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Thermochemistry of adducts of tin(IV) bromide with heterocyclic bases

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Abstract

The compounds $[SnBr_4(L)_n]$ (where L is pyridine (py), 3-methylpyridine (β-pico), 4-methylpyridine (γ-pico), piperidine (pipd), morpholine (morph), piperazine (pipz), 3-cyanopyridine (3-cyanopy), 4-cyanopyridine (4-cyanopy), quinoline (quin) or 2,2′-bipyridine (bipy) and $n = 1$ or 2) were synthesized and characterized by elemental analysis, melting points, thermal studies and IR spectroscopy. The enthalpies of dissolution of adducts, tin(IV) bromide and ligands in 25% (v/v) aqueous 1.2 M HCl in methanol were measured and, by thermochemical cycles, the standard enthalpies for the Lewis acid/base reaction ($\Delta_r H^\theta$), the standard enthalpies of formation ($\Delta_f H^\theta$), the standard enthalpies of decomposition ($\Delta_D H^\theta$), the lattice standard enthalpies ($\Delta_M H^\theta$) and the standard enthalpies for the Lewis acid/base reaction in the gaseous phase ($\Delta_r H^\theta(g)$) are determined. The mean standard enthalpies of the tin-nitrogen coordinated bonds $\bar{D}_{(Sn–N)}$ are estimated.

Keywords: Sn–N bonds; Tin(IV) bromide; Thermochemical parameters; Heterocyclic bases; Thermochemistry

1. Introduction

In a recent article [1] we described the preparation, characterization and thermochemistry of adducts of tin(IV) bromide with substituted acetamide and thioacetamide. The mean enthalpies of the Sn–O and Sn–S coordinated bonds were obtained. Adducts of tin(IV) bromide with ligands containing nitrogen as the donor atom are also reported in the literature [2–5] but there is no information on the energies of the Sn–N coordinated bonds. This paper describes the synthesis of adducts of tin(IV) bromide with heterocyclic bases with the purpose of obtaining the enthalpies involved in the formation of the adducts. Correlations between the coordinated bonds dissociation enthalpies and other thermochemical parameters are also established. Inductive effects on the energy of the Sn–N bonds due to the substitution of hydrogen atoms in the ligands by the electron donor methyl group or by the electron withdrawing cyano group, as well as the effect of substitution of carbon atoms by more electronegative nitrogen or oxygen atoms were also studied. Correlations between the bond energies and the basicity of the ligands are established.

2. Experimental

All the preparations and manipulations were done under a dry nitrogen atmosphere to prevent hydrolysis of the compounds as well as to avoid contact with the toxic compounds.

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2.1. Chemicals

Tin(IV) bromide (99%, Aldrich) was purified by distillation through an efficient column (mp = 30 °C). Pyridine (ACS Reagen), 3-methylpyridine (p.a. Baker), 4-methylpyridine (p.a. Baker), piperidine (99% RPE Analyticals, Carlo Erba), morpholine (99%, ACS Aldrich) and quinoline (98%, Aldrich) were all purified by distillation using an efficient column and stored over 4 Å molecular sieves (bp obtained: 111–112, 139–140, 140–141, 103–104, 123–124 and 111–112/2–20 mmHg, respectively). Piperazine (99%, Aldrich), 3-cyano-pyridine (98% Aldrich) and 4-cyanopyridine (98% Aldrich) were purified by recrystallization from methanol (mp = 107–108, 48–50 and 77–78 °C, respectively). The 2,2′-bipyridine (99%, Aldrich) was purified by recrystallization from ethanol according to the method described by Gallagher et al. [6] (mp = 193–194 °C). Solvents used in the synthesis and calorimetric measurements were purified by distillation and stored over Linde 4 Å molecular sieves.

2.2. Analytical

Carbon, hydrogen and nitrogen contents were determined by microanalytical procedures using a Perkin-Elmer elemental analyzer. Tin was determined by gravimetry as stannic oxide following precipitation of hydrated stannic oxide by ammonia solution from the nitric acid solution of the adducts. The precipitate was ignited to stannic oxide. The filtrate was used for the determination of bromide as silver bromide, using 0.1 M $AgNO_3$ solution.

2.3. Synthesis of the adducts

The adducts were obtained from the reaction of tin(IV) bromide and ligands in solution. A typical procedure is given as follows.

2.3.1. $SnBr_4$-β-pico

On mixing a solution of 1.0 g of $SnBr_4$ (2.3 mmol) in 10 ml of ethanol with 0.45 ml (4.6 mmol) of β-pico, under a stream of dry nitrogen, a white solid appeared. The stirring was continued for several hours. The solid was filtered, washed with three portions of 10 ml of petroleum ether and dried in vacuum and stored in a desiccator over calcium chloride. All the adducts were prepared with a molar ratio tin(IV)/ligand of 1:2. In the cases of the adducts of py and morph carbon tetrachloride was used as a solvent instead of ethanol.

2.4. Infrared spectra

Spectra were obtained with samples in KBr matrix for adducts and solid ligands. For liquid ligands, a film of the ligand sandwiched between NaCl plates was used. A Perkin-Elmer 1600 series FTIR spectrophotometer in the 4000–400 cm^{-1} region was used.

2.5. Thermal studies

TG/DTG and DSC measurements were obtained in argon atmosphere with a Du Pont 951 TG analyzer.

2.6. Calorimetric measurements

All the solution calorimetric determinations were carried out in an LKB 8700-1 precision calorimeter as described elsewhere [7]. The solution calorimetric measurements were performed by dissolving samples from 0.8 to 252.0 mg of the adducts or tin(IV) bromide in 100 ml of 25% (v/v) aqueous 1.2 M HCl in methanol and the ligand in the solution of tin(IV) bromide, maintaining a molar relation equal to the stoichiometry of the respective adduct. The accuracy of the calorimeter was verified by determining the heat of dissolution of tris[(hydroxymethyl)amino]methane in 0.1 mol dm^{-3} HCl. The result (-29.78 ± 0.03 kJ mol^{-1}) is in agreement with the value recommended by IUPAC (-29.763 ± 0.003 kJ mol^{-1}) [8].

3. Results and discussion

All the adducts obtained were solids. The yields ranged from 32 to 94%. The yields, melting points, colors, appearance and analytical data are summarized in Table 1.

3.1. Infrared spectra

The more important IR bands are reported in Table 2. Considerable shifts to lower frequencies with respect to free ligands of the bands attributed to $\nu_{(N\text{–}H)}$ in the pipd, morph and pipz adducts are

Table 1
Yields on preparation, melting points, appearance and analytical data of the adducts

Compound	Yield (%)	Melting point[a]	Appearance	C		H		N		Sn		Br	
				Calculated	Found	Calculated	Found	Calculated	Found	Calculated	Found	Calculated	Found
$[SnBr_4(py)_2]$	67	292–293	White powder	20.14	20.51	1.69	1.75	4.70	4.50	19.90	19.67	53.58	53.48
$[SnBr_4(\beta\text{-pico})_2]$	74	293–294	White powder	23.08	23.33	2.24	2.26	4.49	4.43	19.00	19.35	51.18	51.15
$[SnBr_4(\gamma\text{-pico})_2]$	56	271–272	White powder	23.08	22.97	2.24	2.17	4.49	4.33	19.00	19.11	51.18	51.21
$[SnBr_4(pipd)_2]$	79	195–196	Yellow powder	19.73	19.95	3.64	3.84	4.60	4.79	19.50	19.64	52.52	52.63
$[SnBr_4(morph)_2]$	86	197–198	Yellow powder	15.69	15.62	2.96	2.70	4.57	4.43	19.38	19.42	52.18	52.24
$[SnBr_4(pipz)_2]$	94	288–289	White powder	15.74	15.51	3.30	3.52	9.18	8.99	19.44	19.72	52.35	52.57
$[SnBr_4(3\text{-cyanopy})_2]$	32	216–217	White powder	22.29	22.03	1.25	1.36	8.67	8.36	18.36	18.50	49.44	49.46
$[SnBr_4(4\text{-cyanopy})_2]$	65	266–267	Light yellow powder	22.29	21.99	1.25	1.38	8.67	8.34	18.36	18.40	49.44	49.50
$[SnBr_4(quin)]$	73	250–251	Yellow powder	19.05	19.08	1.24	1.27	2.47	2.69	20.91	20.88	56.32	56.28
$[SnBr_4(bipy)]$	69	330–331	White powder	20.20	19.94	1.36	1.56	4.71	4.80	19.96	19.96	53.76	53.73

[a] With decomposition.

Table 2
Main IR absorption bands (cm^{-1})[a] of complexes and free ligands

Compound	$\nu_{(N–H)}$	$\nu_{(C–C)}$	$\nu_{(C–N)}$	Ring	$\nu_{(C–O–C)}$	$\delta_{(H–N–C)}$	$\alpha_{(C–C–C)}$	$\phi_{(C–C)}$	New bands
py		1573sh					584s	431m	
$[SnBr_4(py)_2]$		1605s					558w	430m	1247w
β-pico		1572sh, 1558s		1206m			652sh	n.o.	
$[SnBr_4(\beta\text{-pico})_2]$		1611m, 1577m		1187s			655s	414m	1254m
γ-pico		1585s, 1545s		1206m			n.o.	439w	
$[SnBr_4(\gamma\text{-pico})_2]$		1635m, 1602s		1189s			548m	420m	1289w
pipd	3276m	1467s				860s, 825m			
$[SnBr_4(pipd)_2]$	3180m	1456s				860m			
morph	3320m	1452s			1097s	889m, 835s			
$[SnBr_4(morph)_2]$	3112m	1460s			1116s	904m, 871vs			
pipz	3275s	1461s				861m, 815s			
$[SnBr_4(pipz)_2]$	3245s	1439s				866s			
3-cyanopy			2231s	1219s					
$[SnBr_4(3\text{-cyanopy})_2]$			2243m	1189s					
4-cyanopy			2236s	1216s					
$[SnBr_4(4\text{-cyanopy})_2]$			2243w	1218m					
quin		1596s		1031m				n.o.	
$[SnBr_4(quin)]$		1595s		966m				1224s	
bipy		1579s		994m				759vs	
$[SnBr_4(bipy)]$		1597s		1027m				769vs	1317m, 722m

[a] Key: α, ring deformation in plane; ϕ, ring deformation out of plane; ν, stretching; δ, angular deformation; Ring, ring breathing; n.o., not observed. Intensity of bands: vs, very strong; m, medium; w, weak; sh, shoulder.

observed, indicating coordination through the nitrogen atom [9,10]. In the morpholine adduct, a positive shift is observed, with respect to the free ligand, of the band attributed to the stretching C–O–C. This excludes the possibility of oxygen-to-tin(IV) coordination [9]. The change observed in the H–N–C deformation region (889–815 cm^{-1}) also affords evidence for the coordination of the ligands through their nitrogen atoms [11]. The adduct of pyridine is distinguished from the free ligand due to the presence, in the adduct, of a weak band at 1247 cm^{-1} and also by the shift of bands at 584 and 431 cm^{-1} in the free ligand, to higher frequencies in the adduct [12]. The infrared spectra of the adducts of β-pico and γ-pico show appreciable shifts to higher frequencies of the bands at 1585–1545 and 1206 cm^{-1} in the free ligands [13,14]. For the adducts of 3-cyanopy and 4-cyanopy this last band is observed at 1189 and 1218 cm^{-1}, respectively (1219 and 1216 in free ligands). The band attributed to the stretching of the nitrile group in the free ligands (2231 and 2236 cm^{-1}, respectively) increases very little in frequency and dramatically decreases in intensity after coordination. These exclude the coordination of 3-cyanopy or 4-cyanopy through the nitrogen atom of the nitrile group [15]. The infrared spectra of these last adducts can be interpreted in terms of coordination of the ligands through the heterocyclic nitrogen atom to the tin(IV) atom [12–15]. The infrared spectra of the bipy adduct shows the appearance of new bands, with relation to the free ligand, after coordination. Two new bands appear at 1317 and 722 cm^{-1} due to the adduct formation [16]. The infrared spectra of the quin adduct shows shifts of several bands after coordination, with respect to the free ligand [17]. The infrared data on the bipy and quin adducts can be interpreted in terms of the coordination of these ligands through the nitrogen atoms to the tin atom [16,17].

3.2. Thermal studies

Thermogravimetry, derivative thermogravimetry and DSC curves of the adducts were done in an attempt to obtain heats of melting, vaporization and sublimation. However, all of the adducts decomposed on heating, so this was not possible.

3.3. Calorimetric measurements

The standard enthalpies of dissolution of tin(IV) bromide, ligands and adducts were obtained as

Table 3
Enthalpies of dissolution at 298.15 K

Compound	Calorimetric solvent	Number of experiments	i	$\Delta_i H^\theta$ (kJ mol^{-1})
$SnBr_{4(s)}$	A	29	1	−90.48 ± 1.21
py$_{(l)}$	2:1 $SnBr_4$-A	4	2	−41.00 ± 0.81
$[SnBr_4(py)_2]_{(s)}$	A	3	3	42.72 ± 1.19
β-pico$_{(l)}$	2:1 $SnBr_4$-A	5	2	−48.93 ± 0.89
$[SnBr_4$(β-pico)$_2]_{(s)}$	A	3	3	65.89 ± 4.30
γ-pico$_{(l)}$	2:1 $SnBr_4$-A	5	2	−41.26 ± 0.31
$[SnBr_4$(γ-pico)$_2]_{(s)}$	A	5	3	36.85 ± 0.78
pipd$_{(l)}$	2:1 $SnBr_4$-A	6	2	−130.73 ± 2.31
$[SnBr_4(pipd)_2]_{(s)}$	A	3	3	70.79 ± 5.21
morph$_{(l)}$	2:1 $SnBr_4$-A	5	2	−104.63 ± 1.96
$[SnBr_4(morph)_2]_{(s)}$	A	5	3	27.91 ± 1.08
quin$_{(l)}$	1:1 $SnBr_4$-A	4	2	−15.99 ± 0.32
$[SnBr_4(quin)]_{(s)}$	A	4	3	49.70 ± 2.23
pipz$_{(s)}$	2:1 $SnBr_4$-A	4	2	−110.20 ± 3.96
$[SnBr_4(pipz)_2]_{(s)}$	A	5	3	35.55 ± 1.16
3-cyanopy$_{(s)}$	2:1 $SnBr_4$-A	4	2	35.56 ± 2.98
$[SnBr_4$(3-cyanopy)$_2]_{(s)}$	A	5	3	60.13 ± 1.46
4-cyanopy$_{(s)}$	2:1 $SnBr_4$-A	5	2	35.98 ± 0.69
$[SnBr_4$(4-cyanopy)$_2]_{(s)}$	A	4	3	21.91 ± 1.36
bipy$_{(s)}$	1:1 $SnBr_4$-A	4	2	8.30 ± 0.44
$[SnBr_4(bipy)]_{(s)}$	A	3	3	16.74 ± 0.25

Key: A, 25% (v/v) 1.2 M HCl in methanol.

previously reported [7]. Table 3 gives the values obtained for the enthalpies of dissolution of $SnBr_4$ ($\Delta_1 H^\theta$), ligand into the solution of $SnBr_4$ ($\Delta_2 H^\theta$) and of the adduct ($\Delta_3 H^\theta$). Uncertainty intervals given in this table are twice the standard deviation of the means of three to six replicate measurements on each compound. Combined errors were calculated from the square root of the sum of the squares of the component errors.

From the values obtained for the standard enthalpies of dissolution and by appropriate thermochemical cycles [7,18], the following thermochemical parameters were determined: the standard enthalpies of the acid/base reactions ($\Delta_r H^\theta$), the standard enthalpies of formation ($\Delta_f H^\theta$), the standard enthalpies of decomposition ($\Delta_D H^\theta$), the standard lattice enthalpies ($\Delta_M H^\theta$) and the enthalpies of the Lewis acid/base reactions in the gaseous phase ($\Delta_r H^\theta(g)$). The $\Delta_r H^\theta(g)$ values can be used to calculate the standard enthalpies of dissociation of the Sn–N bonds [18], being equal to $\bar{D}_{(Sn–N)} = \Delta_r H^\theta(g)/n$ (where $n = 2$). Table 4 lists the values obtained for all these thermochemical parameters for the adducts. For the determination of $\Delta_r H^\theta(g)$ it was necessary to assume that the molar standard enthalpies of sublimation of the adducts were equal to the enthalpies of sublimation or vaporization of 1 mol of the respective ligand [24,25], as melting points and thermal studies showed that the adducts decomposed on heating and were not found in the liquid phase and probably, not in the gaseous phase.

Based on the $\Delta_r H^\theta$ or $\bar{D}_{(Sn–N)}$ values for the adducts, the basicity order of the ligands is obtained for the adducts of the same stoichiometry: pipd > pipz > morph > β-pico > py > γ-pico > 3-cyanopy > 4-cyanopy and quin > bipy. The expected order for pyridine and derivatives would be: γ-pico > β-pico > py > 3-cyanopy > 4-cyanopy due to an inductive effect of substitution of one hydrogen atom in the pyridine ring by the electron donor methyl group in γ-pico or β-pico or, by the electron withdrawing cyano group in 3-cyanopy or 4-cyanopy. This causes the increase or decrease, respectively, of the electronic density available for bonding on the nitrogen atom of the pyridine ring, relative to unsubstituted pyridine. The effect is stronger in *p*-substitution than in *m*-substitution, as was observed here with the exception of γ-pico. Its observed position is after pyridine. Another kind of interaction could be present

Table 4
Summary of the thermochemical results (kJ mol^{-1})

Compound	$\Delta_r H^\theta$	$\Delta_f H^\theta$	$\Delta_l^g H^\theta$ or $\Delta_s H^\theta$	$\Delta_M H^\theta$	$\Delta_D H^\theta$	$\Delta_r H^\theta$(g)	$\bar{D}_{(Sn-N)}$
$SnBr_{4(s)}$		−377.4[a]	62.8[a]				
$py_{(l)}$		101.2 ± 0.7[b]	40.2 ± 0.0[b]				
β-$pico_{(l)}$		61.9 ± 0.5[b]	44.4 ± 0.0[b]				
γ-$pico_{(l)}$		58.5 ± 1.1[b]	45.3 ± 0.4[b]				
$pipd_{(l)}$		88.0[b]	39.1 ± 1.8[b]				
$morph_{(l)}$		−170.2[c]	42.3[c]				
$pipz_{(s)}$		−45.6 ± 1.6[b]	84.2[c]				
3-$cyanopy_{(s)}$		38.0 ± 3.2[d]	89.0 ± 2.8[d]				
4-$cyanopy_{(s)}$		40.9 ± 3.9[d]	83.6 ± 3.5[d]				
$quin_{(l)}$		141.22 ± 0.92[d]	59.32 ± 0.20[d]				
$bipy_{(s)}$		216.4 ± 7.4[e]	81.93 ± 0.33[e]				
$[SnBr_4(py)_2]_{(s)}$	−174.20 ± 1.88	−349.2 ± 2.5		−317.4 ± 2.7	262.1 ± 1.9	−277.2 ± 2.7	138.6 ± 1.4
$[SnBr_4(\beta\text{-pico})_2]_{(s)}$	−205.30 ± 4.55	−458.9 ± 4.7		−356.9 ± 5.0	294.1 ± 4.6	−312.5 ± 5.0	156.3 ± 2.5
$[SnBr_4(\gamma\text{-pico})_2]_{(s)}$	−168.59 ± 1.47	−429.0 ± 2.09		−322.0 ± 2.6	259.2 ± 1.7	−276.7 ± 2.6	138.4 ± 1.3
$[SnBr_4(pipd)_2]_{(s)}$	−292.00 ± 5.89	−845.4 ± 6.0		−433.0 ± 7.2	370.2 ± 6.9	−393.9 ± 7.4	197.0 ± 3.7
$[SnBr_4(morph)_2]_{(s)}$	−222.94 ± 2.55	−940.7 ± 2.7		−370.3 ± 3.4	307.5 ± 3.2	−328.0 ± 4.8	164.0 ± 2.4
$[SnBr_4(pipz)_2]_{(s)}$	−236.23 ± 4.30	−704.8 ± 5.4		−467.4 ± 5.1	404.6 ± 4.7	−383.2 ± 5.2	196.6 ± 2.6
$[SnBr_4(3\text{-cyanopy})_2]_{(s)}$	−115.05 ± 3.53	−416.5 ± 7.4		−355.9 ± 6.9	293.1 ± 6.6	−266.9 ± 7.5	133.5 ± 3.8
$[SnBr_4(4\text{-cyanopy})_2]_{(s)}$	−76.41 ± 1.95	−372.0 ± 8.1		−306.4 ± 7.6	243.6 ± 7.3	−222.8 ± 8.3	111.4 ± 4.2
$[SnBr_4(quin)]_{(s)}$	−156.17 ± 2.55	−392.4 ± 3.4		−278.3 ± 3.2	215.5 ± 2.6	−219.0 ± 3.3	109.5 ± 1.7
$[SnBr_4(bipy)]_{(s)}$	−98.92 ± 1.31	−259.9 ± 7.6		−243.7 ± 2.4	180.85 ± 1.35	−161.7 ± 2.4	80.9 ± 1.2

[a] [19].

[b] [20].

[c] [21].

[d] [22].

[e] [23].

in this ligand, hydrogen bonding or steric hindrance, leading to the weaker than expected Sn–N bonds. The expected basicity order for pipd and derivatives would be: pipd $>$ pipz $>$ morph due to an inductive effect of substitution of one carbon atom in the pipd ring by the more electronegative nitrogen (pipz) and oxygen (morph). This leads to a decrease in the electron density available on the nitrogen atom of the unsubstituted pipd ring. Comparing the adducts obtained here with the adducts of the same stoichiometry and the same ligand formed by tin(IV) chloride [26], from the $\bar{D}_{(\mathrm{Sn–N})}$ values, in general the acidity order observed is: $SnCl_4 > SnBr_4$ as would be expected from the electronegativities of the halogen atoms, chlorine $>$ bromine, $SnCl_4$ being a stronger acid than $SnBr_4$. Some discrepancies are observed in this order for some ligands [26], mostly due to the nature of the bond formed between the donor and acceptor atoms [27].

As a whole it is observed that the mean tin(IV)–nitrogen coordinate bond dissociation energies for the adducts studied here, are higher than the mean dissociation energies of tin(IV)–oxygen or tin(IV)–sulfur bonds in comparable complexes formed by tin(IV) bromide with amides or thioamides [1,28].

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