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Thermochemistry of aniline derivatives complexes of arsenic trihalides

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Abstract

The complexes $AsX_3 \cdot nL$ (where L is 2,6-dimethylaniline(dimean), *o*-, *m*- or *p*-chloroaniline (*o*-, *m*- or *p*-chloran), $n = 1, 3/2, 5/2$ or 3, and X is Cl, Br or I) were synthesized and characterized by melting points, elemental analysis, TG and DTG analysis, mass spectra and IR spectroscopy. From the enthalpies of dissolution in 25% (v/v) ethanolic ethanolamine and using appropriate thermochemical cycles, the following thermochemical parameters for the adducts have been determined: the standard enthalpies for the Lewis acid/base reaction ($\Delta_r H^\ominus$), the standard enthalpies of formation ($\Delta_f H^\ominus$), the standard enthalpies of decomposition ($\Delta_D H^\ominus$), the lattice standard enthalpies ($\Delta_M H^\ominus$), and the enthalpies of the Lewis acid/base reactions in the gaseous phase ($\Delta_r H^\ominus(g)$). The mean standard enthalpies of the arsenic–nitrogen bonds have been estimated.

Keywords: Aniline; Arsenic; DTG; Halide; TGA

1. Introduction

Aniline and *o*, *m*- and *p*-nitroaniline are known to form coordination compounds with arsenic trihalides. Their preparation, characterization and thermochemistry have been reported recently [1–3]. It is expected that other substituted anilines will also form coordination compounds with arsenic trihalides. The substitution of one hydrogen atom in the phenyl group by the electron-withdrawing chlorine atom will cause a decrease in the electronic density in the aromatic ring. Consequently, by an inductive effect, the electronic density on the nitrogen atom will also be decreased. The substitution of two hydrogen atoms by two electron-donating methyl groups will cause an

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increase in the electronic density in the aromatic ring and consequently the electronic density on the nitrogen atom will also be increased. This paper describes the interaction of arsenic trihalides with 2,6-dimethylaniline, and *o*-, *m*- and *p*-chloroaniline to form adducts in which the nitrogen atom is the coordinating atom of the ligands. Calorimetric measurements were made to determine the mean strength of the As–N bonds. The effect of introducing a chlorine atom or two methyl groups into the phenyl group of aniline on the energy of the As–N bond is also studied.

2. Experimental

All preparations and manipulations were carried out in polyethylene glove bags under a nitrogen atmosphere.

2.1. Chemicals

Arsenic trihalides were prepared as described in a previous paper [4]. 2,6-Dimethylaniline, and *o*-, *m*- and *p*-chloroaniline were purified by a method analogous to that employed for purification of aniline [5]. Solvents used in the synthesis of adducts were purified by distillation and stored over Linde 4A molecular sieves. Ethanol (94.7–95.2% (v/v), Carlo Erba) and ethanolamine (99% (p/v), Casa Americana de artigos para laboratórios Ltda.) were used without further purification for preparing the calorimetric solvent. Ethanol was added to 250 ml of ethanolamine until a volume of 1 L of solution was reached.

2.2. Analytical

Carbon, hydrogen and nitrogen contents were determined by microanalytical procedures. Halogen analysis was obtained by gravimetry using standard 0.1 N $AgNO_3$ solution, after the adducts had been dissolved in water [6]. Arsenic content was determined by redox titration of the aqueous solution of adduct samples, with standard 0.05 N iodine solution, to the appearance of the blue color of starch used as an indicator [7].

2.3. Adduct synthesis

The adducts were prepared by the reaction of arsenic trihalides and the ligands in solution. A typical procedure is given below.

2.4. $AsCl_3 \cdot 2.5$(*o*-chloran)

To a solution of 2.5 ml of *o*-chloroaniline (23.9 mmol) in 10 ml of petroleum ether, 2 ml of $AsCl_3$ (23.9 mmol) was added slowly and dropwise with stirring. The stirring was maintained for at least 3 h. After filtration, the white crystals formed were washed with three portions of 20 ml of petroleum ether, and dried for several hours in vacuo.

The compound obtained was stored in a desiccator over calcium chloride. All the adducts were prepared with a donor/acceptor molar ratio of 1/1. In all cases, petroleum ether was used as a solvent for washing. For the adducts of $AsBr_3$ and AsI_3, $CHCl_3$ and CS_2 respectively were used as solvents. Only for *o*-chloran adducts of $AsBr_3$ and AsI_3 was cooling used to precipitate the compounds.

2.5. *Infrared spectra*

These were obtained using samples of the adducts or *p*-chloran in KBr matrix in the region 4000–400 cm^{-1} in a Perkin-Elmer 1600 series FTIR spectrophotometer. For dimean, *o*-, or *m*-chloran, a film of ligand sandwiched between KBr plates was used.

2.6. *Thermal studies*

These were made in an argon atmosphere in a Du Pont 951 TG Analyser, with samples varying in mass from 2 to 17 mg and a heating rate of 10 K min^{-1}.

2.7. *Calorimetric measurements*

All the solution calorimetric determinations were carried out in an LKB 8700-1 precision calorimeter as described before [4, 8]. The solution calorimetric measurements were performed by dissolving samples of 5–200 mg of adduct or trihalide in 100 ml of calorimetric solvent and the ligand in the solution obtained after the measurement of the heat evolved by the dissolution of the arsenic trihalide, always maintaining the molar relation ligand/trihalide equal to the stoichiometry of the adduct.

2.8. *Mass spectra*

The mass spectra of the compounds were recorded on a Finningan 1015 S/L spectrometer.

3. Results and discussion

All the adducts prepared were solids. The yields range from 13 to 65%. The yields, melting points, colours, appearance and analytical data are summarized in Table 1.

3.1. *Infrared data*

The infrared spectra of the adducts show dislocation of some bands and the appearance of new ones when compared with the spectra of free ligands. The pattern of the spectra of the adducts is similar to that of the free ligands. There is a shift of the N–H stretching and bending modes of the adducts to lower frequencies when compared with the free ligands, indicating a weakening of this bond by coordination through the nitrogen atom [9–17]. Table 2 presents the extracted infrared spectral data for N–H

Table 1
Yields in % for the preparation, and the melting points, appearance and analytical data of the adducts

Compound	Yield	M.P./K	Appearance[a]	C		H		N		Halogen		Arsenic	
				Calcd.	Found	Calcd.	Found	Calcd.	Found	Calcd.	Found	Calcd.	Found
$AsCl_3$·2.5 dimean	15	449	wh, cr	49.60	–	5.72	–	7.23	–	21.96	22.45	15.47	15.50
$AsBr_3$·1.5 dimean	58	430–2	s, gr, pw	29.03	28.80	29.03	28.80	4.23	4.26	48.29	48.05	–	–
AsI_3·dimean	65	423	ye, pw	16.66	16.73	1.92	1.84	2.43	2.44	66.00	65.74	–	–
$AsCl_3$·2.5 *o*-chloran	20	483–5	wh, cr	36.02	–	3.02	3.16	7.00	–	21.26	20.89	14.98	14.79
$AsBr_3$·3 *o*-chloran	13	457	wh, pw	31.00	31.21	2.60	2.49	6.03	5.96	34.37	34.40	–	–
AsI_3·*o*-chloran	27	383	br, cr	12.36	12.02	1.04	0.95	2.40	2.29	65.28	65.47	–	–
$AsCl_3$·2.5 *m*-chloran	20	488	wh, cr	36.02	–	3.02	3.09	7.00	–	21.26	21.29	14.98	14.99
$AsBr_3$·3 *m*-chloran	22	449–50	wh, pw	31.00	31.62	2.60	2.45	6.03	6.06	34.37	34.73	–	–
AsI_3·3 *m*-chloran	27	391	ye, pw	25.79	25.44	2.16	2.06	5.01	4.76	45.41	45.11	–	–
$AsCl_3$·2.5 *p*-chloran	21	498	wh, cr	36.02	–	3.02	3.21	7.00	–	21.76	21.39	14.98	14.28
$AsBr_3$·2 *p*-chloran	47	436	wh, pw	25.30	25.01	2.12	2.02	4.92	4.88	42.07	42.57	–	–
AsI_3·3 *p*-chloran	13	448	ye, pw	25.79	25.39	2.16	2.03	5.01	4.96	45.41	45.10	–	–

[a] Key: wh, white; gr, gray; ye, yellow; br, brown; cr, crystals; pw, powder; s, slightly.

Table 2
IR spectral data[a]

Compound	N–H$_{stretching}$/cm^{-1}	N–H$_{deformation}$/cm^{-1}
Dimean	3471m, 3386s	1623s
$AsCl_3$·2.5 dimean	3427m, 3367m	1624s, 1585s
$AsBr_3$·1.5 dimean	3424s, 3333s	1622m, 1583m
AsI_3·dimean	3444m	1628m, 1560m
o-Chloran	3470m, 3379m	1617s
$AsCl_3$·2.5 *o*-chloran	3450m	1612m, 1582m
$AsBr_3$·3 *o*-chloran	3452m	1608m, 1570m
AsI_3·*o*-chloran	3449s	1622m
m-Chloran	3444m, 3362m	1620s, 1597s
$AsCl_3$·2.5 *m*-chloran	3446m	1598s, 1575s
$AsBr_3$·3 *m*-chloran	3456m	1596m, 1553s
AsI_3·3 *m*-chloran	3441m	1596m, 1556m
p-Chloran	3471m, 3381m	1616s
$AsCl_3$·*p*-chloran	3429m	1615m, 1593s
$AsBr_3$·2 *p*-chloran	3444s	1611m, 1555m
AsI_3·3 *p*-chloran	3456m	1637w, 1577w

[a] Intensity of bands: *s*, strong; *m*, medium; *w*, weak.

stretching and bending frequencies of the adducts. Fig. 1 presents the IR spectra of a representative adduct.

3.2. *TG data*

The TG data of the complexes are similar. Nearly all of them involve the ligand together with arsenic trihalide in a single step. The exceptions are as follows: $AsBr_3$·1.5 dimean showed a two-step mass loss, the first with elimination of ligand and part of the arsenic tribromide, and the second with elimination of the rest of the tribromide. AsI_3·*o*-chloran showed a two-step mass loss, the first with elimination of part of the ligand and the second with elimination of the arsenic triiodide and the rest of the ligand. In several compounds, a residue was obtained, probably carbon, as the degradation product of the ligand [3]. Table 3 lists the thermoanalytical data of the adducts. Figs. 2 and 3 present the TG and DTG curves of two representative compounds.

3.3. *Mass spectra*

The mass spectra of the complexes did not show the parent ion. They showed fragmentation of the adducts. Several peaks characteristic of fragmentation of ligands and arsenic trihalides were also observed.

3.4. *Calorimetric measurements*

The standard enthalpies of dissolution of arsenic trihalides, ligands and complexes ($\Delta_i H^\ominus$) were obtained as previously reported [4]. Table 4 gives these standard

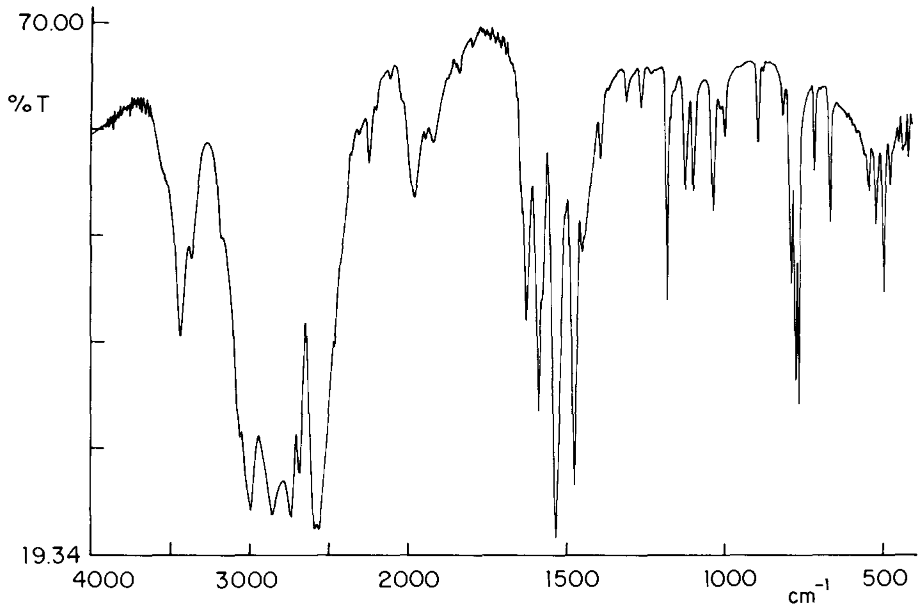

Fig. 1. IR spectra of $AsCl_3$·2.5 dimean.

Table 3
Thermoanalytical data of the compounds AsX_3·*n*L

Compound	Weight loss (%)		Temperature range/K	Attribution
	Calcd.	Obs.		
$AsCl_3$·2.5 dimean	100	99.04	422–673	$AsCl_3$, 2.5 dimean
$AsBr_3$·1.5 dimean	50.70	50.71	387–420	2/9 $AsBr_3$, 1.5 dimean
	49.30	48.87	420–679	7/9 $AsBr_3$
AsI_3·dimean	100	94.23	408–464	AsI_3, dimean
		6.97	464–1018	Residue
$AsCl_3$·2.5 *o*-chloran	100	98.44	402–602	$AsCl_3$, 2.5 *o*-chloran
		2.77	602–1033	Residue
$AsBr_3$·3 *o*-chloran	100	100	411–484	$AsBr_3$, 3 *o*-chloran
AsI_3·*o*-chloran	16.40	16.36	347–371	3/4 *o*-chloran
	83.60	80.71	371–459	1/4 *o*-chloran, AsI_3
		2.93	459–1023	Residue
$AsCl_3$·2.5 *m*-chloran	100	91.69	394–432	$AsCl_3$, 2.5 *m*-chloran
		8.31	432–1026	Residue
$AsBr_3$·3 *m*-chloran	69.00	72.23	422–476	$AsBr_3$, H, N, Cl
		29.08	476–1034	Residue
AsI_3·3 *m*-chloran	100	100	374–451	AsI_3, 3 *m*-chloran
$AsCl_3$·2.5 *p*-chloran	100	100	434–653	$AsCl_3$, 2.5 *p*-chloran
$AsBr_3$·2 *p*-chloran	100	95.35	399–436	$AsBr_3$, 2*p*-chloran
		5.92	392–1020	Residue
AsI_3·3 *p*-chloran	74.21	76.35	395–441	AsI_3, H, N, Cl
		23.65	441–1021	Residue

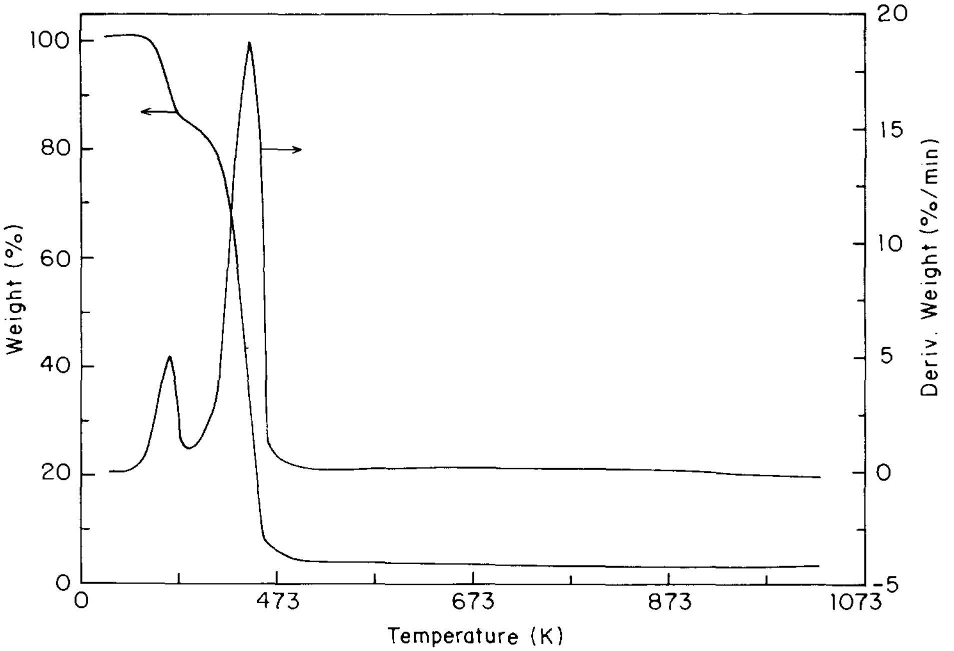

Fig. 2. TG and DTG curves of $AsI_3 \cdot$*o*-chloran.

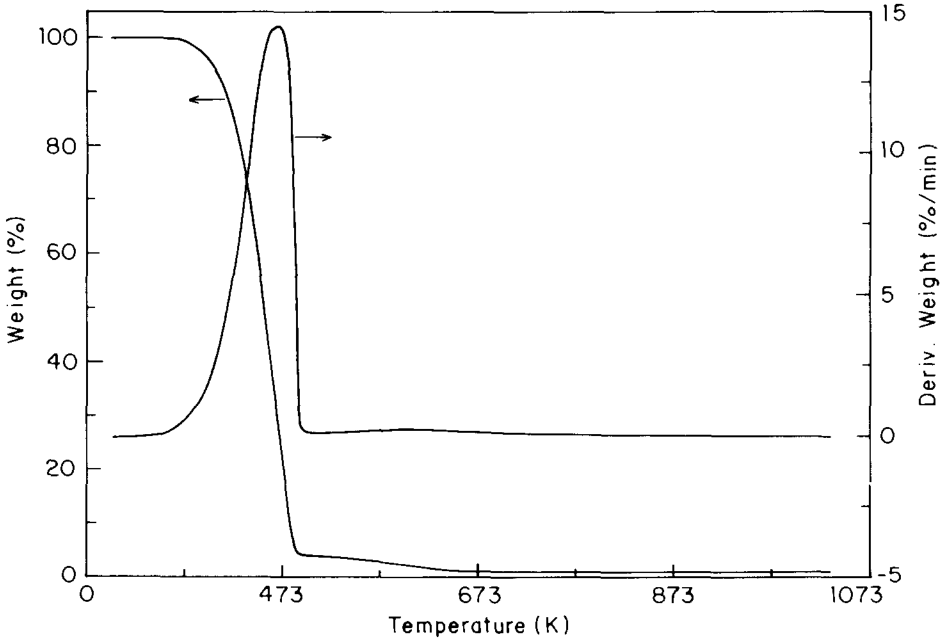

Fig. 3. TG and DTG curves of $AsCl_3 \cdot 2.5$ dimean.

Table 4
Enthalpies of dissolution at 298.15 K

Compounds	Calorimetric solvent[a]	Number of experiments	$\Delta_i H^\ominus$/kJ mol^{-1}
$\mathrm{AsCl_3}$(l)	25% eth.EA	11	$(i=1)-303.96\pm1.47$
Dimean(l)	2.5 ÷ 1$\mathrm{AsCl_3}$ − 25% eth.EA	5	$(i=2)-7.49\pm0.73$
$\mathrm{AsCl_3}$·2.5 dimean(s)	25% eth.EA	4	$(i=3)-99.89\pm1.49$
$\mathrm{AsBr_3}$(s)	25%eth.EA	6	$(i=1)-297.42\pm1.55$
Dimean(l)	1.5 ÷ 1$\mathrm{AsBr_3}$ − 25% eth.EA	3	$(i=2)-4.23\pm0.36$
$\mathrm{AsBr_3}$·1.5 dimean(s)	25% eth.EA	5	$(i=3)-201.38\pm4.29$
$\mathrm{AsI_3}$(s)	25% eth.EA	6	$(i=1)-246.33\pm2.88$
Dimean(l)	1 ÷ 1$\mathrm{AsI_3}$ − 25% eth.EA	6	$(i=2)-1.18\pm0.07$
$\mathrm{AsI_3}$·dimean(s)	25% eth.EA	4	$(i=3)-189.17\pm4.68$
o-Chloran(l)	2.5 ÷ 1$\mathrm{AsCl_3}$ − 25% eth.EA	5	$(i=2)-8.74\pm0.09$
$\mathrm{AsCl_3}$·2.5 *o*-chloran(s)	25% eth. EA	4	$(i=3)-84.73\pm2.55$
o-Chloran(l)	3 ÷ 1$\mathrm{AsBr_3}$ − 25% eth. EA	5	$(i=2)-8.61\pm0.43$
$\mathrm{AsBr_3}$·3 *o*-chloran(s)	25% eth.EA	4	$(i=3)-113.30\pm2.54$
o-Chloran(l)	1 ÷ 1 $\mathrm{AsI_3}$ − 25% eth.EA	5	$(i=2)-2.92\pm0.15$
$\mathrm{AsI_3}$·*o*-chloran(s)	25% eth.EA	4	$(i=3)-232.33\pm3.11$
m-Chloran(l)	2.5 ÷ 1$\mathrm{AsCl_3}$ − 25% eth.EA	6	$(i=2)-15.67\pm0.31$
$\mathrm{AsCl_3}$·2.5 *m*-chloran(s)	25% eth.EA	5	$(i=3)-86.13\pm1.69$
m-Chloran(l)	3 ÷ 1$\mathrm{AsBr_3}$ − 25% eth.EA	5	$(i=2)-11.90\pm1.32$
$\mathrm{AsBr_3}$·3 *m*-chloran(s)	25% eth.EA	3	$(i=3)-113.04\pm0.59$
m-Chloran(l)	3 ÷ 1$\mathrm{AsI_3}$ − 25% eth.EA	3	$(i=2)-15.83\pm0.25$
$\mathrm{AsI_3}$·3 *m*-chloran(s)	25% eth.EA	4	$(i=3)-122.23\pm0.40$
p-Chloran(s)	2.5 ÷ 1$\mathrm{AsCl_3}$ − 25% eth.EA	6	$(i=2)+26.92\pm0.22$
$\mathrm{AsCl_3}$·2.5 *p*-chloran(s)	25% eth.EA	4	$(i=3)-104.01\pm3.42$
p-Chloran(s)	2 ÷ 1$\mathrm{AsBr_3}$ − 25% eth.EA	5	$(i=2)+22.41\pm0.15$
$\mathrm{AsBr_3}$·2 *p*-chloran(s)	25% eth.EA	4	$(i=3)-192.19\pm1.84$
p-Chloran(s)	3 ÷ 1$\mathrm{AsI_3}$ − 25% eth.EA	3	$(i=2)+37.05\pm0.56$
$\mathrm{AsI_3}$·3 *p*-chloran(s)	25% eth.EA	3	$(i=3)-126.97\pm1.43$

[a] Solvent eth.EA, ethanolic ethanolamine.

thermochemical values. Uncertainty intervals given in the table are twice the standard deviations of the means of about five replicate measurements on each compound. Combined errors were calculated from the square root on the sum of the squares of the component errors. From the standard enthalpies of dissolution, the standard enthalpies of the Lewis acid/base reaction ($\Delta_r H^\ominus$), the standard enthalpies of formation ($\Delta_f H^\ominus$), the standard enthalpies of decomposition ($\Delta_D H^\ominus$), the lattice standard enthalpies ($\Delta_M H^\ominus$) and the enthalpies of the Lewis acid/base reactions in the gaseous phase ($\Delta_r H^\ominus$(g)) can be obtained [4, 18].

The $\Delta_r H^\ominus$(g) values can be used to calculate the standard enthalpy of the arsenic–nitrogen ($\bar{D}$(As–N)) bond [4]. Table 5 lists the values for all these parameters. For the determination of $\Delta_r H^\ominus$(g), it was necessary to assume that the molar standard enthalpy of sublimation of each adduct was equal to the enthalpy of vaporization or sublimation of one mol of ligand [4, 18, 20, 21], as melting points, TG studies and mass spectra showed that they decompose on heating and are not found in the gaseous phase.

Table 5
Summary of the thermochemical results (kJ mol^{-1})

Compound	$\Delta_r H^\ominus$	$\Delta_f H^\ominus$	$\Delta_l^g H^\ominus$ or $\Delta_s^g H^\ominus$	$\Delta_M H^\ominus$	$\Delta_D H^\ominus$	$\Delta_r H^\ominus(g)$	$\bar{D}$(As–N)
$AsCl_3$(l)		−305.0[a]	43.5[a]				
$AsBr_3$(s)		−195.5[a]	67.5[a]				
AsI_3(s)		−58.2[a]	95.0[a]				
Dimean(l)		−45.1[b]	66.9[b]				
o-Chloran(l)		−6.8[c]	62.2[c]				
m-Chloran(l)		−11.8[c]	61.9[c]				
p-Chloran(s)		−39.4[c]	90.5[c]				
$AsCl_3\cdot 2.5$ dimean(s)	−211.55 ± 2.22	−629.3		−422.4	378.9	−355.4	142.2
$AsBr_3\cdot 1.5$ dimean(s)	−100.27 ± 4.58	−365.5		−268.2	200.7	−201.3	134.2
$AsI_3\cdot$dimean(s)	−58.34 ± 5.50	−161.6		−220.2	125.2	−153.3	153.3
$AsCl_3\cdot 2.5$ (*o*-chloran)(s)	−227.97 ± 2.94	−550.0		−427.0	383.5	−364.8	145.9
$AsBr_3\cdot 3$(*o*-chloran)(s)	−192.73 ± 3.01	−410.6		−446.8	379.3	−348.6	128.2
$AsI_3\cdot$(*o*-chloran)(s)	−16.92 ± 4.24	−81.9		−174.1	79.1	−111.9	111.9
$AsCl_3\cdot 2.5$ (*m*-chloran)(s)	−233.50 ± 2.26	−568.0		−431.8	388.3	−369.9	148.0
$AsBr_3\cdot 3$(*m*-chloran)(s)	−196.28 ± 2.12	−429.2		−449.5	382.0	−387.6	129.2
$AsI_3\cdot 3$(*m*-chloran)(s)	−139.93 ± 2.92	−233.5		−420.6	325.6	−358.7	119.6
$AsCl_3\cdot 2.5$ (*p*-chloran)(s)	−173.03 ± 3.73	−576.5		−442.8	399.3	−352.3	140.9
$AsBr_3\cdot 2$(*p*-chloran)(s)	−82.82 ± 2.41	−358.7		−331.3	98.2	−240.8	120.4
$AsI_3\cdot 3$(*p*-chloran)(s)	−82.31 ± 3.26	−258.7		−448.8	353.8	−358.3	119.4

[a] Ref. [19]. [b] See text and auxiliary data. [c] See text and auxiliary data.

For the calculation of the thermochemical parameters of the adducts, it was necessary to calculate the standard enthalpies of formation and vaporization or sublimation of the ligands. These enthalpy values are not cited in the literature. They were calculated by a group contribution method [22–25]. Table 6 presents the auxiliary data used for these calculations. The enthalpies of formation in gaseous phase and the enthalpy of vaporization of aniline were used for the calculation of these parameters for dimean; aniline, *o*-, *m*-, and *p*-nitroaniline enthalpies were used for *o*, *m*- and *p*-chloroaniline. To evaluate the contribution of the group $[C_b{-}(C_b)_2\ Cl]$ to the enthalpy of vaporization or sublimation of *o*-, *m*- and *p*-chloran, the relation between the enthalpies of vaporization of benzene and chlorobenzene (Table 6), was established, giving a value of 12.1 kJ mol^{-1}.

For the series of complexes with the same stoichiometry and the same ligand, from the respective $\Delta_r H^\ominus$ or $\bar{D}$(As–N) values, the acidity order $AsBr_3 > AsI_3$ is obtained as would be expected from the electronegativity values of bromine and iodine atoms. If the trihalide is fixed and the ligand changed, we obtain for the adducts of the same stoichiometry, the basicity order *p*-chloran < dimean < *m*-chloran ≈ *o*-chloran. The expected order on the basis of the inductive effect by the substitution of one hydrogen atom in the benzene ring by the electron-withdrawing chlorine atom is *p*-chloran < *m*-chloran ≈ *o*-chloran. Dimean is expected to be a better base than chloran as two hydrogens of the benzene ring are substituted by two electron-donating methyl groups.

Table 6
Auxiliary data

Compound	$\Delta_f H^\ominus$	$\Delta_l^g H^\ominus$ or $\Delta_s^g H^\ominus$
An(g)	87.1 ± 0.8 [a]	55.8 ± 1.1 [a]
o-NO_2 an (g)	63.8 ± 4.2 [a]	73.2 ± 4.3 [a]
m-NO_2 an (g)	58.5 ± 1.3 [a]	72.9 ± 1.6 [a]
p-NO_2 an (g)	59.5 ± 2.5 [a]	101.5 ± 2.5 [a]
C_6H_6(l)		33.9 ± 0.4 [a]
ClC_6H_5(l)		40.3 ± 1.0 [a]

[a] Ref. [26].

The position observed for dimean could be due to a steric hindrance, decreasing the basicity of dimean.

Comparing the $\Delta_r H^\ominus$ or $\bar{D}$(As–N) values of the $AsBr_3$ adducts of chloranilines, aniline [3] and nitroanilines [3] of the same stoichiometry, the basicity order nitroanilines < aniline < chloroanilines is obtained. The expected order is nitroanilines < chloroanilines < aniline, as a nitro group has a higher electron-withdrawing ability than a chlorine atom. The inversion in the expected order could be due to the nature of the bond formed between the donor and acceptor atoms [4, 10, 27, 28].

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