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Thermochemistry of Quinoline and Morpholine Adducts of Cadmium(II) Halides

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The adducts $[CdX_2(L)_n]$, where X = Cl, Br, or I; L = quinoline (quin) or morpholine (morph); and $n = 1$, 1.5, or 2, were synthesized and characterized by melting point and elemental analysis, thermal studies, and IR spectroscopy. From calorimetric studies in solution, the standard enthalpies of formation of the adducts and several thermodynamic parameters were determined. The mean standard enthalpies of the cadmium−nitrogen bonds were calculated.

Introduction

The molecules of morpholine have two donor atoms: nitrogen and oxygen. It is known to form complexes with transition metal(II) halides.[1,2] Quinoline is a monodentate ligand and also forms complexes with transition metal(II) halides.[3−15]

The main purpose of the present article is the calorimetric determination of the energy involved in the formation of the coordinated cadmium(II)−nitrogen bonds as well as the determination of several thermochemical parameters for the adducts formed between cadmium(II) halides with morpholine or quinoline. These data are not available in the literature. Knowledge of the themochemical properties of these compounds helps the understanding of the coordinated metal(II)−nitrogen bonds formed. The characterization of the thermodynamic properties of these compounds could be used in determining their potential applications in catalysis and also in the chromatographic separation of the metal ions.

Experimental Section

Chemicals. Morpholine (Aldrich, > 99 %) and quinoline (Aldrich, 98 %) were purified by distillation using an efficient column and stored over 4 Å molecular sieves (bp obtained at (123 to 124) °C and (111 to 112) °C [(0.26 to 2.66) kPa], respectively). Cadmium(II) chloride and cadmium(II) bromide were synthesized as described in the literature.[16] Cadmium(II) iodide (Aldrich, 99 %) was purified according to Simoni et al.[17]

Adducts Synthesis. The adducts were prepared by the reaction of metal(II) bromides and the ligands in hot ethanol with a molar relation of 1:4. A typical procedure is given below.

Cadmium(II) Bromide−Morpholine. To a solution of 1.0 g of $CdBr_2$, 3.67 mmol, in 100 mL of hot ethanol was added 1.30 mL of morpholine, 14.69 mmol, slowly and dropwise with stirring. The stirring was maintained over several hours. The solid formed was filtered and washed with petroleum ether. The product was dried for several hours in a vacuum and stored in a desiccator over calcium chloride. The purity of the adducts was (99.5 to 99.0) % as shown by chemical analysis.

Analytical Section. Carbon, hydrogen, and nitrogen were determined by microanalytical procedures.[18] Bromine analysis was made by gravimetry using standard N/10 $AgNO_3$ solution after the adducts were dissolved in water.[19]

Spectra. Infrared spectra were obtained using samples of the adducts in a KBr matrix and the IR spectra of the ligands by using a liquid cell with NaCl windows, in the region (4000 to 400) cm^{-1} using a Perkin-Elmer 1600 series FTIR spectrophotometer.

Thermal Studies. The TG/DTG and DSC measurements were obtained in an argon atmosphere in a Du Pont 951 analyzer with samples varying in mass from (4.41 to 6.17) mg (TG/DTG) and from (1.80 to 6.29) mg (DSC) and a heating rate of 10 $K\cdot min^{-1}$ in the (298 to 678) K (DSC) and (298 to 1243) K (TG/DTG) temperatures ranges.

Calorimetric Measurements. All solution calorimetric measurements were carried out in an LKB 8700-1 precision calorimeter, by the ampule-broken method, as previously described.[20] The accuracy of the calorimeter was carried out by determining the heat of dissolution of tris(hydroxymethylamino) methane in 0.1 $mol\cdot dm^{-3}$ hydrochloric acid. The result $(-29.78 \pm 0.03)\ kJ\cdot mol^{-1}$ is in agreement with the value recommended by IUPAC, $(-29.763 \pm 0.003)\ kJ\cdot mol^{-1}$.[21] The uncertainty intervals are twice the standard deviations of the means of about 4 to 6 replicate measurements on each compound.

Results and Discussion

All adducts were solids. The yields range from (70 to 88) %. The yields, capillary melting points, colors, appearance, and analytical data are summarized in Table 1.

Infrared Spectra. The pattern of the infrared spectra of the morpholine adducts is similar to that of the free ligand, although dislocation and splitting of some bands are observed. Considerable shifts to lower frequencies of the N−H stretching modes of the coordinated morpholine are observed. This indicates the coordination of morpholine to the cadmium ion.[22−25] The positive shift of the C−O−C stretching vibration mode that is found at 1097 cm^{-1} in the free morpholine excludes the possibility of oxygen-to-cadmium ion coordination.[23] In the H−N−C deformation region, a great change is observed that affords evidence of the coordination of the N atom of morpholine.[26,27] The IR spectra of quinoline adducts show shifts of several bands with respect to the free ligand after coordination. The IR can be interpreted in terms of the coordination of

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Table 1. Melting Points, Yields on Preparation, Appearance, and Analytical Data of the Adducts

compound	yield (%)	MP[a] (K)	apearance[c]	% C calcd	% C found	% H calcd	% H found	% N calcd	% N found	% X calcd	% X found	% Cd calcd	% Cd found
$[CdCl_2(morph)_{1.5}]$	86	608 to 610[a]	wh. pw.	22.95	23.05	4.33	4.28	6.69	6.71	22.58	22.38	35.80	35.77
$[CdBr_2(morph)_2]$	79	568 to 570[a]	wh. crys.	21.52	21.56	4.06	3.99	6.27	6.34	35.80	35.77	25.18	25.12
$[CdI_2(morph)_2]$	75	478 to 480[a]	l. ye. pw.	17.78	17.90	3.36	3.35	5.18	5.25	46.96	46.85	20.80	20.70
$[CdCl_2(quin)]$	88	623[b]	wh. pw.	34.60	34.85	2.26	3.22	4.48	4.37	22.69	22.50	35.97	35.94
$[CdBr_2(quin)]$	75	623[b]	wh. pw.	26.93	26.90	1.76	1.60	3.49	3.54	39.82	39.61	28.00	27.85
$[CdI_2(quin)_2]$	70	623[b]	wh. pw.	34.62	34.77	2.26	2.19	4.49	4.55	40.64	40.44	18.00	18.05

[a] Melting point with decomposition. [b] Not melting until 623 K. [c] Key: l., light; wh., white; ye., yellow; pw., powder; crys., crystals.

Table 2. Main IR Spectral Data (cm^{-1}) of the Compounds

compound	assignment[a] $\nu_{(N-H)}$	$\nu_{(C-C)}$	$\nu_{(C-O-C)}$	ring	$\delta_{(N-H-C)}$	$\phi_{(C-C)}$
morph	3320m, 2947s	1452s	1097s		889m, 835s	
$[CdCl_2(morph)_{1.5}]$	3224m	1447m	1113m		877vs	
$[CdBr_2(morph)_2]$	3271m	1434m	1414s		873vs	
$[CdI_2(morph)_2]$	3216m	1440m	1111s		874vs	
quin		1596s		103m		n. o.
$[CdCl_2(quin)]$		1584m		953m		776s
$[CdBr_2(quin)]$		1581m		954m		773s
$[CdI_2(quin)_2]$		1585m		950m		780s

[a] Key: ν, stretching; ring, ring breathing; δ, angular deformation; ϕ, ring deformation out of plane. Intensity of bands: vs, very strong; s, strong; m, medium; n.o., not observed.

Table 4. Enthalpies of Dissolution at 298.15 K

compound	calorimetric solvent	number of experiments	i	$\Delta_i H^\theta$ ($kJ \cdot mol^{-1}$)
$CdCl_{2(s)}$	HCl 4.2 M	5	1	4.02 ± 0.11
$morph_{(l)}$	1.5:1 $CdCl_2$−HCl 4.2 M	4	2	-112.76 ± 2.80
$[CdCl_2(morph)_{1.5}]$	HCl 4.2 M	4	3	-30.61 ± 0.49
$CdBr_{2(s)}$	HCl 4.2 M	5	1	13.15 ± 0.36
$morph_{(l)}$	2:1 $CdBr_2$−HCl 4.2 M	5	2	-129.11 ± 1.09
$[CdBr_2(morph)_2]_{(s)}$	HCl 4.2 M	5	3	-25.19 ± 0.83
$CdI_{2(s)}$	HCl 4.2 M	6	1	26.45 ± 0.71
$morph_{(l)}$	2:1 CdI_2−HCl 4.2 M	4	2	-152.67 ± 2.06
$[CdI_2(morph)_2]_{(s)}$	HCl 4.2 M	5	3	-16.41 ± 0.04
$quin_{(l)}$	1:1 $CdCl_2$−HCl 4.2 M	5	2	-40.79 ± 1.16
$[CdCl_2(quin)]_{(s)}$	HCl 4.2 M	5	3	-10.85 ± 0.22
$quin_{(l)}$	1:1 $CdBr_2$−HCl 4.2 M	5	2	-39.76 ± 1.04
$[CdBr_2(quin)]_{(s)}$	HCl 4.2 M	4	3	21.97 ± 0.25
$quin_{(l)}$	2:1 CdI_2−HCl 4.2 M	5	2	-67.84 ± 3.06
$[CdI_2(quin)_2]_{(s)}$	HCl 4.2 M	5	3	24.17 ± 0.26

quinoline through its nitrogen atom.[15] Table 2 presents the extracted infrared spectral data of the adducts.

Thermal Studies. The thermogravimetry of the adducts shows the loss of the ligand in one or two steps of mass loss followed by the loss of the halide and part of the metal in the second or third step of mass loss. A residue is left that is part of the cadmium content. The DSC curves are consistent with the TG data. They present endothermic peaks due to the elimination of ligand or to melting followed by elimination of the ligand. Table 3 presents the thermoanalytical data of the adducts.

Calorimetric Measurements. The standard enthalpies of dissolution of cadmium(II) halides, ligands, and adducts were obtained as previously reported.[20] The standard enthalpies of dissolution were obtained according to the standard enthalpies of the following reactions in solution

$$CdX_{2(s)} + \text{calorimetric solvent} \rightarrow \text{solution A}; \qquad \Delta_1 H^\theta \tag{1}$$

$$nL_{(1)} + \text{solution A} \rightarrow \text{solution B}; \qquad \Delta_2 H^\theta \tag{2}$$

$$[CdX_2(L)_n]_{(s)} + \text{calorimetric solvent} \rightarrow \text{solution C}; \qquad \Delta_3 H^\theta \tag{3}$$

$$\text{solution B} \rightarrow \text{solution C}; \qquad \Delta_4 H^\theta \tag{4}$$

Table 3. Thermal Analysis of the Compounds

compound	apparent melting point (K)	mass loss (%) calcd	mass loss (%) obsd	TG temperature range (K)	species lost	DSC peak temperature	ΔH^θ ($kJ \cdot mol^{-1}$)
$[CdCl_2(morph)_{1.5}]$	608 to 610	27.75	26.68	288 to 479	- L	499	34.85
		13.88	15.51	479 to 508	- 0.5 L	526	79.29
		51.22	51.75	817 to 859	- 2 Cl − 0.8 Cd		
			6.06[a]				
$[CdBr_2(morph)_2]$	568 to 570	19.52	19.34	399 to 461	- L	458	78.67
		19.52	18.99	461 to 484	- L	555	21.45
		58.46	58.99	758 to 811	- 2 Br − 0.9 Cd		
			2.68[a]				
$[CdI_2(morph)_2]$	478 to 480	16.12	16.54	398 to 423	- L	483	42.80
		16.12	15.26	423 to 475	- L	504	70.15
		63.60	64.26	685 to 746	- 2 I − 0.8 Cd		
			3.94[a]				
$[CdCl_2(quin)]$	n.o.	41.34	41.48	378 to 533	- L	536	29.97
		51.47	50.82	802 to 856	- 2 Cl − 0.8 Cd		
			7.70[a]				
$[CdBr_2(quin)]$	n.o.	32.18	30.63	470 to 506	- L	350	1.25
		62.22	64.38	764 to 824	- 2 Br − 0.8 Cd	442	32.38
			4.99[a]				
$[CdI_2(quin)_2]$	471 to 473	20.68	22.23	406 to 432	- L	463	118.71
		20.68	20.26	432 to 487	- L		
		56.84	55.82	682 to 749	- 2 I − 0.9 Cd		
			1.69[a]				

[a] Residue at 1243 K. Key: n.o., not observed.

Table 5. Summary of the Thermochemical Results ($kJ \cdot mol^{-1}$) for the Compounds

compound	$\Delta_r H^\theta$	$\Delta_f H^\theta$	$\Delta_{s,l}{}^g H^\theta$	$\Delta_M H^\theta$	$\Delta_D H^\theta$	$\Delta_r H^\theta(g)$	$\bar{D}_{(Cd-N)}$
$CdCl_{2(s)}$		-391.5^a	181.2^b				
$CdBr_{2(s)}$		-316.2^a	151.2^c				
$CdI_{2(s)}$		-203.3^a	137.7^c				
$morph_{(l)}$		-170.2 ± 1.0^d	2.3 ± 1.0^d				
$quin_{(l)}$		141.22 ± 0.92^e	59.32 ± 0.20^e				
$[CdCl_2(morph)_{1.5}]_{(s)}$	-78.13 ± 2.84	-724.9 ± 3.6	$201 \pm 11^{f,g}$	-322.8 ± 3.4	141.6 ± 3.2	-122 ± 12	81 ± 8
$[CdBr_2(morph)_2]_{(s)}$	-90.77 ± 1.41	-747.4	$171 \pm 11^{f,g}$	-326.6 ± 2.6	175.4 ± 2.4	-155.5 ± 11.3	78.0 ± 5.7
$[CdI_2(morph)_2]_{(s)}$	-109.81 ± 2.18	-653.5 ± 3.1	$158 \pm 11^{f,g}$	-332.1 ± 3.6	194.4 ± 3.0	-174 ± 12	87 ± 6
$[CdCl_2(quin)]_{(s)}$	-25.92 ± 1.11	-276.2 ± 2.5	$209 \pm 11^{f,g}$	-266.4 ± 1.5	85.24 ± 1.13	-57 ± 11	57 ± 11
$[CdBr_2(quin)]_{(s)}$	-48.58 ± 1.13	-223.6 ± 1.8	$179 \pm 11^{f,g}$	-259.1 ± 1.5	107.90 ± 1.15	-80 ± 11	80 ± 11
$[CdI_2(quin)_2]_{(s)}$	-65.56 ± 3.15	13.6 ± 3.8	$166 \pm 11^{f,g}$	-321.9 ± 3.8	184.20 ± 3.18	-156 ± 12	78 ± 6

[a] Ref 28. [b] Ref 29. [c] Ref 30. [d] Ref 31. [e] Ref 32. [f] Ref 33. [g] Ref 34.

Table 6. Auxiliary Data and Enthalpy Changes of the Ionic Complex Formation Process in the Gaseous Phase ($kJ \cdot mol^{-1}$)

compound	$\Delta_f H^\theta$	$\Delta_r H^\theta$	$\Delta_{fI} H^\theta$
$Cl^-{}_{(g)}$	-233.13^a		
$Br^-{}_{(g)}$	-219.07^a		
$I^-{}_{(g)}$	-197^a		
$Cd^{2+}{}_{(g)}$	2623.54^a		
$[CdCl_2(morph)_{1.5}]_{(g)}$	-682.7 ± 4.2	-122 ± 12	-2873 ± 13
$[CdBr_2(morph)_2]_{(g)}$	-576.7 ± 11.3	-155.5 ± 11.3	-2506 ± 12
$[CdI_2(morph)_2]_{(g)}$	-482 ± 12	-174 ± 12	-2456 ± 12
$[CdCl_2(quin)]_{(g)}$	-67 ± 11	-57 ± 11	-2425 ± 11
$[CdBr_2(quin)]_{(g)}$	-14 ± 11	-80 ± 11	-2400 ± 11
$[CdI_2(quin)_2]_{(g)}$	180 ± 12	-156 ± 12	-2451 ± 13

[a] Ref 28.

The application of the Hess law to the series of reactions (1 to 4) gives the standard enthalpies of acid/base reactions ($\Delta_r H^\theta$) according to the reaction

$$CdX_{2(s)} + nL_{(1)} \rightarrow [CdX_2(L)_n]_{(s)}; \qquad \Delta_r H^\theta \qquad (5)$$

where $\Delta_r H^\theta = \Delta_1 H^\theta + \Delta_2 H^\theta - \Delta_3 H^\theta$ because the final thermodynamic state of reaction 2 and 3 is the same and $\Delta_4 H^\theta = 0$, as is also observed for the dilution of solution B into solution C. Table 4 gives the values obtained for the enthalpies of dissolution of CdX_2 ($\Delta_1 H^\theta$), ligand in the solution of CdX_2 ($\Delta_2 H^\theta$), and the adducts ($\Delta_3 H^\theta$). Uncertainty intervals given in this table are twice the standard deviation of the means of four to six replicate measurements.

From the values obtained for the standard enthalpies of reaction 5 ($\Delta_r H^\theta$) and by using appropriate thermochemical cycles,[20] the following thermochemical parameters for the adducts were determined: 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 standard enthalpies of the reaction in the gaseous phase ($\Delta_r H^\theta(g)$). The standard enthalpies of the cadmium(II)−nitrogen bonds are equal to $\bar{D}_{(Cd-N)} = -(\Delta_r H^\theta(g))/n$. Table 5 lists the values obtained for these enthalpies. On the basis of the $\Delta_r H^\theta$ values, the acidity order of the salts, for the adducts of the same stoichiometry and the same ligand, can be established: $CdI_2 > CdBr_2$ (morph) and $CdBr_2 > CdCl_2$ (quin). Using the $D_{(Cd-N)}$ values, the order obtained is the same: $CdI_2 > CdBr_2$ (morph) and $CdBr_2 > CdCl_2$ (quin). The enthalpies for the process of a hypothetical complex formation in the gaseous phase, from cadmium(II) ions, halide ions, and ligand molecules, can be evaluated

$$Cd^{2+}{}_{(g)} + 2X^-{}_{(g)} + nL_{(g)} \rightarrow [CdX_2(L)]_{(g)}; \qquad \Delta_{fI} H^\theta \qquad (6)$$

where $\Delta_{fI} H^\theta = \Delta_f H^\theta(adduct_{(g)}) - \Delta_f H^\theta(Cd^{2+}{}_{(g)}) - 2\Delta_f H^\theta(X^-{}_{(g)}) - n\Delta_f H^\theta(L_{(g)})$.

Table 6 lists the values obtained for these enthalpies values.

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