – <sup>3</sup>H<sub>4</sub> is hypersensitive transitions in four bands.

**(V) Peacock constant<sup>(8)</sup> (K')**

Oscillator strength (P) is directly proportional to νT<sub>6</sub> for hypersensitive transitions, this linear correlation has been proposed by **R. D. Peacock<sup>(8)</sup>**.

$$K' = P_{\text{obs}} / \nu T_6.$$

**(VI) Bonding parameters<sup>(17-19)</sup>**

**(a) Nephelauxetic ratio (β):**

The shifting of electronic spectral bands during complexation can be represented in terms of Nephelauxetic ratio (β) as follows-

$$\beta = \nu_c / \nu_f$$

Where:- ν<sub>c</sub> & ν<sub>f</sub> are wave numbers of f-f transition for spectra of complex and free ion respectively.

**(b) Bonding parameter (b<sup>1/2</sup>):**

$$b^{1/2} = [ \frac{1}{2} (1-\beta) ]^{1/2}$$

**(c) Sinha's covalency parameters (δ%):**

$$\delta = [1 - \beta / \beta] \times 100$$

**(d) Covalency angular overlap parameter (η):**

$$\eta = [1 - \beta^{1/2} / \beta^{1/2}]$$

**(VII) Thermodynamic parameters<sup>(20-22)</sup>**

Work Function for transition

(a) Thermodynamic Efficiency =  
of transition (TET)

Energy absorbed for transition

By using thermodynamic relation-

$$A = E - TS \text{ and } S = K \ln P_{\text{obs}}$$

Following relation may be obtained-

$$A = E - KT \ln P_{\text{obs}}$$

Where:-

A = Work function (cm<sup>-1</sup>)

E = Energy absorbed for hypersensitive transition (cm<sup>-1</sup>)

K = Boltzmann Constant , T = Absolute temp

P<sub>obs</sub> = Oscillator strength of transition, S = Absolute energy.

(b) Partition Function of Transition (Q) =  $g_i e^{-E_i/KT}$

Where: For Tm -  $g_i = 2J + 1 = 9$

Q for lanthanide ion system (doped)

(c) Ratio of Partition (r<sub>p</sub>) =

Q for lanthanide ion (free ion in solvent)

Various parameters for Tm(III) systems were calculated by using partial and multiple regression method, which are tabulated in tables II to VI .

**Table II**

Observed And Calculated Values Of Oscillator Strength (P) And Judd Ofelt Parameters (T<sub>λ</sub>) Of The Bands Recorded For Tm(III) Ion Systems Involving Ligand Environment (1:2 Molar Ratio) In DMSO Solvent

levels	Bands	Tm(III) WITH L <sub>1</sub>		Tm(III) WITH L <sub>2</sub>	
		P <sub>obs</sub> X 10 <sup>6</sup>	P <sub>cal</sub> X 10 <sup>6</sup>	P <sub>obs</sub> X 10 <sup>6</sup>	P <sub>cal</sub> X 10 <sup>6</sup>
		<sup>1</sup> G <sub>4</sub>	2.9252	3.4063	3.1038
	<sup>3</sup> F <sub>2</sub>	0.4453	0.7847	0.4809	0.8673
	<sup>3</sup> F <sub>3</sub>	7.1591	7.4847	7.9598	8.3292
	<sup>3</sup> H <sub>4</sub>	7.4054	7.6909	7.8359	8.1608
	r.m.s. dev. X 10 <sup>7</sup>	3.6543		4.1456	
T <sub>λ</sub> parameter	T <sub>2</sub> X 10 <sup>9</sup>	1.542266		1.584984	
	T <sub>4</sub> X 10 <sup>9</sup>	1.097198		1.231620	
	T <sub>6</sub> X 10 <sup>9</sup>	0.201279		0.222465	
	T <sub>4</sub> / T <sub>6</sub>	5.451137		5.536233	
	T <sub>4</sub> / T <sub>2</sub>	0.711419		0.777055	

**Table III**

Observed And Calculated Values Of Oscillator Strength (P) And Judd Ofelt Parameters (T<sub>λ</sub>) Of The Bands Recorded For Tm(III) Ion Systems Involving Ligand Environment (1:2 Molar Ratio) In DMSO Solvent

levels	Bands	Tm(III) WITH L <sub>3</sub>		Tm(III) WITH L <sub>4</sub>	
		P <sub>obs</sub> X 10 <sup>6</sup>	P <sub>cal</sub> X 10 <sup>6</sup>	P <sub>obs</sub> X 10 <sup>6</sup>	P <sub>cal</sub> X 10 <sup>6</sup>
		<sup>1</sup> G <sub>4</sub>	2.789875	2.492026	4.311733
	<sup>3</sup> F <sub>2</sub>	0.744085	0.533707	1.267967	2.016258
	<sup>3</sup> F <sub>3</sub>	6.048233	5.846387	10.350000	11.076720
	<sup>3</sup> H <sub>4</sub>	5.389475	5.212435	15.293850	15.929253
	r.m.s. dev. X 10 <sup>7</sup>	2.2641		8.1063	
T <sub>λ</sub> parameter	T <sub>2</sub> X 10 <sup>9</sup>	0.969309		3.527779	
	T <sub>4</sub> X 10 <sup>9</sup>	0.913665		1.027779	
	T <sub>6</sub> X 10 <sup>9</sup>	0.137304		0.525352	
	T <sub>4</sub> / T <sub>6</sub>	6.654320		1.956361	
	T <sub>4</sub> / T <sub>2</sub>	0.942594		0.291339	

**Table IV**

Observed And Calculated Values Of Oscillator Strength (P) And Judd Ofelt Parameters ( $T_\lambda$ ) Of The Bands Recorded For Tm(III) Ion Systems Involving Ligand Environment (1:2 Molar Ratio) In DMSO Solvent

levels	Bands	Tm(III) WITH L <sub>5</sub>		Tm(III) WITH L <sub>6</sub>	
		P <sub>obs</sub> X 10 <sup>6</sup>	P <sub>cal</sub> X 10 <sup>6</sup>	P <sub>obs</sub> X 10 <sup>6</sup>	P <sub>cal</sub> X 10 <sup>6</sup>
	<sup>1</sup> G <sub>4</sub>	2.457241	2.996491	3.631338	3.349550
	<sup>3</sup> F <sub>2</sub>	0.489900	0.867577	1.272161	1.075013
	<sup>3</sup> F <sub>3</sub>	6.295100	6.662080	8.994518	8.802915
	<sup>3</sup> H <sub>4</sub>	7.125975	7.445098	8.031722	7.863195
	r.m.s.dev. X 10 <sup>7</sup>	4.0925		2.1411	
T <sub>λ</sub> parameter	T <sub>2</sub> X 10 <sup>9</sup>	1.525788		1.372674	
	T <sub>4</sub> X 10 <sup>9</sup>	0.850077		1.189054	
	T <sub>6</sub> X 10 <sup>9</sup>	0.224374		0.282186	
	T <sub>4</sub> / T <sub>6</sub>	3.788669		4.213727	
	T <sub>4</sub> / T <sub>2</sub>	0.557140		0.866232	

**Table V**

Computed Values Of  $\beta$ ,  $b^{1/2}$ ,  $\delta$  &  $\eta$  For Tm(III) Doped Systems In DMSO Solvent

S. No.	Tm(III) doped systems	Average energy [in cm <sup>-1</sup> ]	$\beta$	$b^{1/2}$	$\delta$	$\eta$
1	Tm(III)-L <sub>1</sub>	12706.48	0.998937	0.023053	0.1064	0.000531
2	Tm(III)-L <sub>2</sub>	12698.41	0.998303	0.029130	0.1700	0.000849
3	Tm(III)-L <sub>3</sub>	12674.27	0.996405	0.042397	0.3608	0.001802
4	Tm(III)-L <sub>4</sub>	12626.26	0.992631	0.060701	0.7424	0.003705
5	Tm(III)-L <sub>5</sub>	12658.23	0.995144	0.049276	0.4880	0.002437
6	Tm(III)-L <sub>6</sub>	12618.30	0.992004	0.063228	0.8060	0.004021

**Table VI**

Thermodynamic Property

S. N.	Tm(III) doped systems	Energy for hypersensitive transition ( <sup>3</sup> H <sub>4</sub> ) (cm <sup>-1</sup> )	Oscillator strength for hypersensitive transition ( <sup>3</sup> H <sub>4</sub> ) (X 10 <sup>6</sup> )	Work function (A) (cm <sup>-1</sup> )	Thermo dynamic efficiency of the transition (TET)	Partition function Q=g <sub>e</sub> e <sup>-E/KT</sup> (X 10 <sup>40</sup> )	Ratio of partition function (r <sub>p</sub> )	Peacock relation (K') = P <sub>obs</sub> / vT <sub>6</sub>
1.	Tm(III)- L <sub>1</sub>	12706.48	7.405425	15167.78	1.193704	2.939415	1.067041	2.895522
2.	Tm(III)- L <sub>2</sub>	12698.41	7.835977	15147.94	1.192900	3.055465	1.109169	2.773840
3.	Tm(III)- L <sub>3</sub>	12674.27	5.389475	15201.78	1.199420	3.430828	1.245430	3.096992
4.	Tm(III)- L <sub>4</sub>	12626.26	15.29385	14936.46	1.182968	4.319866	1.568161	2.305639
5.	Tm(III)- L <sub>5</sub>	12658.23	7.125975	15127.54	1.195076	3.705446	1.345119	2.508995
6.	Tm(III)- L <sub>6</sub>	12618.30	8.031721	15062.68	1.193718	4.488231	1.629279	2.255655

Where:- K = 0.6945, T = 300K

### Results And Discussion

The values of various electronic spectral parameters and thermodynamic parameter have been reported in table-II to VI.

#### Electronic Spectral Parameters

Variation in Symmetry parameter (T<sub>4</sub>/T<sub>6</sub>) & co-ordination parameter (T<sub>4</sub>/T<sub>2</sub>) values show the change in symmetry & co-ordination environment around central metal ion.

- T<sub>4</sub>/T<sub>6</sub> values for Tm(III) systems were found between 1.956361 & 6.65432 and the order of T<sub>4</sub>/T<sub>6</sub> values was found as given below-  
Tm(III)-L<sub>4</sub> < Tm(III)-L<sub>5</sub> < Tm(III)-L<sub>6</sub> < Tm(III)-L<sub>1</sub> < Tm(III)-L<sub>2</sub> < Tm(III)-L<sub>3</sub>
- T<sub>4</sub>/T<sub>2</sub> values for Tm(III) systems were found between 0.291339 & 0.942594 and the order of T<sub>4</sub>/T<sub>2</sub> values was found as given below-  
Tm(III)-L<sub>4</sub> < Tm(III)-L<sub>5</sub> < Tm(III)-L<sub>1</sub> < Tm(III)-L<sub>2</sub> < Tm(III)-L<sub>6</sub> < Tm(III)-L<sub>3</sub>

**From the calculated and observed values of Oscillator strength, the values of r.m.s. deviation for all systems have been computed, low value of r.m.s. deviation prove Judd Ofelt Theory .:**

$Tm(III)-L_5 < Tm(III)-L_3 < Tm(III)-L_1 < Tm(III)-L_2 < Tm(III)-L_6 < Tm(III)-L_4$

The observed oscillator strength values for  ${}^1G_4$ ,  ${}^3F_2$ ,  ${}^4F_3$  and  ${}^3H_4$  band were found between  $2.4572 \times 10^{-6}$  &  $4.3117 \times 10^{-6}$ ,  $0.4453 \times 10^{-6}$  &  $1.2721 \times 10^{-6}$ ,  $6.0482 \times 10^{-6}$  &  $10.3500 \times 10^{-6}$  and  $7.1259 \times 10^{-5}$  &  $15.2938 \times 10^{-6}$  respectively.

- The values of rms deviation ( $\sigma$ ) for Tm(III) systems were found between  $2.1411 \times 10^{-7}$  &  $8.1063 \times 10^{-7}$  and the order of rms deviation was found as given below-

$Tm(III)-L_6 < Tm(III)-L_3 < Tm(III)-L_1 < Tm(III)-L_5 < Tm(III)-L_2 < Tm(III)-L_4$

**Constant values of Peacock relation signifies the validity of Judd-Ofelt theory.**

- The peacock constant ( $K_1'$ ) for Tm(III) systems varies from 2.255655 to 3.096992 and its average value is 2.639441. All systems exhibit constancy in value of  $K_1'$  and also with mean value

**Judd -Ofelt parameters are indicative of degree of metal ligand (M-L) interaction ( $T_2$ ), refractive index of medium ( $T_4$ ) and change in symmetry around the cation ( $T_6$ ).**

$T_2$  values for Tm(III) systems were found between  $0.969309 \times 10^{-9}$  &  $3.527779 \times 10^{-9}$  and the order of  $T_2$  values were found as given below-

$Tm(III)-L_3 < Tm(III)-L_6 < Tm(III)-L_5 < Tm(III)-L_1 < Tm(III)-L_2 < Tm(III)-L_4$

- $T_4$  values for Tm(III) systems were found between  $0.850077 \times 10^{-9}$  &  $1.231620 \times 10^{-9}$  and the order of  $T_6$  values was found as given below-

$Tm(III)-L_5 < Tm(III)-L_3 < Tm(III)-L_4 < Tm(III)-L_1 < Tm(III)-L_6 < Tm(III)-L_2$

- $T_6$  values for Tm(III) systems were found between  $0.137304 \times 10^{-9}$  &  $0.525352 \times 10^{-9}$  and the order of  $T_6$  values was found as given below-

$Tm(III)-L_3 < Tm(III)-L_1 < Tm(III)-L_2 < Tm(III)-L_5 < Tm(III)-L_6 < Tm(III)-L_4$

**Nephelauxetic ratio ( $\beta$ ) depicts that how effectively bands of metal ions are shifted in presence of ligand environment. Low and high value of bonding parameters ( $\beta$ ,  $\delta$ ,  $\eta$ ) indicate weak & strong covalent character in metal-ligand bond respectively.**

- The values of Nephelauxetic ratio for Tm(III) systems were found between 0.992004 & 0.998937 and the order of Nephelauxetic ratio was found as given below-

$Tm(III)-L_6 < Tm(III)-L_4 < Tm(III)-L_5 < Tm(III)-L_3 < Tm(III)-L_2 < Tm(III)-L_1$

**Positive value of bonding parameter( $b^{1/2}$ ) indicates covalent character in metal-ligand bond while negative value shows ionic character in metal-ligand interaction.**

- The values of  $b^{1/2}$  for Tm(III) systems were found between 0.023053 & 0.063228 and the order of  $b^{1/2}$ -value was found as given below-

$Tm(III)-L_1 < Tm(III)-L_2 < Tm(III)-L_3 < Tm(III)-L_5 < Tm(III)-L_4 < Tm(III)-L_6$

**Percentage covalent character in metal ligand bond can be expressed by Sinha's covalency parameter ( $\delta\%$ ). Covalency angular overlap parameter ( $\eta$ ) represents extent of overlapping between metal ion orbital and ligand orbital.**

**Sinha's covalency parameter- $\delta\%$  & covalency angular overlap parameter- $\eta$  also indicate the covalent character in metal ligand bond.**

The values of Sinha's Covalency parameters ( $\delta\%$ ) were found between 0.1064 to 0.8060 and values of Covalency angular overlap parameter ( $\eta$ ) were found between 0.000531 to 0.004021.

### Thermodynamic Parameters

- The values of work function for Tm(III) systems were found between 21306.16 & 21559.83, and the order of work function was found as given below-

$Tm(III)-L_4 < Tm(III)-L_6 < Tm(III)-L_5 < Tm(III)-L_2 < Tm(III)-L_1 < Tm(III)-L_3$

- The values of TET for Tm(III) systems were found between 1.182968 & 1.199420, and the order of work function was found as given below-

$Tm(III)-L_4 < Tm(III)-L_2 < Tm(III)-L_1 < Tm(III)-L_6 < Tm(III)-L_5 < Tm(III)-L_3$

- The values of partition function and ratio of partition function for Tm(III) systems were found between  $2.939415 \times 10^{-40}$  &  $4.488231 \times 10^{-40}$  and 1.067041 & 1.629279 respectively.

Thus, the order of partition function and ratio of partition function are found as given below-

$Tm(III)-L_1 < Tm(III)-L_2 < Tm(III)-L_3 < Tm(III)-L_5 < Tm(III)-L_4 < Tm(III)-L_6$

**The significance of thermodynamic parameters are well understood but their computation from spectroscopic data proposes the microscopic behavior of the f-f transition. The present study finds that the microscopic behavior with respect to TET for Tm(III) doped systems.**

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