Kinetic Studies of the Interaction Between Organotin(IV)chlorides and Tetraaza Schiff Bases: Synthesis and Characterization of Some Novel Tin(IV) Schiff Base Complexes

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ABSTRACT: In this article the kinetics of the interaction between the teteraaza Schiff bases as donor with organotin(IV)chlorides as acceptor was studied in acetonitrile. Teteraaza Schiff bases are (Me_4-Bzo_2[14]tetraeneN_4) (tmtaa), (Me_4-4-CH_3Bzo_2[14]tetraeneN_4) (Metmtaa), (Me_4-4-$ClBzo_2$[14]tetraeneN_4) (Cltmtaa), i.e., [(Me_4-Bzo_2[14]tetraeneN_4)] means that (5,7,12,14-tetramethyldibenzo[b,i][1,4,8,11] tetraazacyclotetradecine) (tmtaa) and organotin(IV)chlorides are methyltin(IV) trichloride, phenyltin(IV)trichloride, dimethyltin (IV)dichloride, diphenyltin(IV) dichloride, and dibutyltin(IV)dichloride. The kinetic parameters and the second-order k_2 rate constants show the donor properties of tetraaza Schiff bases as Me_4-4-CH_3Bzo_2[14]tetraeneN_4 > Me_4-Bzo_2[14]tetraeneN_4 > Me_4-4-$ClBzo_2$[14]tetraeneN_4 and also the acceptor properties of organotin(IV)chlorides as $PhSnCl_3 > MeSnCl_3 > Ph_2SnCl_2 > Me_2SnCl_2 > Bu_2SnCl_2$. An excellent linearity of k_{obs} vs. the molar concentration of the acceptor, the high span of k_2 values, the large negative values of $\Delta S^{\neq}$, and the low $\Delta H^{\neq}$ values suggest an associative (A) mechanism for the acceptor–donor interaction. © 2011 Wiley Periodicals, Inc. Int J Chem Kinet 43: 247–254, 2011

INTRODUCTION

Organotin(IV) complexes have been a subject of interest because of their biological and commercial

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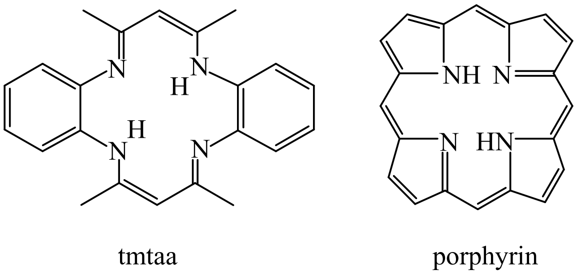


Figure 1 Comparing the structure of tmtaa and porphyrin.

applications. In recent years, there have been more and more reports on the synthesis, antitumor activities, and structural elucidation of various diorganotin Schiff bases. Increasing attention has also been devoted to the organotin(IV) complexes with the Schiff base ligand in view of their special antitumor activities [1–4].

The observation that transition metal Schiff base complexes can function as a neutral donor ligand has led to several investigations of their reactions with tin Lewis acids and to the isolation of the adducts [5–9].

Our group has studied the thermodynamics and the kinetics of adduct formation between organotin(IV)halide acceptors with donors, such as porphyrins and transition metal Schiff base complexes [10–19].

Our interest in the tetraaza macrocycle, tmtaa [(5,7,12,14-tetramethyldibenzo[*b*, *i*] [1,4,8,11]tetraazacyclotetradecine)], is derived from structural/chemical similarities with porphyrins (Fig. 1), in particular with the facile complexation of metal ions following deprotonation. The tmtaa Schiff base has a saddle structure in which the four nitrogen atoms lie in a plane, thereby creating a natural location (cavity size ca. 1.9 Å) for metal encapsulation [20].

In comparison with main group porphyrin and phthalocyanine complexes, very little is known about the other teteraaza macrocycles, such as tmtaa.

Molecular interactions of organotin(IV)halides with a variety of uni- and bidentate nitrogen donor ligands are studied [21]. Owing to the antitumor properties of organotin(IV)halides, their interactions with biological systems have been the subject of a number of investigations [22–25].

The present work deals with the kinetics and mechanism of the acceptor–donor interaction between $RSnCl_3$ and R_2SnCl_2 (R = Ph, Bu, Me) with tetraaza Schiff bases: (Me_4-Bzo_2[14]tetraeneN_4) (tmtaa), (Me_4-4-CH_3Bzo_2[14]tetraeneN_4) (Metmtaa), (Me_4-4-ClBzo_2[14]tetraeneN_4) (Cltmtaa) that [(Me_4-Bzo_2[14]tetraeneN_4)] is (5,7,12,14-tetramethyldibenzo[*b*, *i*][1,4,8,11]tetraazacyclotetradecine) (tmtaa) in acetonitrile as solvent. Some products of the kinetic process were synthesized and characterized by ^{1}H NMR, ^{13}C NMR, ^{119}Sn NMR, IR, UV–vis spectra, and the elemental microanalysis (C.H.N) method.

EXPERIMENTAL

Reagents

All chemicals were used as obtained from Merck (Germany), Fluka (Switzerland), or Acros (France). An analytical-grade solvent from Merck was used without further purification.

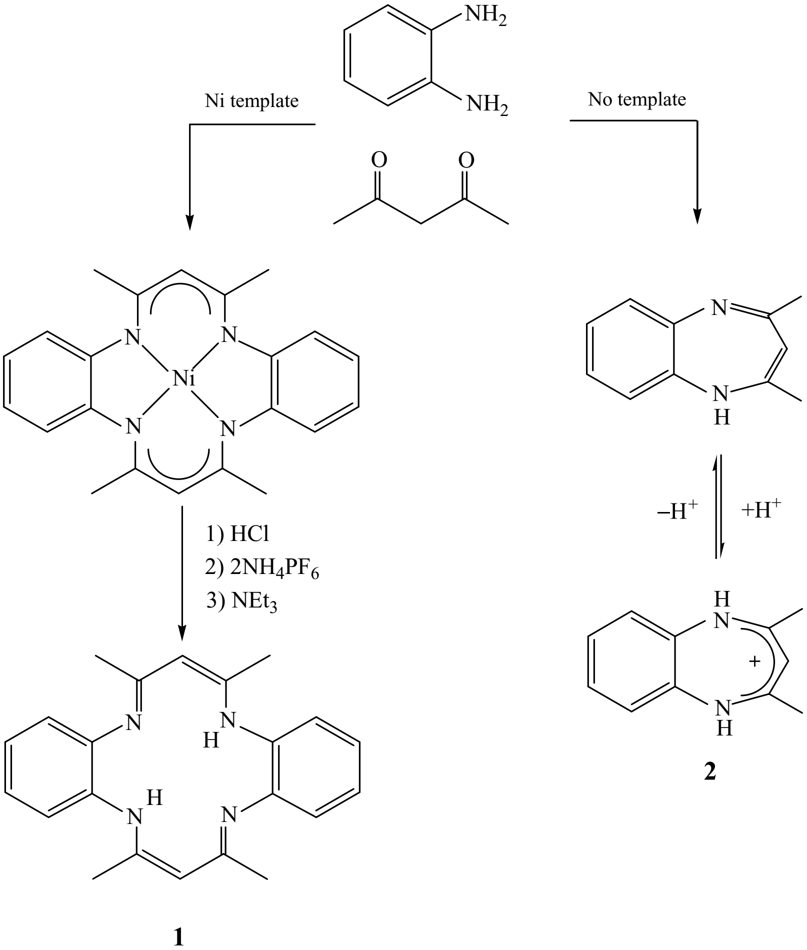
Instruments

Light-absorption measurements in the visible region were made with a Perkin-Elmer-UV–vis spectrophotometer-Lambda 2, equipped with a Lauda-ecoline-RE thermostat. FTIR spectra were run on a Shimadzu FTIR-8300 spectrophotometer. ^{1}H NMR, ^{13}C NMR spectra were recorded on a Bruker Avance DPX-250 spectrometer in DMSO-d_6 solvent using tetramethylsilane (TMS) as an internal standard. ^{119}Sn NMR spectra were recorded on a Bruker Avance DPX-400 spectrometer in the DMSO-d_6 solvent. Elemental microanalyses (C.H.N.) were obtained by using a thermo Finnigan-CHNSO analyzer.

Synthesis

The Schiff bases were prepared according to the method described in the literature [26] (Scheme 1), yielding 60%. The IR, ^{1}H NMR, ^{13}C NMR, and the UV–vis absorption spectra were in good agreement with the published data.

Synthesis of Diorganotin(IV)dichloride Complexes. R_2SnCl_2 (R = Ph, Me, Bu) (0.05 mmol) was dissolved in acetonitrile (5 mL) and added dropwise to tmtaa Schiff base (0.05 mmol) in acetonitrile (10 mL) at $pH > 7$ by adding some drops of NEt_3, and the mixture was stirred for 6 h at room temperature. During the time, yellow Schiff base changed to purple. The change in color was due to the interaction of R_2SnCl_2 with the Schiff base. Addition of *n*-hexane precipitated a purple powdery product. The progress of the reaction was monitored by UV–vis spectra. The tetraaza Schiff base completely changed to the tin(IV) complexes, and the mixture did not show any absorption bands related to the free Schiff base. The results of the elemental analyses for the diorganotin(IV)complexes formed from the tetraaza Schiff base and R_2SnCl_2 (R = Ph, Me, Bu), which were dried in the vacuum oven for 24 h at 75–80°C, were consistent with [R_2Sn(tmtaa)].



Scheme 1 General procedure for synthesis of tmtaa and its derivatives.

[Me2Sn(tmtaa)]. Yield 54%, mp 190–191°C. Anal. Found/Calcd. C, 58.44/58.69; H, 5.71/5.75; N, 11.56/11.41%. ^{1}H NMR (DMSO-d_6, 250 MHz) (ppm): 0.94 (6H, s, $J_{\text{Sn-H}} = 75$ Hz, Sn-CH$_3$), 1.70 (12H, s, CH$_3$), 4.12 (2H, s, CH), 6.52–6.85 (8H, m, aromatic). ^{13}C NMR (DMSO-d_6, 250 MHz) (ppm): 12.35 (Sn-CH$_3$), 38.44 (CH$_3$), 95.29 (CH), 123.38–133.96 (aromatic carbons), 175.61 (CN). ^{119}Sn NMR (DMSO-d_6, 400 MHz) (ppm): −254. IR (KBr, cm^{-1}): υ(C=N), 1631. UV–vis (acetonitrile) ($\lambda_{\max}$, nm): 500, 318.

[Bu$_2$Sn(tmtaa)]. Yield 42%, mp 197°C–199°C. Anal. Found/Calcd. C, 62.02/61.98; H, 7.68/7.97; N, 9.46/9.64%. ^{1}H NMR (DMSO-d_6, 250 MHz) (ppm): 0.91 (6H, t, $J = 7$ Hz, CH$_3$), 1.32–1.75 (12H, m, Sn-CH$_2$CH$_2$CH$_2$), 1.76 (12H, s, CH$_3$), 4.14 (2H, s, CH), 6.47–7.71 (8H, m, aromatic). ^{13}C NMR (DMSO-d_6, 250 MHz)(ppm): 15.42, 19.86, 28.76, 30.25 (Sn-Bu), 38.44 (CH$_3$), 93.21 (CH), 123.38–133.96 (aromatic carbons), 175.47 (CN). ^{119}Sn NMR (DMSO-d_6, 400 MHz) (ppm): −267. IR (KBr, cm^{-1}): υ(C=N), 1635. UV–vis(acetonitrile) ($\lambda_{\max}$, nm): 486, 300(sh).

[Ph$_2$Sn(tmtaa)]. Yield 42%, mp 198–199°C. Anal. Found/Calcd.: C, 66.84/66.37; H, 5.17/5.24; N, 9.37/9.10%. ^{1}H NMR (DMSO-d_6, 250 MHz) (ppm): 1.74 (12H, s, CH$_3$), 4.16 (2H, s, CH), 6.47–7.71 (8H, m, aromatic), 7.45–7.76 (10H, m, Sn-C$_6$H$_5$). ^{13}C NMR (DMSO-d_6, 250 MHz) (ppm): 38.44 (CH$_3$), 93.21 (CH), 123.38–139.76 (aromatic carbons), 175.47 (CN). ^{119}Sn NMR (DMSO-d_6, 400 MHz) (ppm): −246. IR (KBr, cm^{-1}): υ(C=N), 1635. UV–vis(acetonitrile) ($\lambda_{\max}$, nm): 498, 318.

Kinetic Studies

Kinetic data were determined spectrophotometrically. In all cases (runs from 10–40 ± 0.1°C), the procedure involves adding a sample of a given acceptor to a solution containing the tetraaza Schiff base ligand at pH > 7. Light-absorption measurements in the UV–vis region were made with a Perkin-Elmer-UV–vis spectrophotometer-Lambda 2, equipped with a Lauda-ecoline-RE thermostat. The kinetics was followed at a predetermined wavelength, where the difference in absorption between the substrate and the adduct was the largest. The kinetics was followed under pseudo-first-order conditions for the acceptor concentrations; the substrate concentration was kept constant at about 1.4×10^{-5} M, and the excess concentration of each acceptor varied in the range 10–600-fold in excess. The temperature was controlled in a thermostated cell compartment at 10–40 ± 0.1°C, and 10-mm quartz cells were used. The pseudo-first-order constants were calculated by fitting the data to $\ln[(A_t - A_\infty)/(A_0 - A_\infty)] = -k_{\mathrm{obs}}t$ (where A_t = absorbance at time t, A_0 = absorbance at $t = 0$, and A_∞ = absorbance at $t = \infty$) by means of a linear least-squares computer program. A_t was determined after each 10–60 s depending on the system. The second-order rate constants k_2 were obtained from the slope of the linear plots of k_{obs} versus [A] (acceptor concentration). The activation enthalpy and the activation entropy parameters, $\Delta H^{\neq}$ and $\Delta S^{\neq}$ (see Table III later in the paper) were obtained by fitting the data to linear least-squares computer programs of the Eyring equation (1):

$$\ln(k_2/T) = -\Delta H^{\#}/\mathrm{RT} + \Delta S^{\#}/R + 23.8 \qquad (1)$$

RESULTS AND DISCUSSION

NMR Spectra

The general approach involves deprotonation of H_2tmtaa and the subsequent reaction of the tmtaa^{2-} dianion with R_2SnCl_2.

$$\mathrm{H_2tmtaa} \xrightarrow{\mathrm{PH}\geq 7} \mathrm{tmtaa}^{2-} \xrightarrow{\mathrm{R_2SnCl_2}} \mathrm{R_2Sn(tmtaa)} \qquad (2)$$

where R = Me, Bu, Ph.

The compounds with microanalytical and spectroscopic data are listed in the Experimental section. The spectroscopic data are consistent with a proposed structure (solution) in which the SnR_2 unit is bonded to the four nitrogen atoms, resulting in a six-coordinate geometry around the metal center. Such an arrangement, depicted in Fig. 2 with the ligand in its saddle conformation and the two substituent groups(R) mutually cis, has been reported.

In the ^{1}H NMR spectra of the diorganotin(IV)complexes, both the aromatic ring and the CH, CH_3 protons of the ligand appear as a sharp singlet.

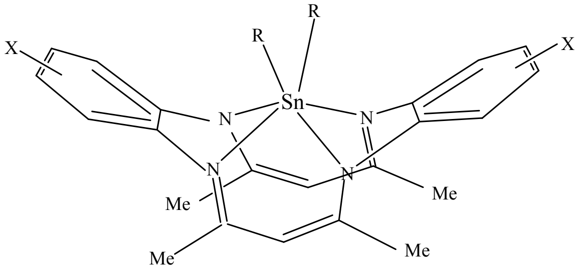


Figure 2 Proposed structure of [R_2Sn(X-tmtaa)] X = H, Cl, Me.

In ^{1}H NMR spectra of [Me_2Sn(tmtaa)], protons of two methyl groups attached to the Sn also appear as one sharp singlet with a high field shift, with respect to the corresponding parent Me_2SnCl_2 signal. The ^{13}C NMR spectra chemical shift assignments for carbon atoms in different environment are summarized in the Experimental section; again a single high field resonance in the case of [Me_2Sn(tmtaa)] confirms the equivalence of the methyl groups attached to the metal.

Holeĉek and coworkers' results show that in the di- and triorganotin complexes, the ^{119}Sn NMR spectra can be used as an indicator of the coordination number of the tin atom. In the range of +200 to −60, −90 to −4190, −210 to −400, and −440 to −540 ppm, the coordination numbers of the tin are four, five, six, and seven, respectively [27–30]. The complexes in the present investigation exhibit the ^{119}Sn spectra in the range −240 to −270 ppm, suggesting that the tin atoms are six coordinate.

ELEMENTAL ANALYSIS

Elemental analyses of these separately synthesized products are in good agreement with the proposed adducts formed at the end of the kinetic runs.

Kinetic Study

Tables I–III show the rate constants and the activation parameters for the interaction of acceptors with the tetraaza Schiff base in acetonitrile as a solvent at various temperatures. Although most of the kinetic studies that have been published were carried out in interfering (i.e., coordinating) solvents, it is necessary to investigate the nature of this interference before the mechanistic assignment is secured [31]. Plots of k_{obs} versus [A] exhibit an almost zero intercept (Fig. 3). This indicates that solvation of the Schiff base in acetonitrile is negligible. The rate law of Eq. (3) is compatible with

Table I k_2 ($M^{-1}s^{-1}$) Rate Constants for the Reaction of Organotinchlorides with tmtaa, [tmtaa] = 1.4×10^{-5} M, in CH_3CN at Different Temperatures

	Temperature (°C)			
Organotin Chloride	10	20	30	40
$PhSnCl_3$	100.9 (6.8)	110.4 (9.7)	120.8 (6.9)	129.4 (6.3)
$MeSnCl_3$	69.8 (3.6)	79.1 (3.9)	90.0 (4.0)	105.9 (3.6)
Ph_2SnCl_2	21.7 (0.7)	27.4 (2.0)	31.8 (1.5)	36.1 (2.1)
Me_2SnCl_2	9.3 (0.4)	11.3 (0.5)	14.1 (0.5)	17.1 (0.7)
Bu_2SnCl_2	2.3 (0.1)	2.5 (0.1)	2.8 (0.1)	3.2 (0.1)

Numbers in parentheses are the standard deviations of k_2.

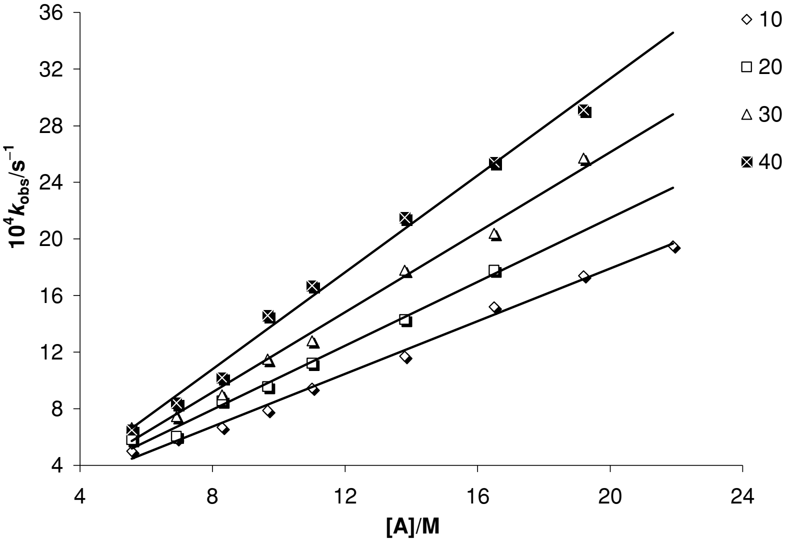


Figure 3 Plots of k_{obs} vs. Bu_2SnCl_2 molar concentrations [A] at different temperatures.

the adduct formation according to Eq. (4).

$$k_{obs} = k_2[A] \tag{3}$$

$$X - \text{tmtaa} + R_2SnCl_2/(RSnCl_3) \rightarrow [R_2Sn/(RSnCl).X - \text{tmtaa}] \tag{4}$$

where X = H, Cl, Me and R = Me, Bu, Ph.

Table II k_2 ($M^{-1}s^{-1}$) Rate Constants for the Reaction of Ph_2SnCl_2 with Tetraaza ligands [ligand] = 1.4×10^{-5}M, in CH_3CN at Different Temperatures

	Temperature (°C)			
	10	20	30	40
Metmtaa	27.6 (0.9)	34.0 (1.5)	41.1 (1.6)	50.9 (2.5)
tmtaa	21.7 (0.7)	27.4 (2.0)	31.8 (1.5)	36.1 (2.1)
Cltmtaa	16.4 (0.6)	22.6 (1.0)	28.9 (1.2)	32.7 (1.3)

Numbers in parentheses are the standard deviations of k_2.

The k_2 values are obtained from the slope of the linear plots of k_{obs} versus the acceptor concentration [A] (Fig. 3).

The isosbestic points in the absorption spectra (Fig. 4) suggest that only one reaction is in progress (Eq. (4)); it indicates that different products was not produced. Also the electronic spectra of the adducts formed in the kinetic study were the same as the electronic spectra of the separately synthesized diorganotin(IV) complexes (Fig. 5).

The activation parameters $\Delta H^{\#}$ and $\Delta S^{\#}$ were obtained from the standard linear Eyring plots of $\ln(k_2/T)$ versus $1/T$ with a good correlation of 0.97–0.99 (Fig. 6).

The results show that there is a linear rate dependence on the concentration of the acceptor. Typical plots of k_{obs} versus Bu_2SnCl_2 molar concentrations of the tmtaa ligand at different temperatures are shown in Fig. 3.

Table III Activation Parameters $\Delta H^{\#}$, $\Delta S^{\#}$, and $\Delta G^{\#}$ for the Reaction of Organotinchlorides with Schiff Bases in CH_3CN

Adduct	$\Delta H^{\#}$(kJ mol^{-1})	$\Delta S^{\#}$(J K^{-1} mol^{-1})	$\Delta G^{\#}$(kJ mol^{-}1)^{a}
$PhSnCl_3$. tmtaa	3.7 (0.2)	−193.3 (0.6)	64.2 (0.3)
$MeSnCl_3$. tmtaa	7.7 (0.7)	−182.5 (2.2)	64.8 (0.9)
Ph_2SnCl_2. Metmtaa	12.4 (0.4)	−173.4 (1.4)	66.7 (0.6)
Ph_2SnCl_2. tmtaa	9.9 (1.0)	−184.1 (3.5)	67.5 (1.5)
Ph_2SnCl_2.Cltmtaa	14.4 (1.6)	−170.3 (5.4)	67.7 (2.4)
Me_2SnCl_2.tmtaa	12.7 (0.4)	−181.6 (1.4)	69.5 (0.6)
Bu_2SnCl_2.tmtaa	3.7 (0.2)	−225.0 (0.8)	74.1 (0.3)

Numbers in parentheses are the standard deviations.

[a] Calculated from $\Delta G^{\#} = \Delta H^{\#} - T\Delta S^{\#}$ at $T = 313$ K.

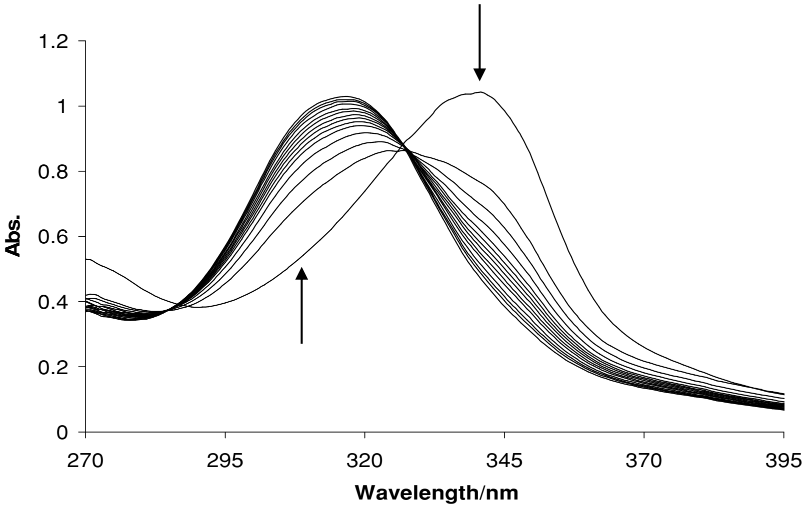


Figure 4 Absorption spectra of tmtaa with Bu_2SnCl_2 at 30°C in CH_3CN with isosbestic points. Time interval = 30 s.

Donor Properties of Ligands

The existence of methyl (—CH_3) groups as electron-releasing groups makes the (Me_4-4-CH_3Bzo_2[14] tetraeneN_4) ligand a better donor compared with (Me_4-Bzo_2[14]tetraeneN_4), and also electron-withdrawing groups, such as chloride (—Cl), reduce the donor property of (Me_4-4-ClBzo_2[14]tetraeneN_4); thus the formation constants decrease according to the following sequence: Metmtaa > tmtaa > Cltmtaa.

Acceptor Properties of Organotin Chlorides

Comparing the k_2 values, the following trend of acidity for the acceptors' use was assessed: $PhSnCl_3$ > $MeSnCl_3$ > Ph_2SnCl_2 > Me_2SnCl_2 > Bu_2SnCl_2.

It is clear that acceptors with lower steric hindrance were better Lewis acids, such as $PhSnCl_3$ and $MeSnCl_3$. The electron-withdrawing group (Ph—) on the tin center makes $PhSnCl_3$ a stronger acceptor than $MeSnCl_3$. This trend is also observed for Ph_2SnCl_2, Me_2SnCl_2, and Bu_2SnCl_2 and indicates that replacing the methyl group by a more bulky butyl group on an organotin(IV) compound weakens interactions. The butyl group can affect the interaction in two ways: (1) a more bulky butyl group makes adduct formation unfavorable because of its greater steric hindrance than a methyl group [31], and (2) butyl groups, though, have better electron-releasing properties to reduce the acceptor strength of a diorganotin(IV) Lewis acid and to decrease its interaction with Schiff bases.

Elucidation of the Mechanism

Reaction rates almost always increase with temperature. The expression of the temperature dependence is always given for the rate constant, rather than the rate.

The activation parameters bring out several features: The quite small enthalpy represents very rapid reaction. The values of $\Delta H^{\#}$ most simply relate to the differences in bond enthalpies between the transition state

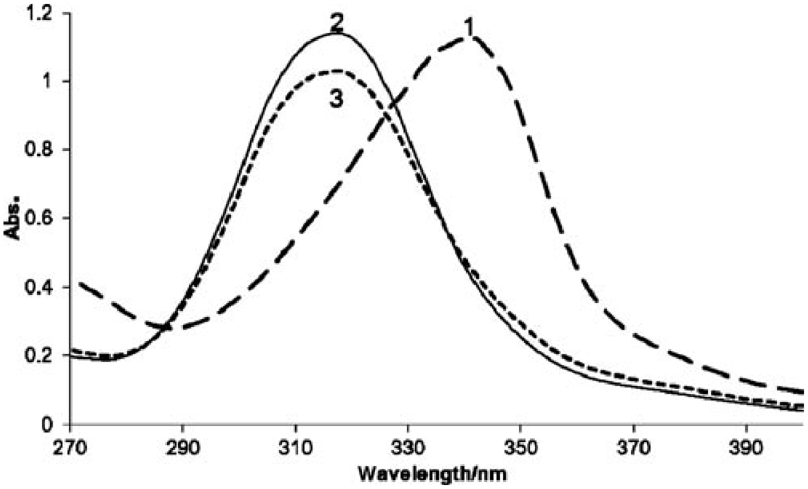


Figure 5 Electronic spectra of tmtaa ligand (1.4×10^{-5} M) (1), the end point of titration of ligand with Me_2SnCl_2 (2), and separately synthesized $[Me_2Sn(tmtaa)]$ adduct (7.6×10^{-4} M) (3) in acetonitrile.

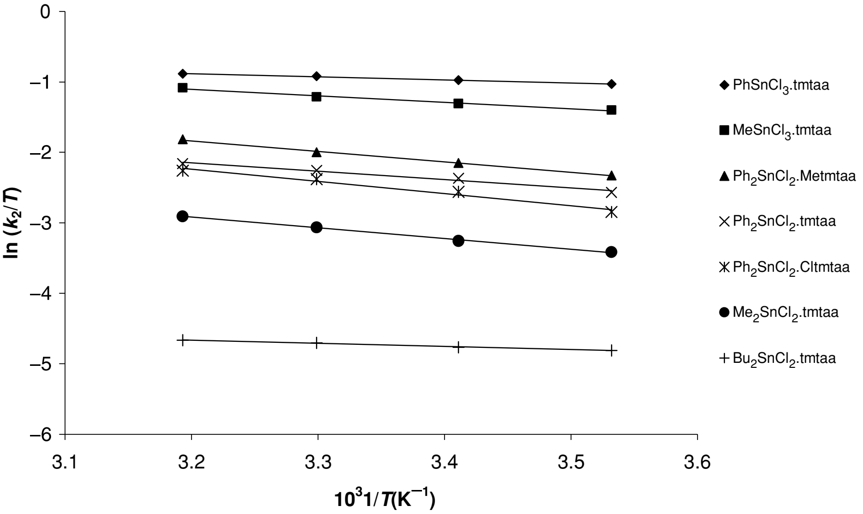


Figure 6 Eyring plots for adduct formation of Schiff bases with different acceptors in acetonitrile.

and the reactants. In solution reactions, it is tempting to assign the value of $\Delta H^{\#}$ for a unimolecular reaction to the bond dissociation enthalpy of the bond being cleaved in the activated process. The relationship may be true in certain cases, but differences in the solvation energies of the reactants and transition state cannot necessarily be ignored, particularly for species bearing ionic charges.

The activation entropy associated with k_2 is particularly large, and negative, thus $\Delta S^{\#}$ reflects the loss of entropy from the union of the two reaction partners into a single transition state.

The k_2 values for the ligands entry show high span (more than 10-fold); therefore, they suggest the dependence on the nature of the acceptor. Also the linear plots of k_{obs} versus [A], the large negative values of $\Delta S^{\#}$, and the low $\Delta H^{\#}$ values suggest an associative (A) mechanism.

CONCLUSION

The results of this work can be summarized as follows:

1. We measured the relative donor properties (Lewis basicity) of different Schiff bases toward organotin(IV)chlorides. In a kinetic study, this trend of basicity was obtained due to electronic effects: Me_4-4-CH_3 Bzo_2[14]tetraeneN$_4$ > Me_4-Bzo_2[14]tetraeneN$_4$ > Me_4-4-ClBzo_2[14]tetraeneN$_4$.

2. The formation constants for a given donor (Schiff base) change according to the following trend for organotin(IV)compounds due to the electronic and steric effects: $PhSnCl_3 > MeSnCl_3 > Ph_2SnCl_2 > Me_2SnCl_2 > Bu_2SnCl_2$.
3. The kinetics and mechanism of adduct formation were studied. The high span of k_2 values, the linear plots of k_{obs} versus [A], the large negative values of $\Delta S^{\#}$, and the low values of $\Delta H^{\#}$ suggest an associative (A) mechanism.

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