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Thermochemical studies of two copper(II) complexes with *N*-benzoyl-*N*′,*N*′-dialkylurea derivatives

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Abstract

The standard ($p^0 = 0.1$ MPa) molar enthalpies of formation of crystalline bis(*N*-benzoyl-*N*′,*N*′-diethylureato)copper(II), $Cu(BETU)_2$, and bis(*N*-benzoyl-*N*′,*N*′-diisobutylureato)copper(II), $Cu(BIBU)_2$, were measured, at $T = 298.15$ K, by solution–reaction isoperibol calorimetry, respectively, as $-(833.7 \pm 9.7)$ and $-(1050.6 \pm 12.1)$ kJ mol^{-1}. The standard molar enthalpy of sublimation, at $T = 298.15$ K, of $Cu(BETU)_2$ was determined by a Knudsen effusion technique as (180 ± 37) kJ mol^{-1}. These values were used to derive the standard molar enthalpy of formation of $Cu(BETU)_2$ in gaseous phase, and to evaluate the mean metal–ligand dissociation enthalpy of the complex relative to that of the ligand with hydrogen. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Bis(*N*-benzoyl-*N*′,*N*′-diethylureato)copper(II); Bis(*N*-benzoyl-*N*′,*N*′-diisobutylureato)copper(II); Enthalpy of formation; Enthalpy of sublimation; Dissociation enthalpy

1. Introduction

The relative simple synthesis of acylcalcogenourea derivatives from readily available starting materials allows for the preparation of a wide variety of ligands with interesting properties. Several of these compounds have been found to be useful ligands for the potential determination of traces of the transition metals, by means of normal phase chromatography [1], and have been shown to selectively extract metals in the form of stable neutral metal chelates [2]. Although the important applications of these apparently simple complexes, a survey of the literature show that very little have been published concerning the chemistry of them. Remarkably, little is known about the energetics of the metal–ligand binding in these metal chelates, apart the reported titration calorimetric studies by Dietze et al. [3], on the complex formation of *N*-benzoyl-chalcogeno(O, S, Se)ureas with metal ions of the fourth to sixth periods of the periodic table of elements.

The knowledge of the energetics of the metal–ligand bond formation is of great for a deeper understanding of a wide range of chemical processes. In order to contribute to the knowledge of the metal–ligand binding on these kind of quelates, we started a systematic thermochemical study of calcogenourea derivatives [4] and its metallic complexes, involving O_4 or O_2S_2 donor atoms sets, in view to compare the

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influence of such boundary on the energetics of the binding. The synthesis of acylurea derivatives has been successful only for two compounds, *N*-benzoyl-*N′*,*N′*-diethylurea, PhCONHCON(Et)$_2$, HBETU, and *N*-benzoyl-*N′*,*N′*-diisobutylurea, PhCONHCON(iBu)$_2$, HBIBU, which have been used to prepare the corresponding copper(II) complexes.

This paper presents the experimental determination of the standard molar enthalpies of formation, at $T = 298.15$ K, of the bis(*N*-benzoyl-*N′*,*N′*-diethylureato)copper(II), Cu(BETU)$_2$, and bis(*N*-benzoyl-*N′*,*N′*-diisobutylureato)copper(II), Cu(BIBU)$_2$, complexes in the crystalline state. The standard molar enthalpy of sublimation of Cu(BETU)$_2$ has also been measured by a Knudsen effusion method. The experimental results are used to derive the dissociation enthalpies of the ligands to the copper(II) relative to the binding to hydrogen, i.e. the difference $\langle D\rangle(\mathrm{Cu-L}) - D(\mathrm{H-L})$.

2. Experimental

2.1. Synthesis and characterisation of compounds

The ligands *N*-benzoyl-*N′*,*N′*-diethylurea, HBETU, and *N*-benzoyl-*N′*,*N′*-diisobutylurea, HBIBU, were prepared accordingly to the method of Herzschuh et al. [5], as it has been described in a previous paper [4].

The complexes Cu[PhCONHCON(Et)$_2$]$_2$, Cu(BETU)$_2$, and Cu[PhCONHCON(^{i}Bu)$_2$]$_2$, Cu(BIBU)$_2$, were prepared as follows.

The synthesis of Cu(BETU)$_2$ complex was carried as previously described [5]. A solution of 2.0 g (0.01 mol) of monohydrated copper acetate in 80 cm^3 of methanol–water mixture (3:1), was added to a solution of 4.4 g (0.02 mol) of ligand in methanol (30 cm^3) and then mixed with 0.02 mol of NH$_3$ from a 1 mol dm^{-3} solution. The mixture was stirred and the complex was precipitated by addition of water and allowed to rest. The needles obtained were washed with water and dried in the desiccator. The light blue powder was recristallised from xylol, and presents a melting point of 143–144°C.

Cu(BIBU)$_2$, was synthesised in the same way as the former complex, and the light blue powder, after recrystallisation from ethanol presented a melting point of 127–129°C.

The purity of the samples was checked by IR spectroscopy and by elemental analysis; the mass fractions w of C, H and N were as follows: for Cu(BETU)$_2$, CuC$_{24}$H$_{30}$N$_4$O$_4$, found $10^2\ w(\mathrm{C}) = 57.27$, $10^2\ w(\mathrm{H}) = 5.67$, $10^2\ w(\mathrm{N}) = 10.86$, calculated $10^2\ w(\mathrm{C}) = 57.45$, $10^2\ w(\mathrm{H}) = 5.98$, $10^2\ w(\mathrm{N}) = 11.16$; for Cu(BIBU)$_2$, CuC$_{32}H_{46}N_4O_4$, found $10^2\ w(\mathrm{C}) = 62.38$, $10^2\ w(\mathrm{H}) = 7.04$, $10^2\ w(\mathrm{N}) = 9.34$, calculated $10^2\ w(\mathrm{C}) = 62.57$, $10^2\ w(\mathrm{H}) = 7.55$, $10^2\ w(\mathrm{N}) = 9.12$.

2.2. Solution–reaction calorimetry

The solution and reaction enthalpies were measured in a Dewar isoperibol solution–reaction calorimeter, whose basic auxiliary equipment and technique used were previously described [6,7]. This calorimeter has been equipped with a newly designed all-glass reaction vessel [8], containing 120.0 cm^3 of solvent, equipped with a twin-bladed stirrer and an ampoule holder that allows the introduction of new ampoules into the reaction vessel without having to take the reaction vessel out of the thermostatic bath. The vessel was submerged in a water thermostatic bath maintained at $T = (298.150 \pm 0.001)$ K by a TRONAC PTC-41 controller. The temperatures of the calorimeter were measured with a quartz crystal thermometer (Hewlett-Packard HP 2804-A) to 10^{-4} K, every 10 s; the thermometer was interfaced to a PC using the program LABTERMO [8] to control the system. The adiabatic temperature changes were calculated using the equal area method [9]. The calorimeter was calibrated electrically for each experiment. The accuracy and performance of the calorimetric system were tested by measuring the molar enthalpy of solution of THAM, tris(hydroxymethyl)aminomethane (BDH, Thermochemical Standard) in 0.100 mol dm^{-3} HCl (aq) at $T = 298.15$ K, with six independent measurements giving $\Delta_r H_m^0 = -(29.763 \pm 0.016)$ kJ mol^{-1}, in good agreement with the value reported by Kilday et al. [10], $-(29.770 \pm 0.032)$ kJ mol^{-1}.

The thermochemical reaction (1) was used for determining the enthalpies of formation of both CuL$_2$ complexes.

$$\begin{aligned}&48.64\,\mathrm{H_2O(l)} + \mathrm{Cu(CH_3COO)_2\cdot H_2O(cr)} + 2\mathrm{HL(cr)}\\&\quad\rightarrow 2\mathrm{CH_3COOH}\cdot 24.82\mathrm{H_2O(l)} + \mathrm{CuL_2(cr)}\end{aligned}\qquad(1)$$

The reactions represented by Eq. (1) were carried out in a solution of HCl 4.202 mol dm^{-3} and 1,4-dioxan mixture (1:3, v/v) as calorimetric solvent (120 cm^3). Ampoules containing the reactants were added consequently to the solvent in the order they are written in the above equation and the corresponding $\Delta_i H_m$ values were measured. To a second portion of the same solvent were added, also consequently, ampoules containing the products in the same order they are written in Eq. (1), and the corresponding $\Delta_i H_m$ values were measured. Rigorous control of the stoichiometry was maintained through each series of experiments to ensure that the final solutions resulting from dissolution and reactions of the reactants were of the same composition of those from the dissolution and reaction of the products. This was tested by braking ampoules of the final solution resulting from the dissolution of the reactants, into the final solution of the dissolution of all the products in the calorimeter, and no enthalpy change being detected.

2.3. Enthalpies of sublimation

The standard molar enthalpies of sublimation Cu(BETU)$_2$ was measured by the Knudsen effusion method using the apparatus as described by Burkinshaw and Mortimer [11], with the detailed modifications previously reported [12]. The equipment was tested with several compounds of known standard molar enthalpies of sublimation (benzanthrone, squaric acid, and 4-hydroxy-2-methylquinoline) and good agreement was obtained. The vapour effusing from the Knudsen cell was allowed to condense on a quartz crystal positioned above the effusion hole. The changes in the frequency of oscillation Δf of the quartz crystal are directly proportional [13] to the mass of substance, Δm, deposited in its surface.

$$\Delta f = C_f \Delta m \tag{2}$$

where C_f is a proportionally constant. The experimental measured rate of resonance frequency change, v; in the time t, for the quartz crystal, $v = \Delta f / t$, is proportional to the rate of sublimed mass of the crystalline sample, $\Delta m/t$

$$v = C_f \frac{\Delta m}{t} \tag{3}$$

which can be related to the vapour pressure, p, according to the Knudsen equation

$$p = \left(\frac{\Delta m}{t}\right) a^{-1} \left(\frac{2\pi RT}{M}\right)^{1/2} \tag{4}$$

where ($\Delta m/\mathrm{d}t$) is the rate of mass loss, a the effective hole area, and M is the molar mass of the effusing vapour. From Eqs. (3) and (4),

$$p = vT^{1/2} \left[\frac{(2\pi RT/M)^{1/2}}{aC_f}\right] \tag{5}$$

and, therefore, according to the equation of Clausius–Clapeyron and Eq. (5),

$$\ln p = -\left(\frac{a}{T}\right) + b \tag{6}$$

a plot of ln ($vT^{1/2}$) against $1/T$ should gives a straight line of slope equal to $\Delta_{cr}^{g} H_m^0(\langle T \rangle)/R$. From six independent sets of experimental measurements of the frequency of the quartz oscillator at convenient temperature intervals, six independent results for the enthalpy of sublimation of the compound, referred to the mean temperature $\langle T \rangle$ of each experimental range, were obtained.

3. Results

Table 1 lists the average values of at least six independent measurements of the molar enthalpies of solution and reaction for each chemical species used on the solution calorimetry of both copper(II) complexes from which the standard molar enthalpies of reaction, $\Delta_r H_m(\mathrm{CuL_2})$, were derived by means of Eq. (7). The uncertainties given are twice the standard deviation of the mean, in accordance with normal thermochemical practice.

$$\Delta_r H_m(\mathrm{CuL_2}) = 48.64\Delta_1 H_m + \Delta_2 H_m + 2\Delta_3 H_m - 2\Delta_4 H_m - \Delta_5 H_m \tag{7}$$

Table 2 lists the standard molar enthalpy of the reaction (1), calculated by means of Eq. (7), in which $\Delta_i H_m$ are the molar enthalpies of solution and reaction of each chemical species, as defined in Table 1. The standard molar enthalpies of formation of the complexes in the crystalline state, also listed in Table 2, were derived by means of the following auxiliary

Table 1
Solution calorimetric results for the copper(II) complexes of both ligands, $PhCONHCONEt_2$ (HBETU) and $PhCONHCON^iBut_2$ (HBIBU)

I	Reactant	Solvent	Solution formed	$\Delta_i H_m$/(kJ mol^{-1})
1	H_2O	$HCl\cdot 12.61H_2O$/1,4-dioxan (1:3)	A_1	$-(1.005 \pm 0.014)$
2	$Cu(CH_3COO)_2\cdot H_2O$	A_1	A_2	15.72 ± 0.67
3	HBETU	A_2	A_3	24.70 ± 0.18
3a	HBIBU	A_2	A_4	25.99 ± 0.23
4	CH_3COOH in $24.82H_2O$	$HCl\cdot 12.61H_2O$/1,4-dioxan (1:3)	B_1	$-(25.43 \pm 0.41)$
5	$Cu(BETU)_2$	B_1	B_2	9.47 ± 0.18
5a	$Cu(BIBU)_2$	B_1	B_3	21.77 ± 0.56
6	Solution A_3	B_2	B_2	0.00
6a	Solution A_4	B_3	B_3	0.00

Table 2
Derived standard molar enthalpy values (kJ mol^{-1}) at $T = 298.15$ K

Compound	$\Delta_r H^0_m$	$\Delta_f H^0_m$(cr)	$\Delta^g_{cr} H^0_m$	$\Delta_f H^0_m$(g)
$Cu(BETU)_2$	57.6 ± 1.3	$-(833.7 \pm 9.7)$	180.9 ± 3.7	$-(652.8 \pm 10.4)$
$Cu(BIBU)_2$	47.9 ± 1.4	$-(1050.6 \pm 12.1)$	$(181 \pm 10)^a$	$-(870 \pm 16)^a$

[a] Values were estimated.

quantities: $\Delta_f H^0_m(H_2O, l)/(kJ\,mol^{-1}) = -(285.83 \pm 0.04)$ [14]; $\Delta_f H^0_m$ (CH_3COOH *in* 24.82 H_2O, l)/(kJ mol^{-1}) $= -(485.156 \pm 1.10)$ [14]; $\Delta_f H^0_m$ ($Cu(CH_3COO)_2\cdot H_2O$, cr)/(kJ mol^{-1}) $= -(1189.1 \pm 0.5)$ [14]; $\Delta_f H^0_m$ (HBETU, cr)/(kJ mol^{-1}) $= -(479.2 \pm 4.8)$ [4]; $\Delta_f H^0_m$ (HBIBU, cr)/(kJ mol^{-1}) $= -(582.8 \pm 6.0)$ [4].

The results for the measurement, by the Knudsen method, of the standard molar enthalpy of sublimation of $Cu(BETU)_2$ are summarised in Table 3, where are indicated the experimental temperature ranges and the standard molar enthalpy of sublimation at their mean temperature, $\Delta^g_{cr} H^0_m(\langle T \rangle)$ as well as the parameter of the Clausius–Clapeyron equation corresponding to the slope, obtained using a least square fitting of the experimental data. The values of $\Delta^g_{cr} H^0_m(\langle T \rangle)$ were corrected to $T = 298.15$ K assuming $\Delta^g_{cr} C^0_{p,m} = -50\,J\,K^{-1}\,mol^{-1}$, estimated for the complex in accordance with similar previous estimations [11], to yield $\Delta^g_{cr} H^0_m$ $[Cu(BETU)_2] = (180.9 \pm 7.5)\,kJ\,mol^{-1}$, where the uncertainties assigned are twice the overall standard deviations of the mean.

The enthalpy of sublimation of $Cu(BIBU)_2$ could not be experimentally measured in our laboratory, since it did not sublime as the vapour pressure of the crystals is too low until the melting temperature.

Table 3
Experimental standard molar enthalpies of sublimation, $\Delta^g_{cr} H^0_m$, at $\langle T \rangle$, and standard molar enthalpies of sublimation at $T = 298.15$ K, for bis(*N*–benzoyl–*N*′,*N*′-diethylthioureato)copper(II), $Cu(BETU)_2$

Series	$[T_i; T_f]$/K	Slope/K	$\Delta^g_{cr} H^0_m \langle T \rangle$/(kJ mol^{-1})	$\Delta^g_{cr} H^0_m$ ($T = 298.15$ K)/(kJ mol^{-1})
A	(404.7; 413.2)	19617	163.10	168.66
B	(402.7; 413.2)	20780	172.77	178.27
C	(403.2; 413.7)	20806	172.99	178.48
D	(403.6; 412.7)	20959	173.43	178.94
E	(404.2; 414.6)	22155	184.21	189.81
F	(405.0; 415.2)	22305	185.45	191.06

$\langle \Delta^g_{cr} H^0_m\,(T = 298.15\,K) \rangle = (180.9 \pm 7.5)\,kJ\,mol^{-1}$.

So, the methods available in our laboratory for measuring enthalpies of sublimation of crystals, the Knudsen effusion technique and the Calvet microcalorimetry, failed to provide this parameter. Considering the similar structure of both ligands, taking in account the more bulky alkyl substituent of the BIBU and the fact that the experimental standard molar enthalpies of sublimation of both gaseous ligands are equal within the experimental uncertainty associated to them (HBIBU, (137.5 ± 4.5) kJ mol^{-1}) [4] and HBETU, (132.2 ± 2.8) kJ mol^{-1}) [4], we estimate the value of 181 kJ mol^{-1} for the standard molar enthalpy of sublimation of Cu(BIBU)$_2$, to which we assignee a large uncertainty of ± 10 kJ mol^{-1}.

Table 2 summarises the standard molar enthalpies of formation, in the crystalline and in the gaseous states, as well as the standard molar enthalpies of sublimation at $T = 298.15$ K, for the compounds studied in this work.

4. Discussion

The present work reports the first thermochemical contribution to the study of metal–ligand bond enthalpies in metallic complexes of *N*-acyl-chalcogenourea derivatives. The mean metal–ligand dissociation enthalpy $\langle D\rangle(\mathrm{M{-}L})$ for these complexes involving two bidentate O,O-donor ligands, is defined as the enthalpy of the homolytic disruption reaction (8).

$$\mathrm{ML(g)} \rightarrow \mathrm{M(g)} + \mathrm{L(g)} \qquad (8)$$

The dissociation enthalpy of the metal–ligand bond includes the energy associated with the difference in structure of the ligand when bound and free, i.e. the radical reorganisation energy. However, it is not possible the determination of the absolute metal–ligand bond dissociation enthalpies in the chelate system, since the value of $D(\mathrm{H{-}L})$ is not known.

The effect of the reorganisation energies can be eliminated by considering the dissociation enthalpy of the metal–ligand relative to the binding to hydrogen, what is equivalent to the value of the difference $\langle D\rangle(\mathrm{Cu{-}L}) - D(\mathrm{H{-}L})$, given by

$$\begin{aligned}\langle D\rangle(\mathrm{Cu{-}L}) &- D(\mathrm{H{-}L})\\ &= \tfrac{1}{2}\{\Delta_f H^0_m(\mathrm{Cu,g}) - \Delta_f H^0_m(\mathrm{CuL_2,g})\}\\ &\quad + \Delta_f H^0_m(\mathrm{HL,g}) - \Delta_f H^0_m(\mathrm{H,g}) \qquad (9)\end{aligned}$$

Using the following auxiliary data for $\Delta_f H^0_m$(H, g) $= (218.00 \pm 0.01)$ kJ mol^{-1} [15], $\Delta_f H^0_m$(Cu, g) $= (337.6 \pm 1.2)$ kJ mol^{-1} [15], $\Delta_f H^0_m$(HBETU, g) $= -(347.0 \pm 5.6)$ kJ mol^{-1} [4], and $\Delta_f H^0_m$(HBIBU, g) $= -(445.3 \pm 7.4)$ kJ mol^{-1} [4], the values obtained for $\langle D\rangle(\mathrm{Cu{-}L}) - D(\mathrm{H{-}L})$ are for the HBETU and the HBIBU copper(II) complexes, respectively, $-(70 \pm 8)$ and $-(60 \pm 11)$ kJ mol^{-1}, which are equal within the experimental uncertainities associated with them. This has also been found experimentally for other complexes of copper(II) with nine non-fluorinated β-diketones [16] and eight fluorinated β-diketones [17] and for a series of complexes of a given metal with the same type of ligand, such as β-diketone complexes [18].

Considering the values for the dissociation enthalpy of N–H in diphenylamine and in di(4-methyl)phenylamine, respectively, $D(\mathrm{N{-}H}) = (361 \pm 6)$ kJ mol^{-1} [19] and $D(\mathrm{N{-}H}) = (365 \pm 6)$ kJ mol^{-1} [19], and assuming a similar value $D(\mathrm{N{-}H}) = (365 \pm 6)$ kJ mol^{-1} for HBETU and for HBIBU, it is possible to derive the values for $\langle D\rangle(\mathrm{Cu{-}L})$ as (295 ± 10) kJ mol^{-1} and as (280 ± 10) kJ mol^{-1} for the enthalpy of dissociation of metal–ligand, respectively, in Cu(BETU)$_2$ and Cu(BIBU)$_2$, which are again equal within the experimental uncertainities associated with them. Although they must be equivalent in gaseous phase, it does not seem sensible to apportion the sum between the two copper–oxygen bonds, since the only kind of complexes with a similar boundary are the metallic β-diketonates, for which $\langle D\rangle(\mathrm{Cu{-}L}) = (280 \pm 10)$ kJ mol^{-1}, independently of the β-diketonate ligand [17,20]. At the present, with only one $\langle D\rangle(\mathrm{Cu{-}L})$ value available from experimental results for the complex bis(*N*-benzoyl-*N*′,*N*′-diethylureato)-copper(II), it seems only possible to foresee that the Cu–L bond dissociation enthalpies in these two classes of compounds are equal, or very similar, within the uncertainties associated to them.

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