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Adducts of antimony triiodide and 2-aminomethylpyridines: Synthesis, characterization and thermochemistry

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Abstract

Solid adducts of formula $SbI_3 \cdot L$ [L = 2-amino-3-methylpyridine (2A3P), 2-amino-4-methylpyridine (2A4P), 2-amino-5-methylpyridine (2A5P), 2-amino-6-methylpyridine (2A6P)] were synthesized and characterized by elemental analysis, IR spectroscopy, thermogravimetry and DSC. Thermochemical parameters of the adducts were determined through solution-reaction calorimetry. The ratio metal–ligand was 1:1 for all adducts based on elemental analysis. Infrared results showed that the ligands coordinate to antimony through the nitrogen atom of the aromatic ring. Thermal degradation of adducts starts at 112, 122, 188, and 137 °C for SbI_32AxP (where $x = 3$, 4, 5 and 6, respectively). Decomposition temperatures correlate with metal–ligand bond strength. The enthalpy of reaction: $SbI_3(c) + nL(c,l) = SbI_3 \cdot nL$ (solid) $\Delta_r H^\circ_m$ (solid) is −97.45, −56.43, −73.56 and −81.45 kJ mol^{-1}, respectively, for 2A3P, 2A4P, 2A5P and 2A6P adducts. The standard enthalpies of decomposition, as well as the lattice enthalpies and the enthalpies of the Lewis acid–base reaction in the gas phase were calculated through thermochemical cycles. The standard mean antimony-nitrogen bond enthalpies in $SbI_3 \cdot 2A3P$, $SbI_3 \cdot 2A4P$, $SbI_3 \cdot 2A5P$, and $SbI_3 \cdot 2A6P$ were estimated as 175, 135, 154, and 164 kJ mol^{-1}, respectively.

Keywords: Adducts; Aminopyridines; Thermochemistry; Antimony

1. Introduction

Several studies report on complexes of pyridine and its derivatives with transition metals [1–12], but no information about the synthesis and characterization of antimony complexes with 2-aminopyridine is available in the literature. Antimony is an element with biological and pharmacological importance [13,14], and the bases derived from 2-aminopyridine are used in pharmacology and medicines [15,16]. The aim of this investigation is to contribute to the coordination chemistry of p blocks elements with bases derived from amino methyl pyridines.

This paper reports the synthesis and characterization of adducts of general formula $SbI_3 \cdot L$ where L is 2-amino-3-methyl-, 2-amino-4-methyl, 2-amino-5-methyl- and 2-amino-6-methyl pyridine. Attention is focused on determination of thermochemical parameters by solution-reaction calorimetry. This investigation explored the formation of adducts between antimony (III) iodide and heterocyclic bases to evaluate the enthalpies involved. The coordinated bond dissociation enthalpies are correlated with other thermochemical parameters. The inductive effect on the energy of the antimony–nitrogen bond due to the substitution of hydrogen atoms in the pyridine ring by the electron donor methyl and amino groups is considered.

2. Experimental

The solvents used in all preparations were distilled and stored over Linde 4A molecular sieves. All 2-aminomethylpyridines (Aldrich) were used without further purification. The antimony trichloride (Merck) was dried

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under vacuum before being used. All preparations and manipulations were carried out under dry nitrogen atmosphere.

All adducts were prepared by slow addition of SbI_3 to ligand in 1:1 molar ratio with benzene as solvent under anhydrous conditions. In a typical procedure, a solution of 2.4 g of SbI_3 (4.7 mmol) dissolved in 30 cm^3 of benzene was added to a solution of 0.50 g of the ligand (4.5 mmol) in 10 cm^3 of benzene with magnetic stirring. The reaction mixture was stirred for at least 3 h. After the removal of the solvent, the dark red solid was washed with benzene and dried under vacuum. The compounds were stored in a desiccator over calcium chloride. The yields in all preparations were 80–90%. Iodide was determined by potentiometric titration with standard 0.1 mol dm^{-3} $AgNO_3$ solution [17] after dissolution of the adducts in water. Nitrogen was determined by Kjeldahl's method [18]. Carbon and hydrogen contents were determined on a Perkin-Elmer model 2400 analyzer. At least two determinations were performed for each sample.

Infrared spectra in the 4000–400 cm^{-1} range were obtained with a BOMEN model MB-102 spectrophotometer on sample mulls in KBr disc for adducts and ligands.

The melting temperatures for all compounds were determined with a model MQAPF-301 apparatus.

Thermogravimetric curves were obtained with a Shimadzu TG-50 thermobalance by heating under nitrogen. The carrier gas flow was 0.83 mL s^{-1}, the sample mass was 5.0 ± 0.5 mg and the heating rate was 10 K min^{-1}.

Reaction-solution calorimetric measurements were performed in a Hart Scientific model 4300 isoperibol calorimeter. Ampules containing 5–50 mg of substrate were prepared in a dry-box and broken into the glass reaction vessel charged with 25 mL of calorimetric solvent at 298.15 ± 0.02 K. The accuracy of calorimetric measurements was checked by measuring the thermal effect of dissolution of tris(hydroxymethyl) aminomethane (THAM) in standard hydrochloric acid solution. The value obtained was -29.70 ± 0.10 kJ mol^{-1}, in agreement with the recommended value -29.736 ± 0.002 kJ mol^{-1} [19,20].

3. Results and discussion

3.1. Characterization

Elemental analyses of the red adducts agree with the general formula $SBI_3{\cdot}L$ (L = 2A3P, 2A4P, 2A5P and 2A6P) as shown in the Table 1.

The infrared spectra of the adducts, $SbI_3{\cdot}L$, are given in Table 2, together with the assigned wave numbers of ligands [21–23]. The assigment of NH_2 stretching was based on previous reports by Carmona et al. [24], Akyuz [25], and Long [26]. The 2-amino-*x*-methylpyridine ligands (*x* = 3, 4, 5, and 6) have two nitrogen atoms with lone pairs of electrons. Although the ring nitrogen is known to be more basic than the amino nitrogen [9,27], it is necessary to determine whether the coordination occurs through the ring nitrogen or nitrogen amino. When the nitrogen amino is involved in complex formation, NH_2 stretching and bending modes shift to lower wave numbers, whereas NH_2 twisting and wagging modes shift to higher wave numbers [28,29]. When the pyridine ring nitrogen is involved in complex formation, certain vibrational modes increase in value due to both coupling with M–N (pyridine) bond vibrations and alterations of the force field [30,31]. The $\nu(NH_2)$ vibrational modes are found to be higher in wave number than those of the crystalline or liquid ligands (Table 2). However, certain ring and CH modes (ν_3, ν_4, ν_6, ν_7, ν_8, ν_9, ν_{13}, ν_{14}, ν_{15}) are found to exhibit all the characteristics of a ligand coordinated through the ring nitrogen [28,29,30–33]. Therefore, we conclude that the ring nitrogen and not the amino nitrogen is involved in complex formation.

Decomposition of the adducts was studied by thermogravimetry in the range 300–800 K. The data for all adducts, except $SbI_3{\cdot}2A3P$, showed identical behaviour with mass loss in a single step assigned to the release of the ligand molecules together with triiodide. For $SbI_3{\cdot}2A3P$, the ligand loss occurred in two steps, the first step from 400–570 K associated with the elimination of ligand groups and three iodide atoms and the second step in the range from 650–800 K, corresponding to volatilization of elemental antimony. These results establish the thermal stability in the following order: $SbI_3{\cdot}2A5P < SbI_3{\cdot}2A4P < SbI_3{\cdot}2A6P$. The mass losses 99.7, 99.9, 98.7 and 97.7% for $SbI_3 2AxP$ (where *x* = 3, 4, 5 and 6, respectively), are very close to those expected from the formulas.

3.2. Calorimetry

The energetics of ligand-metal interaction can be determined from the collected thermochemical data for a set of individual reactions. Initially, the molar enthalpy of reaction in condensed phase

$$SbI_3(cr) + nL(cr, l) = SbI_3 \cdot nL(cr) \qquad \Delta_r H_m^\circ \qquad (1)$$

Table 1
Carbon (C), hydrogen (H), nitrogen (N), and iodide (I) contents calculated (experimental) values in percentages and melting point (m.p.) for $SbI_3{\cdot}L$ adducts (L = 2A*x*P, where *x* = 3, 4, 5, 6)

Adduct	%C	%H	%N	%I	m.p. (K)
$SbI_3{\cdot}2A3P$	11.79 (12.02)	1.31 (1.33)	4.56 (4.50)	58.32 (58.96)	373
$SbI_3{\cdot}2A4P$	11.79 (11.60)	1.31 (1.23)	4.56 (4.48)	58.32 (58.43)	403
$SbI_3{\cdot}2A5P$	11.79 (11.88)	1.31 (1.22)	4.56 (4.60)	58.32 (58.06)	373
$SbI_3{\cdot}2A6P$	11.79 (11.56)	1.31 (1.30)	4.56 (4.52)	58.32 (58.76)	376

Table 2
Infrared assignments for free ligands and adducts

Assignment	2A3P	SbI_3·2A3P	2A4P	SbI_3·2A4P	2A5P	SbI_3·2A5P	2A6P	SbI_3·2A6P
$\nu_a(NH_2)$	3460s	3504s 3424s	3430s	3451s 3400vs	3454s	3483s 3435vs	3465s	3524s 3390vs
$\nu_s(NH_2)$	3376s 3200s	3325s 3163s	3300s 3131s	3297s 3174s	3303s 3170s	3321s 3187s	3360s 3198s	3279s 3176s
ν(CH)	3075w	3087m	3053m	3077m	3033m	3085w	3074m	3071m
ν(CH)	3022w	3023w	3029m	3020m	3010m	3039w	–	3040shm
$\delta(NH_2)$	1624s	1656vs	1648vs	1656vs	1632s	1664vs	1624s	1660vs
νring, ν_4	1600s	1618s	1618vs	1624s	1606s	1622s	1600s	1632s
νring, ν_{13}	1581m	1568s	1560vs	1559w	1563s	1549m	1581s	1563vw
νring, ν_5	1476s	1462w	1490vs	1481w	1505vs	1525w	–	–
$\nu_a(CH_3)$, ν_{14}	1452vs	1441w	1451vs	1454w	1465shm	1457w	1466vs	1470vw
$\nu_s(CH_3)$, ν_{15}	1385w	1389w	1374w	1368w	1395s	1401w	–	1438vw
$\nu(C{-}CH_3)$	1325vw	1357w	1336w	1300w	1319m	1317w	1339m	1381w
$\nu(C{-}NH_2)$, ν_3	1293w	1307w	1311s	1271vw	1265m	1261vw	1282w	1307w
δ(CH), ν_6	1198w	1196vw	1180m	1183w	1142s	1142m	1163w	1174w
NH_2 twisting	1139vw	1137vw	1127w	1125shw	–	1107w	1106w	1099w
νring, ν_7	1039vw	1040vw	1039w	1032w	1021m	1034w	1036w	1053w
νring wreathing	997w	1004w	983m	986w	983vw	989w	989w	1004w
γ(CH)	959w	951w	962w	969w	962w	953w	934vw	948vw
ν(CH), ν_{24}	766s	770m	792vs	775w	750m	756m	786s	794m
NH_2 wagging	673w	681w	700m	711w	–	710w	670w	670w
δring, ν_{19}	600w	592m	617vw	617w	632m	644m	635w	627m
δring, ν_{10}	560w	546m	578vw	587w	580shw	573m	595w	597m
γring, ν_{26}	530w	517m	522w	525m	516m	512s	–	512m
γring, ν_{22}	434w	427m	449vs	444m	447m	447m	431w	417m

s: strong, w: weak, m: medium, sh: sharp, vw: very weak, vs: very strong.

can be obtained at 298.15 K from the reaction-solution enthalpies ($\Delta_i H^\circ_m$) of each reagent and the products dissolved sequentially in dimethylformamide (DMF) by applying the thermochemical cycle:

$$SbI_3(c) + \text{solvent} = \text{Sol A}; \quad \Delta_1 H^\circ_m$$

$$\text{SolA} + L(l, c) = \text{Sol B}; \quad \Delta_2 H^\circ_m$$

$$SbI_3 \cdot L(c) + \text{solvent} = \text{Sol C}; \quad \Delta_3 H^\circ_m$$

Then,

$$\Delta_r H^\circ_m = \Delta_1 H^\circ_m + \Delta_2 H^\circ_m - \Delta_3 H^\circ_m \qquad (1')$$

The enthalpy changes for the processes used in the thermodynamic cycle are listed in Table 3. These values were obtained from at least four independent measurements.

Using enthalpies of formation $\Delta_f H^\circ_m$ of the adducts in condensed phase and the molar enthalpies of formation of antimony triiodide and ligands, the $\Delta_r H^\circ_m$ values were obtained for reaction (1)

$$\Delta_f H^\circ_m\,(\text{adduct}) = \Delta_f H^\circ_m(SbI_3) + \Delta_f H^\circ_m\,(\text{ligand}) \qquad (2)$$

Molar enthalpies of formation of ligands in gas phase were estimated by group contribution [34] and semi-empirical calculation [35]. Both methods generated very close results. The enthalpy of formation of adducts were calculated by AM1 method [35], this method yields enthalpy values slightly different for the four isomers considered. Sublimation enthalpies of ligands were calculated from Eq. (3) [36].

$$\Delta^{g}_{cr} H^\circ_m(L, 298\,K) = \int_{298\,K}^{T_{\mathrm{fus}}} C_{p,m}(cr)\,dT + \Delta^{i}_{cr} H^\circ_m(T_{\mathrm{fus}}) + \int_{T_{\mathrm{fus}}298\,K}^{T_{\mathrm{vap}}} C_{p,m}(l)\,dT + \Delta^{g}_{l} H^\circ_m(T_{\mathrm{vap}}) + \int_{298K}^{T_{\mathrm{fus}}} C_{p,m}(g)\,dT \qquad (3)$$

$C_{p,m}(cr)$ and $C_{p,m}(l)$ values were determined from DSC data while $C_{p.m}(g)$ was estimated from group contribution proposed by Joback and Reid [37].

Since the ligands were isomers with little difference in their heat capacities [38], only one ligand was estimated, and value of molar heat capacity obtained was used for all ligands.

The standard molar enthalpies of decomposition ($\Delta_D H^\circ_m$) and lattice enthalpy ($\Delta_M H^\circ_m$) are related to the following reactions:

$$SbI_3 \cdot nL(cr) = SbI_3(cr) + L(g); \qquad \Delta_D H^\circ_m$$

Table 3
Molar enthalpy changes in kJ mol^{-1} for dissolution reactions at 298.15 K involving the formation of SbI_3·L adducts (L = 2A*x*P where *x* = 3, 4, 5, 6)

Adduct	$\Delta_1 H^\circ_m$	$\Delta_2 H^\circ_m$	$\Delta_3 H^\circ_m$
SbI_3·2A3P	−93.98 ± 0.04	−72.34 ± 0.04	−68.92 ± 0.04
SbI_3·2A4P	−93.96 ± 0.04	−27.29 ± 0.02	−65.89 ± 0.02
SbI_3·2A5P	−93.95 ± 0.04	−28.12 ± 0.03	−54.18 ± 0.02
SbI_3·2A6P	−93.97 ± 0.04	−79.53 ± 0.01	−94.36 ± 0.01

The calorimetric solvent used was *N*,*N*-dimethylacetamide (DMA).

Table 4
Molar enthalpies in kJ mol^{-1} for the adducts

Measurement	$SbI_3 \cdot 2A3P$	$SbI_3 \cdot 2A4P$	$SbI_3 \cdot 2A5P$	$SbI_3 \cdot 2A6P$
$\Delta_r H_m^\circ$	-97.4 ± 0.02	-56.5 ± 0.42	-73.0 ± 0.23	-81.9 ± 0.02
$\Delta_f H_m^\circ(\text{cr})$	-147.2	-114.5	-130.4	-129.4
$\Delta_D H_m^\circ(\text{g})$	202.7	161.2	177.3	194.6
$\Delta_M H_m^\circ$	-253.8	-220.5	-236.1	-243.3
$\Delta_f H_m^\circ(\text{g})^b$	-68.4	-33.0	-49.3 (-48.8)	48.2
<D> (Sb—N)[a]	199.1	158.3	174.7	183.6
<D> (Sb—N)[b]	175.3	135.6	153.9	163.7

Parameters assigned with a and b were estimated using the first and second hypothesis, respectively. Values in parenthesis were calculated through AM1 method.

$$SbI_3 \cdot n\text{L(cr)} = SbI_3\text{(g)} + \text{L(g)}; \qquad \Delta_M H_m^\circ$$

These enthalpy values can be calculated by means of the expressions:

$$\Delta_D H_m^\circ = -\Delta_r H_m^\circ + \Delta_{cr}^{g} H_m^\circ(\text{L}) \tag{4}$$

$$\Delta_M H_m^\circ = \Delta_D H_m^\circ + \Delta_{cr}^{g} H_m^\circ(SbI_3) \tag{5}$$

From the molar enthalpies of sublimation of ligands determined by DSC data and also from estimative methods [39] and auxiliary data of enthalpy of sublimation of SbI_3 [40], the values of $\Delta_D H_m^\circ$ and $\Delta_M H_m^\circ$ were derived. The results are presented in Table 4.

To determine the metal-nitrogen bond enthalpy of the adducts, information about the acid–base reaction in the gaseous phase is desirable [41–45] $\Delta_r H_m^\circ(\text{g})$, and it is obtained from the reaction:

$$SbI_3 \cdot n\text{L(g)} = SbI_3\text{(g)} + n\text{L(g)} \qquad \Delta_g H_m^\circ$$

The enthalpy change ($\Delta_M H_m^\circ$) is given by the expression:

$$\Delta_g H_m^\circ = \Delta_M H_m^\circ + \Delta_{cr}^{g} H_m^\circ\,(\text{adduct}) \tag{6}$$

where $\Delta_{cr}^{g} H_m^\circ$ (adduct) is the sublimation enthalpy of the adduct.

Using $\Delta_g H_m^\circ$ values for these adducts, the mean metal-nitrogen bond enthalpy can be calculated by the expression:

$$\langle \text{D} \rangle(\text{Sb–L}) = \frac{\Delta_g H_m^\circ}{n} \tag{7}$$

where n represents the number of ligands coordinated to antimony in the adduct. The mean antimony-nitrogen bond enthalpy, <D> (Sb–N), depends on the enthalpy of sublimation of the compound, which was unstable during the heating process. All attempts to directly measure the thermochemical parameters for adducts in gas phase were unsuccessful due to the difficulty in subliming the adducts. To resolve this problem, we assumed the enthalpy $\Delta_{cr}^{g} H_m^\circ$ is equal to: a) $\Delta_{cr}^{g} H_m^\circ$ (ligand), b) $[n\Delta_{cr}^{g} H_m^\circ\,(\text{ligand}) + \Delta_{cr}^{g} H_m^\circ(SbI_3)]/(n+1)$, c) $\{[\Delta_{cr}^{g} H_m^\circ\,(\text{ligand})]^n [\Delta_{cr}^{g} H_m^\circ(SbI_3)]\}^{1/(n+1)}$ and d) $\Delta_{cr}^{g} H_m^\circ(SbI_3)$ [41,46]. Another hypothesis is a linear relation between the molar enthalpy in solid state and the molar enthalpy in gas state for an extensive number of organic and organometallic compounds described in literature [47,48]. A better correlation was obtained if the range of sublimation enthalpy decreases and enthalpy estimations were performed in solid and liquid states.

Data on sublimation enthalpy between 60 and 90 kJmol^{-1} for 64 compounds were used for estimating the enthalpy of sublimation of adducts as shown in Fig. 1. The obtained relation was:

$$\Delta_f H_m^\circ(\text{g}) = 1.0016\Delta_f H_m^\circ(\text{s}) + 75.52 \tag{8}$$

Expressed in kJ mol^{-1} with a coefficient of correlation of 0.9998. The difference between the estimated and experimental values of formation enthalpy in gas state using this expression was less than 10 kJ mol^{-1}.

The values of formation enthalpy in gas state of adducts were estimated by using each of the four hypothesis and a linear equation as expression 8. In some cases [49–53], the regression generated values that agreed with data obtained from the first hypothesis 'a'. However, for other adducts the values agreed with the ones obtained from the second hypothesis 'b'. This behaviour suggests that the approximations can be appropriate in some cases.

Therefore, we adopted the following methodology to estimate formation enthalpy of the adducts in gaseous state: (1) We firstly estimated each one of the hypothesis in gas state. (2) Secondly we substituted each value obtained in the linear equation at solid state. (3) The data at the gas phase were

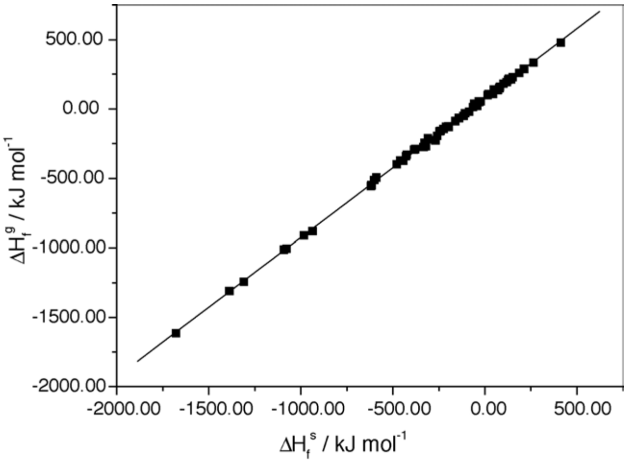

Fig. 1. Correlation between molar enthalpy of formation in gas and solid states of selected organic and organometallic compounds at 298.15 and 1 atm.

selected by comparing the values obtained in the solid phase with those obtained by calorimetry (Table 3), a procedure that generated a greater agreement among formation enthalpy values in the solid phase. The values of $\Delta_f H_m^\circ$(cr) and $\Delta_f H_m^\circ$(g) of adducts are shown in Table 4. In this table, the Sb–N bonding enthalpies were based on assumptions of the hypothesis 'a' for some adducts [41,48], for which the hypothesis was reasonable within ± 10 kJ mol^{-1} and on assumptions of the hypothesis 'b' for others [41,48]. The N–Sb enthalpy from hypothesis a and the hypothesis b differed by 24, 23, 21 and 20 kJ mol^{-1} for adducts with 2A3P, 2A4P, 2A5P and 2A6P. The most probable Sb–N bonding enthalpy was the one obtained from enthalpy of formation of adducts with the second hypothesis. The estimation performed by AM1 [35] was satisfactory for $SbI_3\cdot 2A5P$, in good agreement with the experimental value. The values for other compounds were in total disagreement.

The results obtained from the molar formation enthalpies of adducts in the condensed phase showed a substantial difference between the ligands, hence of the adducts. Thus, it is suggested that the substitution of hydrogen by methyl group was promising. For a series of adducts with the same stoichiometry, ΔS° values can be considered constant and a direct correlation between ΔG° and ΔH° values can be obtained. The stability sequence of the adducts is 2a3P > 2a6P > 2a5P > 2a4P, considering the data of $\Delta_r H_m^\circ$, $\Delta_M H_m^\circ$, $\Delta_D H_m^\circ$ and $\Delta_f H_m^\circ$.

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