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A thermochemical study of adducts of 2-pyrrolidone with zinc(III), cadmium(II) and mercury(II) halides

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Abstract

The standard molar enthalpies of acid–base reactions in the condensed state ($\Delta_r H^{\ominus}$) of the adducts $MX_2 \cdot n$BuL (where M is Zn, Cd, Hg; X is Cl, Br, I; $n = 1, 2, 3$; BuL is 2-pyrrolidone) were determined from enthalpies of dissolution ($\Delta_i H^{\ominus}$) of metal halide ligands and adducts, at 298.15 K, obtained by calorimetric methods in solution. Using the values of $\Delta_r H^{\ominus}$ and auxiliary data from the literature the standard enthalpies of formation ($\Delta_f H^{\ominus}$), decomposition ($\Delta_d H^{\ominus}$) and lattice enthalpy ($\Delta_{latt} H^{\ominus}$) of these adducts were determined. The enthalpy of vaporization of BuL, $\Delta_{vap} H^{\ominus}$(BuL), was found to be 41.71 ± 0.56 kJ mol^{-1} and, from this value, the enthalpies of reaction in the gaseous state ($\Delta_g H^{\ominus}$) were estimated. The values of $\Delta_g H^{\ominus}$ were used in calculations of the mean dissociation enthalpy of the metal–oxygen bond, $\bar{D}$(M–O), of the obtained adducts, and are correlated with energetic parameters of the metal halides.

Keywords: Enthalpy; Halide; Pyrrolidone

1. Introduction

The study of lactams as useful models for determining the structure of biologically important peptides and proteins has been mentioned [1]. Among the existing series, 2-pyrrolidone (BuL) is the simplest cyclic amide and, because of its structural

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similarity to cyclic peptides, is of great interest in the study of biological systems [2]. It is also interesting to mention the importance of oxygen-bonded complexes, in which interactions with atoms of transition metals are present, in certain biological processes, as has been emphasized in Ref. [3]. In this work considerable attention has been paid to compounds of BuL with zinc, cadmium and mercury halides.

The literature deals with structural aspects of some compounds with BuL. However, only a few thermochemical studies have been carried out, such as obtaining thermochemical data of BuL complexes with zinc chloride [3,4]. We report here on a study of the thermochemistry of $ZnX_2 \cdot n$BuL (where $n = 2, 3$; X is Cl, Br, I), $MX_2 \cdot$ BuL (where M is Cd, Hg; X is Cl, Br) and $CdI_2 \cdot$ 2BuL adducts, with emphasis on evaluating the metal–oxygen interactions in these compounds. Another objective is to correlate the thermochemical data for the adducts in the gaseous phase with some energetic parameters of their respective metal halides.

2. Experimental

2.1. Chemicals

2-Pyrrolidone, reagent grade, was obtained from Aldrich and purified by vacuum distillation at 393 K. We prepared anhydrous zinc chloride according to a method described in the literature [5], and $CdBr_2 \cdot 4H_2O$ was made anhydrous by heating for 10 h in vacuo at 423 K [6]. The remaining pure metal halides (Aldrich) were used without further purification, although they were vacuum-dried before use and EDTA titration was used to check their purity by analyzing the metal [7,8]. All the air-sensitive metal halides were handled in a dry-nitrogen atmosphere using a glove-bag. The solvents (Merck) used in all preparations were purified following literature procedures [9] and kept dry.

2.2. Preparations

The adducts were prepared by the following procedures. $ZnCl_2 \cdot$ 2BuL, $HgCl_2 \cdot$ BuL and $HgBr_2 \cdot$ BuL were crystallized from an ethanolic solution of the appropriate metal halide to which BuL, dissolved in ethanol, was added in the mole ratios 1:2, 1:2.5 and 1:4, respectively. To obtain the adducts $ZnBr_2 \cdot$ 2BuL, $CdBr_2 \cdot$ BuL and $CdI_2 \cdot$ 2BuL, the corresponding crystalline metal halide was dissolved in ethanol and pure BuL was added to the resulting solution at respective molar ratios of 1:2, 1:4 and 1:3. $CdCl_2 \cdot$ BuL was obtained in the same way, but from a methanolic solution at a 1:2.5 stoichiometric ratio. The $ZnI_2 \cdot$ 3BuL and $ZnL_2 \cdot$ 2BuL adducts were prepared by refluxing, for at least 6 h, BuL and zinc iodide dissolved in ethanol in a ratio corresponding to the desired stoichiometry. This operation was also performed for $ZnCl_2 \cdot$ 3BuL and $ZnBr_2 \cdot$ 3BuL. Attempts to obtain an adduct of BuL with mercury iodide were unsuccessful. Except for $CdCl_2 \cdot$ BuL, $HgCl_2 \cdot$ BuL and $CdBr_2 \cdot$ BuL, which started to precipitate during the ligand addition, all the adducts precipitated after evaporation of the solvent in vacuum. Either hexane or ethyl acetate, or a mixture of both, was added to the paste obtained in most cases. After several consecutive evaporations, crystals were obtained. In all cases the adducts were filtered, washed with either hexane or ethyl acetate, and dried in vacuum for at least 6 h. The adducts were obtained in approximately 80% yields and, since the majority were extremely sensitive to moist air, all manipulations were performed in a glove-bag flushed with nitrogen.

The adducts were characterized by elemental analysis (where metal, carbon, nitrogen and hydrogen contents were determined), thermal analysis and infrared spectroscopy. The metal content was determined by standard methods [7,8], and the C, H and N contents using a Perkin-Elmer model 240 instrument. The results were in strict agreement with the proposed formulation for the adducts. The infrared spectra of the compounds in the 4000–400 cm^{-1} region were obtained on a Perkin-Elmer 283 B recording spectrophotometer using the Nujol mull technique with a polystyrene film as reference. Melting points were determined using a HWS SG2000 apparatus with the samples in sealed capillary tubes. Thermogravimetric curves were obtained in the range 303–1073 K using a Perkin-Elmer TGA 7 thermobalance where a heating rate of 278 K min^{-1} was employed under a dynamic flow of dry nitrogen.

2.3. Calorimetry

All calorimetric determinations were performed in a Parr 1451 Solution Calorimeter adapted to a system of elastic calibration. Fig. 1 shows a scheme of the calorimetric reaction vessel in which a suitable calorimetric solvent (100 cm^3) is held in a glass Dewar, (A), while either a solid or a liquid reactant is held in a sealed glass (B) rotating cell (C) immersed in the calorimetric solvent. The reaction is started by depressing a push rod (D) which drops the contents of the cell into the surrounding liquid and the reaction proceeds to completion under the vigorous stirring action of the rotating cell. Temperature changes by either dissolution or flow of the electrical current through the calibration resistance (E) are sensed by a thermistor (F) and recorded on a strip chart.

2.4. Determination of $\Delta_{vap}H^{\ominus}(BuL)$

The enthalpy of vaporization of BuL was determined from vapour pressure measurements of the liquid sample at several temperatures. Such measurements were performed by distilling BuL under reduced pressure using an appropriate vacuum/nitrogen line.

The value 41.71 ± 0.56 kJ mol^{-1} was obtained from the Clausius–Clapeyron equation, by applying the least-squares method to the points of a straight line (correlation coefficient 0.9989) obtained by plotting log P vs. $1/T$. The slope is a function of the standard enthalpy of vaporization of the liquid.

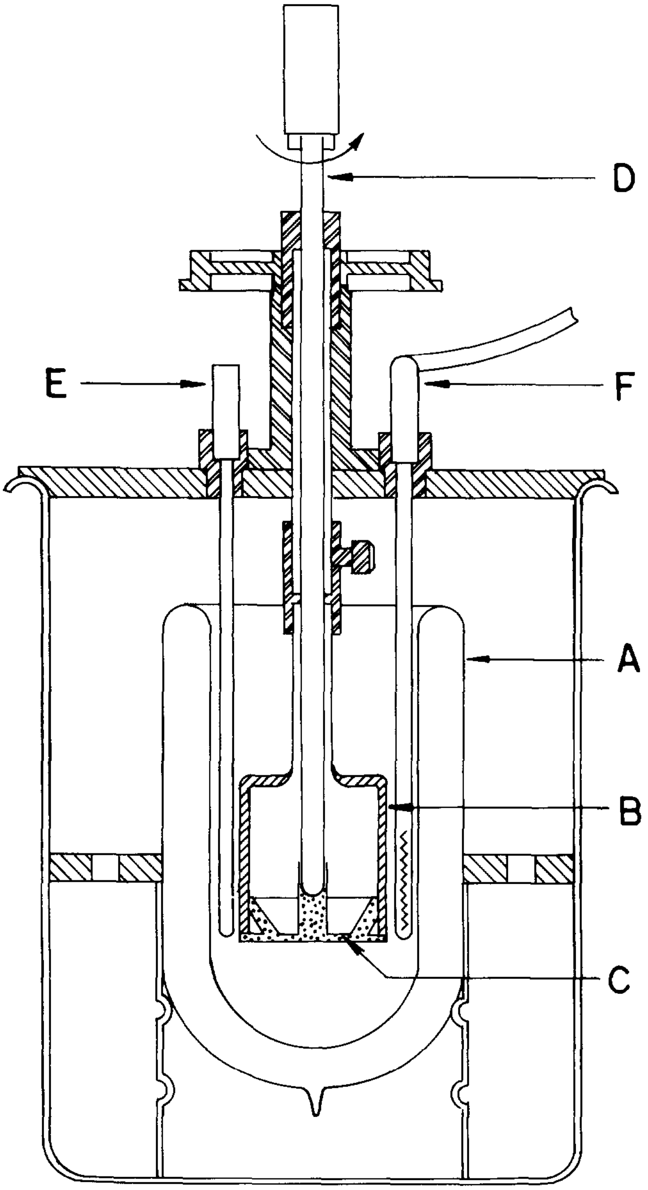

Fig. 1. Scheme of the calorimetric reaction vessel.

3. Results and discussion

The melting points and thermogravimetric determinations (see Table 1) showed that for $ZnCl_2 \cdot 2BuL$ and $ZnI_2 \cdot 2BuL$ adducts, the m.p. results were reproduced after cooling the melted sample and reheating, and the intervals of decomposition

Table 1
Thermochemical, thermogravimetric and m.p. results for $MX_2 \cdot n$BuL adducts, where T_d denotes the beginning of decomposition in thermogravimetric determinations and ΔT is the temperature interval of m.p. and/or decomposition in m.p. determinations

Adduct	$\Delta_i H$ in kJ mol^{-1}			Solvent	$-\Delta_r H_m^\ominus$ in kJ mol^{-1}	T_d in K	ΔT in K
	$\Delta_1 H$	$\Delta_2 H$	$\Delta_3 H$				
$ZnCl_2 \cdot 3$BuL	-43.33 ± 0.27 [a]	$+4.77 \pm 0.02$	$+66.49 \pm 0.19$	Ethanol	105.05 ± 0.33	353	361.7–362.1
$ZnCl_2 \cdot 2$BuL	-43.33 ± 0.27 [a]	$+4.62 \pm 0.11$	$+34.62 \pm 0.36$	Ethanol	73.03 ± 0.38	384	365.1–365.4
$CdCl_2 \cdot$ BuL	-59.98 ± 0.19	$+3.57 \pm 0.05$	-25.60 ± 0.14	[c]	30.72 ± 0.24	418	[d]
$HgCl_2 \cdot$ BuL	$+4.50 \pm 0.04$ [a]	$+4.54 \pm 0.19$	$+37.25 \pm 0.12$	Ethanol	28.21 ± 0.23	355	406.5–407.2
$ZnBr_2 \cdot 3$BuL	-47.69 ± 0.37 [b]	$+4.43 \pm 0.04$	$+57.76 \pm 0.37$	Ethanol	101.02 ± 0.59	355	372.8–373.1
$ZnBr_2 \cdot 2$BuL	-47.69 ± 0.37 [b]	$+4.16 \pm 0.04$	$+27.80 \pm 0.34$	Ethanol	71.73 ± 0.50	391	396.1–396.7
$CdBr_2 \cdot$ BuL	$+15.66 \pm 0.18$	-10.54 ± 0.13	$+44.42 \pm 0.12$	2.5 M HCl	39.30 ± 0.25	405	[d]
$HgBr_2 \cdot$ BuL	$+3.14 \pm 0.03$ [a]	$+4.31 \pm 0.01$	$+32.26 \pm 0.31$	Ethanol	24.81 ± 0.31	350	399.7–400.2
$ZnI_2 \cdot 3$BuL	-52.88 ± 0.35 [a]	$+4.79 \pm 0.02$	$+57.97 \pm 0.17$	Ethanol	106.06 ± 0.39	304	364.9–365.8
$ZnI_2 \cdot 2$BuL	-52.88 ± 0.35 [a]	$+4.14 \pm 0.01$	$+26.66 \pm 0.44$	Ethanol	75.40 ± 0.35	400	366.4–367.1
$CdI_2 \cdot 2$BuL	-15.48 ± 0.19 [b]	$+4.66 \pm 0.01$	$+39.19 \pm 0.16$	Ethanol	50.03 ± 0.25	369	402.1–403.0

[a] Ref. [12]. [b] Ref. [13]. [c] Ethanol + 20% of diethanolamine. [d] Did not melt until 523 K.

found by thermogravimetry occur at temperatures greater than the m.p. detected for both, suggesting that these adducts melt without decomposition. The $CdCl_2 \cdot BuL$ and $CdBr_2 \cdot BuL$ adducts did not melt until 523 K. However, thermogravimetric results showed that the first step of mass loss occurs at a lower temperature than this. The other adducts obtained have higher m.p. than those observed for the beginning of the decomposition process in thermogravimetric determinations. In addition, reproducibility of the m.p. for the previously melted samples was not observed. Thermogravimetric curves for $ZnX_2 \cdot nBuL$ (where X is Cl, Br, I; $n = 2, 3$) adducts showed that the mass loss occurs in two very distinct steps, being best defined for chlorides, then bromides and finally iodides. In the first step the ligand is released, and this is followed by loss of the metal halide. The $CdX_2 \cdot BuL$ (where X is Cl, Br) adducts undergo mass loss in two very well-defined steps, are indicating ligand loss and the other metal halide sublimation. For the $CdI_2 \cdot BuL$ adduct the first step of mass loss indicates the release of one molecule of the ligand together with fragmentation of the second molecule. The second inflection at 603 K indicates the termination of the total loss of two BuL molecules and, at the same time, a halide loss that is complete at approximately 763 K. The mercury adducts lose mass continuously without any perceptible inflection.

The infrared spectra of the adducts showed that the absorption bands due to vibrations of the COHN group were the most affected by a coordination that, as can be observed (Table 2), occurs through the oxygen of BuL. The wavenumber of

Table 2
Wavenumbers (in cm^{-1}) of the absorption band maxima of the CONH group in the infrared spectra of 2-pyrrolidone and its adducts

Compound	ν(NH)	$\Delta\nu$(NH)	ν(CO)	$\Delta\nu$(CO)	ν(CN)	$\Delta\nu$(CN)
BuL	3225		1690		1280	
$ZnCl_2 \cdot 3BuL$	3326	+101	1669	−21	1290	+10
	3164		1645	−45		
$ZnCl_2 \cdot 2BuL$	3290	+65	1660	−30	1298	+10
	3209		1638	−52		
$CdCl_2 \cdot BuL$	3251	+26	1679	−11	1309	+29
$HgCl_2 \cdot BuL$	3309	+84	1661	−29	1286	+06
$ZnBr_2 \cdot 3BuL$	3324	+99	1688	−19	1302	+22
	3145		1656	−34		
$ZnBr_2 \cdot 2BuL$	3326	+101	1667	−23	1290	+10
			1658	−32		
$CdBr_2 \cdot BuL$	3298	+73	1666	−24	1306	+26
$HgBr_2 \cdot BuL$	3247	+22	1674	−16	1292	+12
$ZnI_2 \cdot 3BuL$	3336	+111	1664	−26	1305	+25
	3131		1656	−34		
$ZnI_2 \cdot 2BuL$	3328	+103	1652	−38	1303	+23
			1638	−52		
$CdI_2 \cdot 2BuL$	3311	+86	1653	−37	1296	+16

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CO stretching decreases by about 16–52 cm^{-1} to a value of 1690 cm^{-1} for the free ligand. In the infrared spectra of the zinc adducts the carbonyl bands appear as a doublet in which two absorption maxima are separated by 24–9 cm^{-1}. This splitting was observed for $\text{ZnCl}_2 \cdot n\text{BuL}$ (where $n = 2, 3$), being attributed to the existence of non-equivalent ligands, characterized by different vibrational wavenumbers [10]. The ν(NH) in the adducts is higher than that observed in the spectra of the free ligand, possibly due to the formation of hydrogen bonds between the NH group of BuL and the halide anion. The band at 1280 cm^{-1}, due to ν(CN) [11], shifts to higher wavenumbers after complexation.

The thermochemical data were obtained from calorimetric determinations of the enthalpies of dissolution [12,13]: of the metal halide ($\Delta_1 H$); of the ligand in a solution of the metal halide ($\Delta_2 H$); of the adduct ($\Delta_3 H$) in appropriate solvents.

$$\text{MX}_2(\text{s}) \xrightarrow[\text{solvent}]{\Delta_1 H} \text{solution 1} \tag{1}$$

$$\text{solution 1} + n\text{BuL} \xrightarrow{\Delta_2 H} \text{solution 2} \tag{2}$$

$$\text{MX}_2 \cdot n\text{BuL(s)} \xrightarrow[\text{solvent}]{\Delta_3 H} \text{solution 2} \tag{3}$$

$$\text{MX}_2(\text{s}) + n\text{BuL(l)} \xrightarrow{\Delta_r H^\ominus} \text{MX}_2 \cdot n\text{BuL(s)} \tag{4}$$

The enthalpy of reaction ($\Delta_r H^\ominus$) was obtained by applying Hess' law to the system of thermochemical Eqs. (1), (2) and (3) where $\Delta_r H^\ominus = \Delta_1 H + \Delta_2 H - \Delta_3 H$. The values of the enthalpies of dissolution were obtained by applying the least-squares method [14] to at least five determinations whose mean values are listed in Table 1 together with the results obtained for $\Delta_r H^\ominus$. Using the $\Delta_r H^\ominus$ data and existing literature data [15–19] (see Table 3) $\Delta_f H^\ominus$ and $\Delta_d H^\ominus$ values were determined. $\Delta_{\text{latt}} H^\ominus$ and $\Delta_g H^\ominus$ values were obtained, respectively, for the thermodynamic Eqs. (5) and (6), from which $\Delta_{\text{latt}} H^\ominus = \Delta_r H^\ominus - \Delta_{\text{sub}} H^\ominus(\text{MX}_2) -$

Table 3
Auxiliary thermochemical data (in kJ mol^{-1}) used in $\Delta_f H^\ominus$ and $\Delta_d H^\ominus$. Energetic parameters of the metal halides [24]

Compound	$-\Delta_f H^\ominus$	$\Delta_{\text{sub}} H^\ominus$	E_t	E_i	E_c
BuL	286.2 [a]				
ZnCl_2	415.1 [b,c]	149.0 [c]	328.3	168.1	160.2
CdCl_2	391.5 [b,c]	181.2 [d]	328.7	191.5	137.2
HgCl_2	224.3 [b,c]	83.3 [e]	224.6	150.1	74.4
ZnBr_2	328.7 [b,c]	131.0 [e]	276.0	126.7	149.3
CdBr_2	316.2 [b,c]	151.5 [e]	279.8	150.5	129.2
HgBr_2	170.7 [b,c]	84.1 [d]	188.2	116.7	71.5
ZnI_2	208.0 [b,c]	120.0 [e]	214.5	66.9	146.4
CdI_2	203.3 [b,c]	137.7 [e]	218.7	92.4	127.1

[a] Ref. [15]. [b] Ref. [16]. [c] Ref. [17]. [d] Ref [18]. [e] Ref. [19].

Table 4
Summary of the thermochemical results (in kJ mol^{-1}) for $MX_2 \cdot n$BuL adducts

Adduct	$-\Delta_f H^\ominus$	$\Delta_d H^\ominus$	$-\Delta_{latt} H^\ominus$	$-\Delta_g H^\ominus$	$\bar{D}$(M–O)
$ZnCl_2 \cdot 3BuL$	1379	230	379	337	112
$ZnCl_2 \cdot 2BuL$	1061	157	306	264	132
$CdCl_2 \cdot BuL$	708	72	254	212	212
$HgCl_2 \cdot BuL$	539	70	153	111	111
$ZnBr_2 \cdot 3BuL$	1188	226	357	315	105
$ZnBr_2 \cdot 2BuL$	972	155	286	244	122
$CdBr_2 \cdot BuL$	642	81	232	191	191
$HgBr_2 \cdot BuL$	482	66	151	109	109
$ZnI_2 \cdot 3BuL$	1173	231	351	309	103
$ZnI_2 \cdot 2BuL$	856	159	279	237	118
$CdI_2 \cdot 2BuL$	826	133	271	229	114

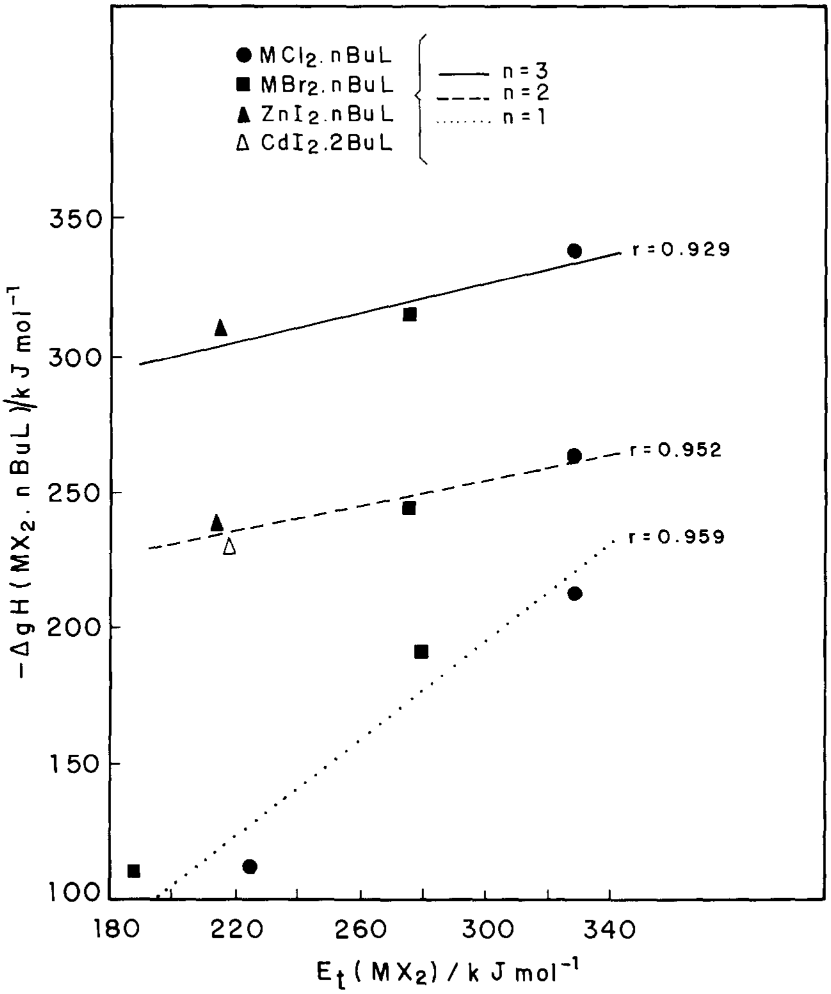

Fig. 2. $\Delta_g H^\ominus(MX_2 \cdot n\text{BuL})$ vs. E_t (MX_2).

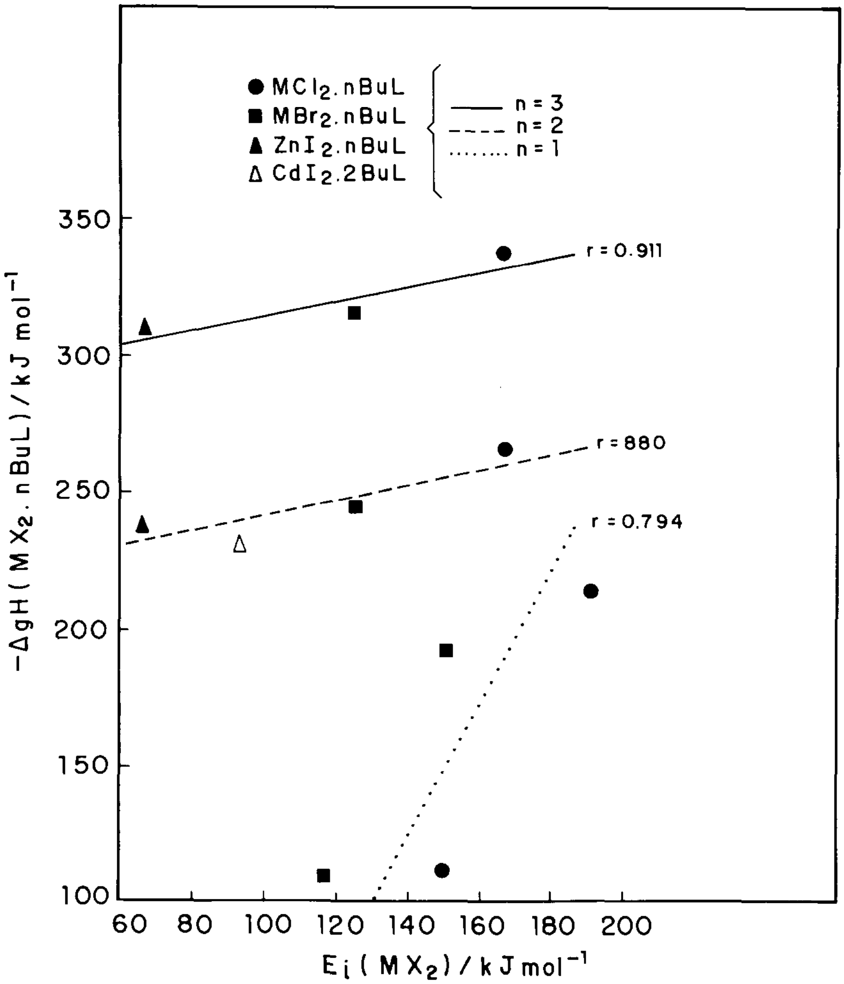

Fig. 3. $\Delta_g H^\ominus(\text{MX}_2 \cdot n\text{BuL})$ vs. E_i (MX_2).

$n(\Delta_{\text{vap}} H^\ominus(\text{BuL}))$ and $\Delta_g H^\ominus = \Delta_{\text{latt}} H^\ominus + \Delta_{\text{sub}} H^\ominus(\text{MX}_2 \cdot n\text{BuL})$ are derived. Because $\Delta_{\text{sub}} H^\ominus(\text{MX}_2 \cdot n\text{BuL})$ is experimentally inaccessible, $\Delta_g H^\ominus$ values were estimated by assuming $\Delta_{\text{sub}} H^\ominus(\text{MX}_2 \cdot n\text{BuL}) \approx \Delta_{\text{vap}} H^\ominus(\text{BuL})$ [20,21].

$$\text{MX}_2(\text{g}) + n\text{BuL}(\text{g}) \xrightarrow{\Delta_{\text{latt}} H^\ominus} MX_2 n\text{BuL}(\text{s}) \qquad (5)$$

$$\text{MX}_2(\text{g}) + n\text{BuL}(\text{g}) \xrightarrow{\Delta_g H^\ominus} \text{MX}_2 n\text{BuL}(\text{g}) \qquad (6)$$

The values of the mean enthalpy of dissociation of the metal–oxygen bond $\bar{D}(\text{M–O})$ were determined from its relation with $\Delta_g H^\ominus$ according to

$$D(\text{M–O}) = -\Delta_g H^\ominus / n \qquad (7)$$

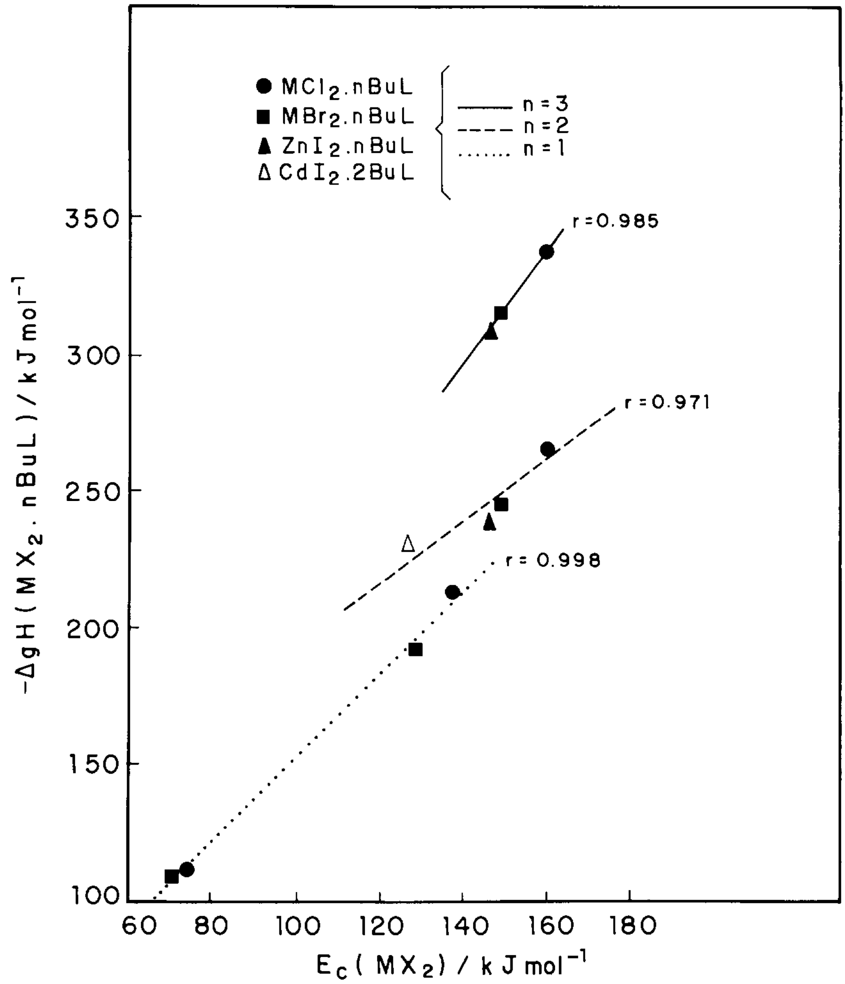

Fig. 4. $\Delta_g H^{\ominus}(MX_2 \cdot n\mathrm{BuL})$ vs. E_c (MX_2).

The $\Delta_r H^{\ominus}$ parameter has been considered in the evaluation of the relative basicity of the ligands and of the relative acidity of the metal halides [22]. In this work the adducts obtained do not present the same stoichiometry throughout the halides series. However, comparison of the $\Delta_r H^{\ominus}(MX_2 \cdot n\mathrm{BuL})$ values is made by presuming that substitution of similar ligands in adducts of the same metal halide does not alter the coordination around the central atom. In this case, it is observed that the relative acidity of the metal halides with relation to BuL obeys the sequence Zn > Cd > Hg. The values of $\Delta_f H^{\ominus}$ have been considered in comparison with the thermodynamic stability of the adducts [23]. The $\Delta_f H^{\ominus}(MX_2 \cdot n\mathrm{BuL})$ values show that the thermodynamic stability follows the sequence $ZnX_2 \cdot n\mathrm{BuL} > CdX_2 \cdot n\mathrm{BuL} > HgX_2 \cdot n\mathrm{BuL}$ (where X is Cl, Br, I).

Excluding $\bar{D}$(M–O) values, the remaining thermodynamic quantities (Table 4) show that the intensity of the acid–base interaction decreases from zinc to mercury, establishing the following sequence of donor–acceptor interactions, when the metal is fixed and only the halide is varied: Cl > Br > I.

Correlation of $\Delta_g H^\ominus$ values with the total bond energy (E_t), ionic energy (E_i) and covalent energy (E_c) of the metal halides, whose values were obtained by Sanderson [24] (see Table 3) were examined. The corresponding graphs are shown in Figs. 2–4 with the respective correlation coefficients (r) for each series of points distinguishing adducts of the same stoichiometry. On examining these graphs, it can be seen that the covalent energy of the metal halides is better correlated with $\Delta_g H^\ominus$, possibly suggesting a greater influence of the covalent character in the energetic contribution of the metal halides to the strength of metal–ligand interactions in these adducts.

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