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Research Paper

Interaction of Organotin (IV) Moieties with O-O Donor Ligands

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Abstract: Equilibrium studies of dimethyltin (DMT) (IV) and dioctyltin (DOT) (IV) cation with oxygen donor ligands were carried out potentiometrically for 1:1 systems in 10% ethanol medium at three ionic strengths ($\mu = 0.05, 0.10, 0.15M$) and at three different temperatures 20°C, 30°C, 40°C. The participation of different ligand functional group in binding to organotin is discussed. Thermodynamic formation constants of protonated, non protonated and hydroxo complexes have been calculated. Stability constants of generalized species (SCOGS) computer program was applied for computation. The concentration distribution of the various complex species in solution has been evaluated as a function of pH and is presented in the form of speciation curves.

Keywords: Diorganotin complexes (iv), Hydroxo complex, non protonated species, protonated species

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Introduction

The coordination chemistry of tin is extensive with various geometries and coordination number known for both inorganic and organometallic complexes^[1]. The organotin complexes have been of great interest because of their versatile bonding modes^[2-8] and biological significance^[9-21]. Organotin carboxylates are of interest in view of their structural diversity. Depending on the type of carboxylic acid, the complexation equilibria of organotin (IV) cation is expected to differ from each other.

It is evident from the literature that oxygen donor ligands coordinate to organotin moiety in monodentate or bidentate fashion depending on the steric and electronic attributes of organic substituents on tin and/ or the carboxylate moiety. Higher coordination number can be generated either by inter and/or intramolecular interaction especially in complexes where tin bonds to electronegative atom such as oxygen and nitrogen. However, no considerable attention is devoted to investigate the metal ligand equilibria involving DOT (IV) cation in solution. The present communication describes the results of equilibrium studies of DMT (IV) and DOT (IV) with O-O donor ligands in 1:1 binary systems.

Material and Methods

All the chemicals used were of analytical grade and the solutions of ligands were prepared in doubly distilled CO₂ free water. DMT (IV) and DOT (IV) metal were prepared by dissolving their accurate weighed amounts in ethanol. An Elico digital pH-meter model LI-127 with ATC probe and combined electrode type (CL-51B-Glass body, range 0-14 pH unit, 0-100°C automatic/manual) with accuracy ± 0.01 was used for pH measurement. The pH meter was calibrated with aqueous buffers (pH 4.0 and 9.20) before and after titration.

Following sets of solutions/ titration mixture were prepared with the ratio of 10% alcohol and 90% water by keeping total volume 50mL and titrated against 0.10M NaOH solution, ionic strengths ($\mu = 0.05, 0.10, 0.15M$) is maintained by adding different concentration of NaNO₃ solution to each titration mixture at temperature 20 \pm 1°C, 30 \pm 1°C and 40 \pm 1°C.

The complex of each titration mixtures is as follows:-

1. Mixture 1 :- HNO₃ (2.0 x 10⁻³M) (Acid titration)
2. Mixture 2 :- HNO₃ (2.0 x 10⁻³M) + ligand (1.0 x 10⁻³M) (Ligand titration)
3. Mixture 3:- HNO₃ (2.0 x 10⁻³M) + ligand (1.0 x 10⁻³M) + Metal (1.0 x 10⁻³M) (Metal : Ligand (1:1) titration)

Results and Discussion

The acid dissociation constants of dicarboxylic acids were determined by the method of Martell and Chaberek as modified by Dey et al. [22 - 25]. The values of the dissociation constants of oxalic acid, malonic acid, succinic acid, salicylic acid, sulfosalicylic acid and dinitrosalicylic acid were determined under the same experimental conditions

[20°C, 30°C and 40°C), $\mu=0.05, 0.10$ and $0.15M$ $NaNO_3$] which are used for the study of organotin (IV) complex equilibria. The value of dissociation constants obtained coincide well with the literature values [26-29]. The SCOGS [30-32] computer program was used to refine the overall protonation constants. The pK_a values as born out for acids are given in Table 1.

Table 1: Protonation Constant of Ligands at different Temperatures and Ionic Strengths

Parameters	20°C				30°C				40°C			
	0.05 M	0.10M	0.15M	$\mu \rightarrow 0$	0.05 M	0.10M	0.15M	$\mu \rightarrow 0$	0.05 M	0.10M	0.15M	$\mu \rightarrow 0$
Oxalic acid												
$\log \beta_{HL}$	4.65	4.47	4.38	4.50	4.29	4.21	4.03	4.55	4.21	4.18	4.02	4.40
$\log \beta_{H2L}$	7.25	7.21	6.61	7.50	6.88	6.74	5.25	7.30	4.74	4.52	4.26	5.30
Malonic acid												
$\log \beta_{HL}$	7.06	6.80	6.76	7.60	6.26	6.24	6.20	6.50	6.22	5.57	5.83	6.23
$\log \beta_{H2L}$	12.42	12.31	12.00	12.32	11.03	10.96	10.95	11.30	10.71	9.91	9.55	11.28
Succinic acid												
$\log \beta_{HL}$	5.56	5.55	5.48	6.65	5.51	5.45	5.40	5.80	5.35	5.30	5.13	5.70
$\log \beta_{H2L}$	9.97	9.88	9.87	10.45	9.79	9.63	9.60	10.15	9.50	9.35	9.12	10.10
Salicylic acid												
$\log \beta_{HL}$	11.17	10.24	10.02	12.70	10.53	10.23	10.00	11.40	9.61	9.58	9.52	9.80
$\log \beta_{H2L}$	14.87	13.86	13.83	16.20	13.67	13.44	13.02	14.10	12.65	12.59	12.34	12.90
5-Sulfosalicylic acid												
$\log \beta_{HL}$	10.75	9.90	9.20	12.60	9.42	9.33	9.00	10.10	9.04	8.92	8.88	9.40
$\log \beta_{H2L}$	14.22	13.56	13.08	15.60	12.48	12.34	12.08	13.00	12.22	12.01	11.76	12.70
3-5 Dinitrosalicylic acid												
$\log \beta_{HL}$	8.29	8.17	8.00	9.10	8.04	7.94	7.41	8.60	7.66	7.40	7.27	8.30
$\log \beta_{H2L}$	12.08	11.69	10.83	13.05	11.56	10.60	10.00	12.90	10.62	9.98	9.87	11.60

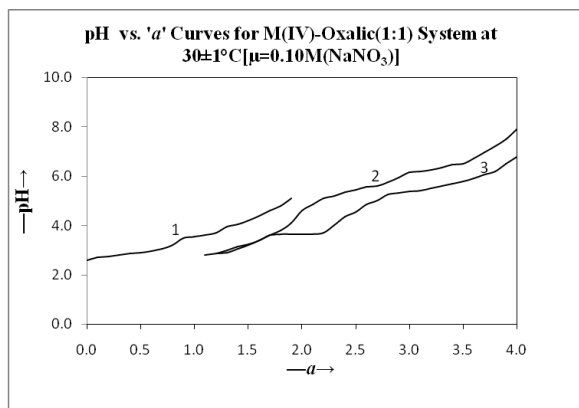


Figure 1

The stability constant of 1:1 binary complexes of the chosen ligands were determined at three different temperatures (20°C, 30°C and 40°C) and at three different ionic strengths $\mu = 0.05, 0.10, 0.15M$ $NaNO_3$ on the basis of titration curves obtained by plotting pH against moles of alkali per moles of metal/ligand ('a') representative titration curves are given in Figure 1 and 2.

Analysis of the complexed ligand curves in each figure indicates that the free ligand solutions shift the buffer region of the metal-ligand to lower

pH values. This shows that complexation reaction proceeds by releasing of protons from such ligands. The thermodynamic formation constant of the 1:1 binary species are tabulated in Table 2 to 7, it is observed that the formation of MLH species occur in acidic pH range.

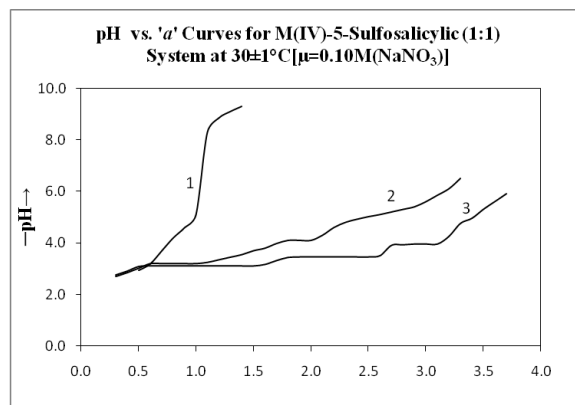


Figure 2

Figure: 1 and 2. pH vs a curves at 30°C [μ=0.10M(NaNO₃)] curve (1) respective ligand titration, curve(2) DMT(IV)-metal -ligand titration, curve (3) DOT(IV)-metal ligand titration

Table 2

Parameter	DMT(IV)-Oxalic			DOT(IV)-Oxalic		
	20°C	30°C	40°C	20°C	30°C	40°C
$\log \beta_{ML}$	7.05	6.90	5.20	7.30	5.80	5.30
$\log \beta_{ML(OH)}$	12.30	12.20	12.18	12.55	12.20	11.70
$\log \beta_{ML(OH)_2}$	15.75	15.70	15.00	15.30	15.20	15.00
$\log K_{ML}^N$	5.00	4.95	4.90	4.70	4.65	4.60
$\log K_{ML(OH)}^N$	7.20	7.12	7.10	7.82	7.80	7.36
$\log K_{ML(OH)_2}^N$	10.90	10.80	10.60	10.80	10.70	10.60

Table 3

Parameter	DMT(IV)-Malonic			DOT(IV)-Malonic		
	20°C	30°C	40°C	20°C	30°C	40°C
$\log \beta_{MLH}$	11.95	11.70	11.40	-	-	-
$\log \beta_{ML}$	8.20	8.10	7.55	9.90	9.10	8.56
$\log \beta_{ML(OH)}$	19.00	18.90	17.95	20.10	19.70	18.80
$\log \beta_{ML(OH)_2}$	22.30	22.75	21.00	22.65	21.90	22.40
$\log K_{MLH}^N$	3.95	3.94	3.85	-	-	-
$\log K_{MLH}^{MLH}$	8.42	8.35	8.30	8.70	8.60	8.40
$\log K_{ML(OH)}^N$	10.60	10.70	10.50	11.70	11.45	11.10
$\log K_{ML(OH)_2}^N$	13.75	13.70	13.65	14.45	13.65	13.60

Table 4

Parameter	DMT(IV)-Succinic			DOT(IV)-Succinic		
	20°C	30°C	40°C	20°C	30°C	40°C
$\log\beta_{MLH}$	12.70	12.00	11.90	12.50	12.50	11.40
$\log\beta_{ML}$	8.50	8.40	7.20	8.10	7.98	7.80
$\log\beta_{ML(OH)}$	18.10	17.90	17.80	16.60	16.55	16.20
$\log\beta_{ML(OH)_2}$	22.00	20.70	20.60	21.00	20.89	18.10
$\log K_{MLH}^M$	5.30	5.02	5.00	4.75	4.70	4.68
$\log K_{ML}^{MLH}$	8.00	7.65	7.60	7.65	7.60	7.50
$\log K_{ML(OH)}^{ML}$	10.40	10.05	10.03	10.10	10.00	9.78
$\log K_{ML(OH)_2}^{ML(OH)}$	13.70	13.30	13.00	13.50	13.03	13.0040

Table 5

Parameter	DMT(IV)-Salicylic			DOT(IV)-Salicylic		
	20°C	30°C	40°C	20°C	30°C	40°C
$\log\beta_{MLH}$	15.50	15.40	15.10	16.35	16.18	15.50
$\log\beta_{ML}$	12.10	11.60	10.70	13.42	13.10	12.40
$\log\beta_{ML(OH)}$	28.15	27.30	26.75	27.40	27.20	27.10
$\log\beta_{ML(OH)_2}$	30.10	30.08	29.70	30.28	29.60	28.30
$\log K_{MLH}^M$	3.40	3.30	3.28	3.38	3.30	3.25
$\log K_{ML}^{MLH}$	12.98	12.50	12.30	12.50	12.25	12.15
$\log K_{ML(OH)}^{ML}$	15.30	15.05	14.60	15.15	15.05	14.70
$\log K_{ML(OH)_2}^{ML(OH)}$	17.60	17.30	16.60	17.90	17.80	17.50

Table 6

Parameter	DMT(IV)-5-Sulfosalicylic			DOT(IV)-5-Sulfosalicylic		
	20°C	30°C	40°C	20°C	30°C	40°C
$\log\beta_{MLH}$	14.95	14.70	13.80	15.90	14.70	14.30
$\log\beta_{ML}$	10.95	10.85	9.95	13.10	12.20	11.25
$\log\beta_{ML(OH)}$	24.70	24.50	24.50	26.82	25.35	25.90
$\log\beta_{ML(OH)_2}$	27.15	26.20	26.50	28.90	28.70	28.35
$\log K_{MLH}^M$	3.40	3.38	3.30	3.22	3.20	3.10
$\log K_{ML}^{MLH}$	11.20	11.18	11.17	11.62	11.61	11.60
$\log K_{ML(OH)}^{ML}$	14.00	13.50	13.40	14.80	14.52	14.50
$\log K_{ML(OH)_2}^{ML(OH)}$	16.80	16.15	16.00	18.00	17.90	17.40

Table 7

Parameter	DMT(IV)-3,5-Dinitrosalicylic			DOT(IV)-3,5-Dinitrosalicylic		
	20°C	30°C	40°C	20°C	30°C	40°C
$\log\beta_{MLH}$	14.50	12.30	10.45	15.00	13.00	12.91
$\log\beta_{ML}$	10.00	9.00	8.48	11.00	9.00	8.73
$\log\beta_{ML(OH)}$	20.22	20.20	20.17	22.10	21.10	21.09
$\log\beta_{ML(OH)_2}$	23.22	23.20	23.19	24.50	23.60	23.50
$\log K_{MLH}^M$	3.32	3.30	3.22	3.30	3.15	3.12
$\log K_{ML}^{MLH}$	9.02	9.00	8.89	8.80	8.75	8.72
$\log K_{ML(OH)}^{ML}$	11.50	11.30	11.29	13.30	12.70	12.60
$\log K_{ML(OH)_2}^{ML(OH)}$	14.60	14.45	14.38	15.60	14.80	14.70

- Thermodynamic formation constants were obtained by extrapolating the $\log\beta$ vs $\sqrt{\mu}$ plot to zero ionic strength.

Distribution Diagrams

Using the stability constants of the complexes and equilibrium constants of the ligands under the same experimental conditions, the percentage concentration of each complex species involving

metal ion and ligand as a function of pH has been calculated [30-32]. The graphical representation of speciation profile are given in the formation of speciation curves in Figure 3 to 14 are discussed here under:

1. Organotin (IV)-oxalic acid systems (Figure 3 and 4)

Curves 1 depicting the concentration of free metal ion shows a continuous decrease thereby indicating association with the ligand. The formation of ML species commences from pH \approx 2.8 (curve2) and continuously decreases upto pH 4.0. Above pH 4.0 the formation of monohydroxo species in negligible amount is evident (curve3). The predominant species is $ML(OH)_2$ as indicated by a high rise in percentage (\approx 70%) in curve 4.

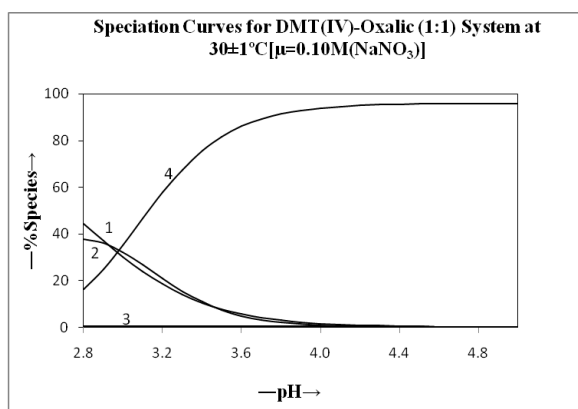


Figure 3

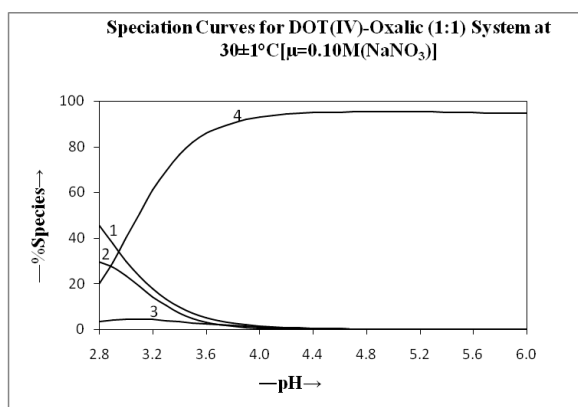


Figure 4

2. Organotin (IV)- Malonic acid system: (Figure 5 and 6)

It can be observed from the curves that MLH species is formed in low percent (\approx 20%) (curve2) in the initial pH range \approx 3.0. The formation of non protonated species (ML) with deprotonation of MLH reaches upto 60% (curve3). However in case of DOT(IV) malonic acid system MLH species is insignificant hence ignored. The percentage of dihydroxy species reached upto 95% approx. whereas monohydroxo species is insignificant.

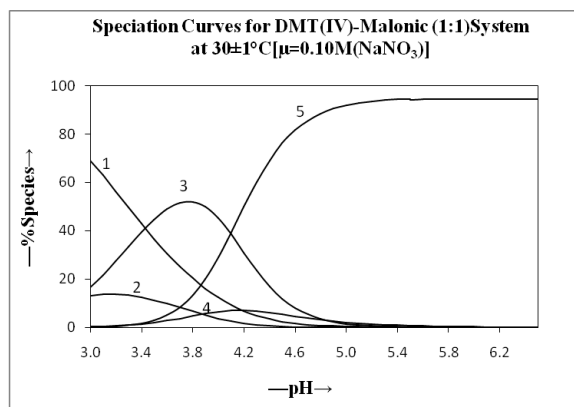


Figure 5

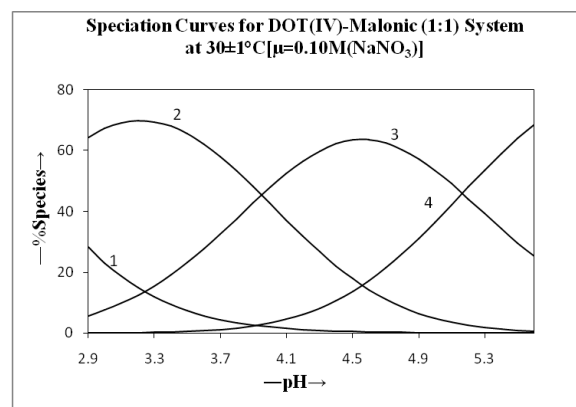


Figure 6

Speciation curves for DMT (IV)-Malonic (Figure 5), DOT(IV)- Malonic systems (Figure 6) (1:1) at 30°C [$\mu=0.10M(NaNO_3)$] : Curve (1) : [M], (2) [ML], (3) [ML(OH)], (4) [ML(OH)₂], in Figure. (5) Curve (2), [MLH]

3. Organotin (IV)- succinic systems: (Figure 7 and 8)

Distribution curves of DMT (IV) succinic acid and DOT (IV) succinic acid system show a very low percentage of free metal ion in the very initial stage. This is probably due to the fact that metal ion gets associated with monoprotonated ligand (HL) in the initial stage at low pH leading to the formation of protonated complex MLH as the predominant species (curve2).With the increase of pH,deprotonation of MLH species

leads to the formation of ML complex (curve 3). In the higher pH range formation of dihydroxo complex $ML(OH)_2$ is observed to be the major species (curve 5).

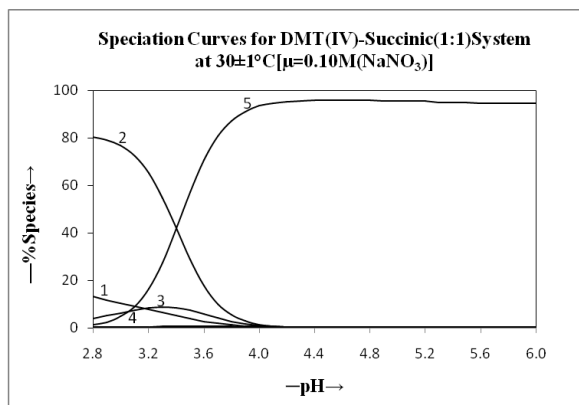


Figure 7

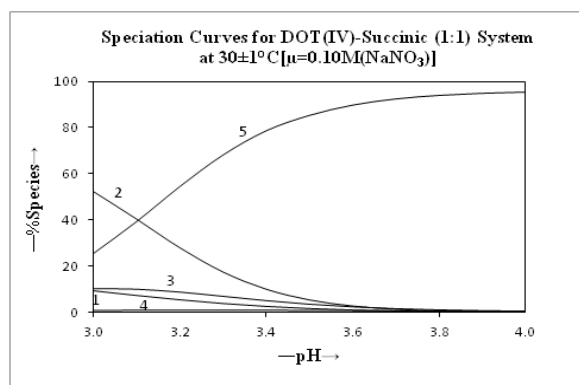


Figure 8

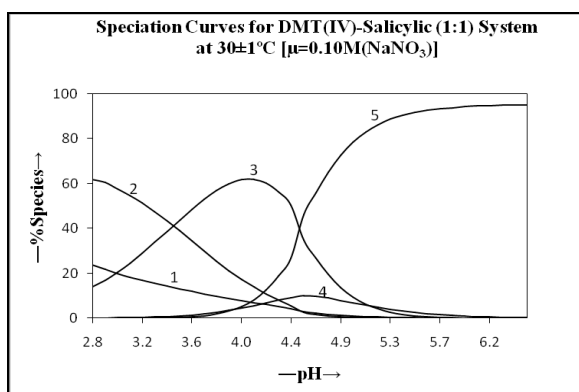


Figure 9

4. Organotin (IV) -salicylic acid system: (Figure 9 and 10)

Initial percentage of MLH species is significantly high nearly 60%, but decreases regularly with the increase of pH. Formation of ML species reaches its maximum (65% approx.) at pH 4.1. With the further increase of pH, formation of hydroxo complex is noticed it is seen that the former is insignificant but

the latter attain the value as high as 95%, thereby supporting the formation of $ML(OH)_2$ complex as the major species.

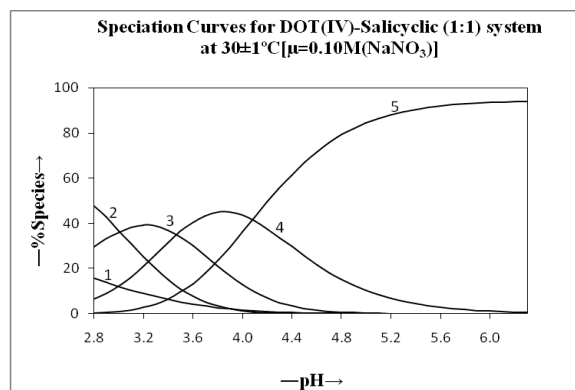


Figure 10

5. Organotin (IV) 5-sulfosalicylic acid system: (Figure 11 and 12)

The facts observed in the distribution curve of DMT (IV) 5-sulfosalicylic acid and DOT(IV) 5-sulfosalicylic acid follow the same trends as described for DMT (IV) salicylic acid and DOT(IV) salicylic system. However the percentage of ML, MLOH and $ML(OH)_2$ species are much higher and that of MLH is quite low. It can be attributed to the non-existence of the protonated form of the ligand in the pH range where the complexation occurs.

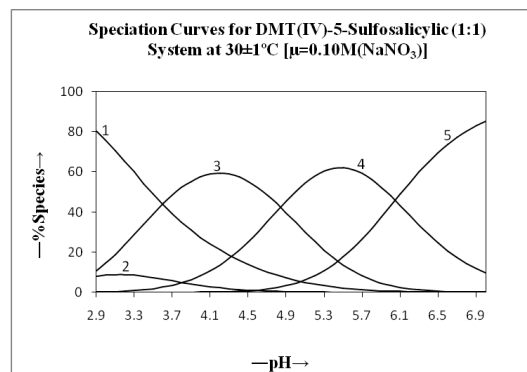


Figure 11

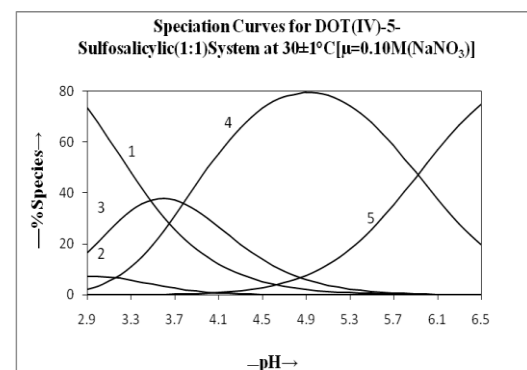


Figure 12

6. Organotin (IV) -3,5-dinitrosalicylic acid system: (Figure 13 and 14)

The trends are same as described for system with salicylic acid and 5-sulfosalicylic acid.

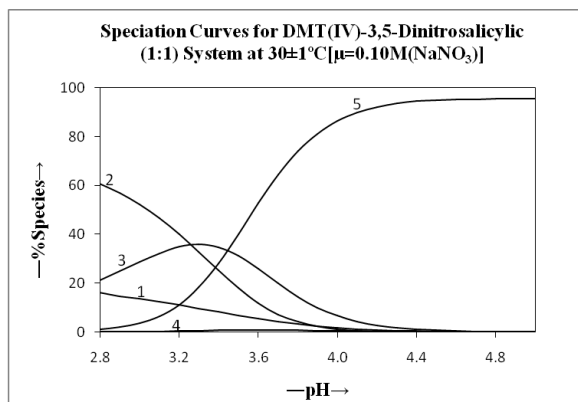


Figure 13

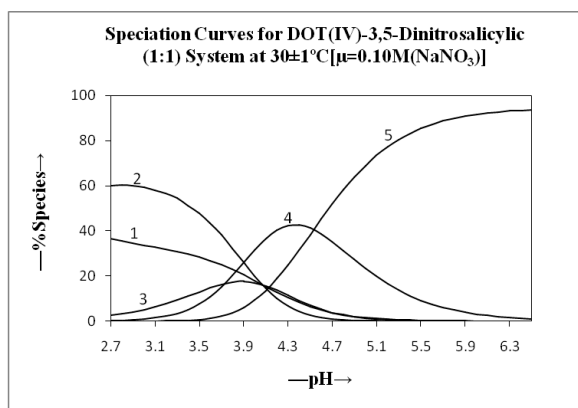


Figure 14

Speciation curves for DMT (IV)-Succinic (Figure 7), DOT(IV)- Succinic systems (Figure 8) DMT (IV)-Salicylic, (Figure 9) DOT(IV)- salicylic systems (Figure 10) DMT (IV)-5-Sulfosalicylic, (Figure 11) DOT(IV)- 5-Sulfosalicylic systems (Figure 12) DMT (IV)-3,5-Dinitrosalicylic, (Figure 13) DOT(IV)- 3,5-Dinitrosalicylic systems (Figure 14) (1:1) at 30°C [μ=0.10M(NaNO₃)] :
 Curve (1) : [M], (2) [MLH], (3) [ML], (4) [ML(OH)], (5) [ML(OH)₂]

Conclusion

On comparing the formation of various species in oxalic, malonic and succinic acid, it is understood that the formation of MLH species could not be ascertained with the former ligand whereas MLH species are predominant in latter two ligands. The absence of MLH species is due to the intramolecular hydrogen bonding in the monoanion. It become so obvious from this that the formation of ML species in case of oxalic acid systems by the association of organotin (IV) moiety with the dissociated form of the ligand whereas in case of

malonic and succinic acid formation of ML species occur by deprotonation of MLH species. The stability constant of ML species in reference to these ligand are depicted in the order oxalic > malonic > succinic. This can be attributed to the presence of additional -CH₂ group successively, thereby increasing the ring size of the metal-ligand complex in this order, and positive inductive effect of -CH₂ group. These two factors probably sets the observed trend.

On comparing the stabilities of salicylic, 5-sulfosalicylic and 3,5-dinitrosalicylic acid show that the complexes of salicylic acid are more stable as compared to that of 5-sulfosalicylic acid and 3,5-dinitrosalicylic acid. This can be attributed to the presence of electron withdrawing -SO₃H group in 5-sulfosalicylic acid and -NO₂ group in 3,5-dinitrosalicylic acid.

The comparison of the stabilities of the complex species formed with DMT (IV) and DOT (IV) indicates that the dioctyltin (IV) complexes have been found to be less stable than the dimethyltin (IV) complexes due to steric hindrance of bulky group^[33-34].

It is observed that the formation constant of the complexes increases with decrease in ionic strengths of the medium. The stability constants decrease with increase in temperature which suggests that low temperature is favourable for complexation.

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