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THERMOCHEMICAL PROPERTIES OF THE CHELATE COMPLEXES OF SOME 3*d*-ELECTRON ELEMENTS WITH 2,2,6,6-TETRAMETHYLHEPTANE-3,5-DIONE

PART II. COMPLEXES OF NICKEL(II), COPPER(II) AND ZINC(II)

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ABSTRACT

The standard molar enthalpies of formation of the chelate nickel(II), copper(II) and zinc(II) complexes with 2,2,6,6-tetramethylheptane-3,5-dione (dipivaloylmethane, HDPM) were determined by the solution calorimetry method. The following values of $\Delta H_f^0(\mathrm{M(DPM)_2(s)}$, 298.15 K) (kJ mol^{-1}) were obtained: $\mathrm{Ni(DPM)_2}$, -1195.7 ± 7.6; $\mathrm{Cu(DPM)_2}$, -1094.8 ± 7.6; $\mathrm{Zn(DPM)_2}$, -1263.8 ± 7.6. The enthalpies of hypothetical dissociation processes of complexes in the gaseous phase were calculated as a measure of the mean bond dissociation energies $\langle D \rangle$(M–O) and the mean coordinate bond dissociation energies $\langle D_{\mathrm{CB}} \rangle$(M–O): $\mathrm{M(DPM)_2(g) = M(g) + 2DPM^{\cdot}(g)}$ and $\mathrm{M(DPM)_2(g) = M^{2+}(g) + 2DPM^{-}(g)}$ respectively.

The thermochemical crystal-field stabilization energies were determined for iron(II), cobalt(II), nickel(II) and copper(II) complexes.

INTRODUCTION

In the last decade considerable interest in the study of thermochemical properties of complex compounds in the solid and gaseous phases has been observed. Burkinshaw and Mortimer have published a broad review [1] covering the problem. The goal of these studies is the determination of the metal–ligand bond energy, a value indispensable for the complete description of phenomena in biological and catalytical processes, based upon the breaking of bonds and the formation of new bonds between metals and ligand donor atoms. The value of the metal–ligand bond energy provides valuable information about the complex structure and the crystal-field effect. It could also be regarded as the criterion of applicability of a theory describing a chemical bond. Such studies, using the β-diketonates of the first series of transition metals in the +2 oxidation state, on the influence of different substituents in the chelate ring of the complex on the dissociation energy of the metal–ligand bond have been carried out by us for many years.

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In Part I we reported the thermochemical characteristics of manganese(II), iron(II) and cobalt(II) dipivaloylmethanates [2]. Now we have extended the study to nickel(II), copper(II) and zinc(II) dipivaloylmethanates.

EXPERIMENTAL

Materials

Dipivaloylmethane (EGA Chemie K.G.) was repeatedly distilled under reduced pressure, using the fraction of boiling point 367–368 K under a pressure of 50 hPa for our measurements. $NiCl_2 \cdot 6H_2O$, $CuCl_2 \cdot 2H_2O$ and $ZnCl_2$ of analytical purity were used. $HCl \cdot 11.618H_2O$ was prepared by dilution of the concentrated acid; its concentration was checked by alkalimetric titration. 1,4-Dioxane was purified by the routine method described elsewhere [3].

Chelates

Nickel(II) dipivaloylmethanate was obtained according to the method outlined by Fackler and Cotton [4]. The ethanolic solution of dipivaloylmethane was added dropwise with continuous stirring to the aqueous solution of nickel containing an excess of sodium acetate. The green precipitate was filtered off, washed with water–ethanol solution and dried over P_2O_5 at 373 K under reduced pressure. The pink–red product was purified by repeated sublimation in vacuo. The results of the elemental analysis were the following. Found: Ni, 13.80; C, 62.10; H, 9.08. Calculated: Ni, 13.81; C, 62.14; H, 9.01 mass%.

Copper(II) dipivaloylmethanate was obtained by the method described by Hammond et al. [5]. To the aqueous copper acetate solution, a methanolic solution of dipivaloylmethane was added dropwise with continuous stirring. The dark blue complex precipitate settled out. It was filtered off, dried in air and repeatedly crystallized from *n*-heptane; dark navy blue crystals were obtained. The results of the elemental analysis were the following. Found: Cu, 14.77; C, 61.40; H, 8.95. Calculated: Cu, 14.77; C, 61.44; H, 8.91 mass%.

Zinc(II) dipivaloylmethanate was obtained by the method described elsewhere [5]. To an aqueous zinc nitrate solution the stoichiometric amount of dipivaloylmethane in ethanol was added followed by concentrated ammonia solution. The white complex was purified by repeated sublimation at 350 K under reduced pressure. The results of the elemental analysis were the following. Found: Zn, 15.15; C, 61.00; H, 8.75. Calculated: Zn, 15.13; C, 61.18; H, 8.87 mass%.

Apparatus and measurement procedure

The measurements of the molar heat of reaction were performed by the batch technique in a precise isoperibol calorimeter described in a previous paper [6]. For the determination of the standard molar enthalpies of formation of the complexes in the solid phase, the formal thermochemical decomposition reactions of the complexes were used:

$$\mathrm{M(DPM)_2(s) + 2HCl \cdot 11.618H_2O(l) = MCl_2 \cdot \mathit{n}H_2O(s) + 2HDPM(l)} \\ \mathrm{+ (23.236 - \mathit{n})H_2O(l)} \qquad (1)$$

Their enthalpies $\Delta H(1)$ were determined from Hess's law as a combination of the molar enthalpies of the successive dissolving of stoichiometric amounts of the reaction substrates and products, carried out according to the appropriate thermodynamic cycle [2]. A mixture of 75% (v/v) 1,4-dioxane and 25% (v/v) 4.36 M HCl was used as solvent.

RESULTS AND DISCUSSION

The results of the calorimetric measurements are presented in Table 1.

By means of the experimental data in Table 1 the enthalpies of reaction (1) were calculated from the relation

$$\Delta \mathrm{H}(1) = \Delta \mathrm{H}(2) + 2\Delta \mathrm{H}(3) - \Delta \mathrm{H}(4) - 2\Delta \mathrm{H}(5) \\ - (23.236 - n)\Delta \mathrm{H}(6) + \Delta \mathrm{H}(7)$$

The results are presented in Table 2. At the strictly preserved stoichiometry the solutions 2 and 5 are thermodynamically equivalent. This was confirmed by their identical UV spectra. Thus $\Delta H(7) = 0.00$.

The standard molar enthalpies of formation of the solid complexes were calculated using the following literature data for ΔH_f^0 (kJ mol^{-1}): HDPM(l), -587.7 ± 3.8 [7]; $\mathrm{HCl \cdot 11.618H_2O(l)}$, -3482.79 ± 0.01 [8]; $\mathrm{H_2O(l)}$, -285.83 ± 0.04 [8]; $\mathrm{NiCl_2 \cdot 6H_2O(s)}$, -2116.3 [9]; $\mathrm{CuCl_2 \cdot 2H_2O(s)}$, -807.5 [9]; $\mathrm{ZnCl_2(s)}$, -415.9 [9]. The calculated values are presented in Table 2.

The literature values for the standard enthalpies of formation of nickel(II) dipivaloylmethanate [10,11] and copper(II) dipivaloylmethanate [12] are compared in Table 3 with our calculated values. In their calculations the quoted authors applied the value, $\Delta H_f^0(\mathrm{HDPM(l)}) = -611.7 \pm 4.2$ kJ mol^{-1}, evaluated from the Laidler bond energy scheme. Our calculations are based on the most recent value, -587.7 ± 3.8 kJ mol^{-1} [7], determined experimentally by the bomb calorimetry method. To compare our values with the literature ones, we have recalculated the latter, taking the new value of $\Delta H_f^0(\mathrm{HDPM(l)})$ into account (Table 3). It is to be noted that the value of the enthalpy of formation of nickel(II) dipivaloylmethanate calculated by Fedotova and Igumenov [11] is very different from that calculated by Irving and Ribeiro da Silva [10] and by us. For copper(II) dipivaloylmethanate the agreement is somewhat better (the difference is within the limit of the sum of the uncertainties attached to those values).

After the determination of the standard enthalpies of formation of complexes in the solid phase, the next step in the determination of the metal–ligand bond energy is the estimation of the molar enthalpies of the dissociation reactions of the complexes into the component metal atoms and ligand radicals (eqn. (8)) or metal ions and ligand ions (eqn. (9)) in the gaseous phase:

$$M(DPM)_2(g) = M(g) + 2DPM^{\cdot}(g) \tag{8}$$

$$M(DPM)_2(g) = M^{2+}(g) + 2DPM^{-}(g) \tag{9}$$

TABLE 1

Calorimetric results at 298.15 K and mean molar enthalpy changes, each with the standard deviation of the mean ($\Delta\theta$, corrected temperature changes; γ, the heat capacity of the calorimeter)

Metal	n (mmol)	$\Delta\theta \times 10^3$ (K)	γ (J K^{-1})	Q (J)	$\Delta H(i)$ (kJ mol^{-1})
Reaction (2): $M(DPM)_2(s)$ + solvent = solution 1					
Ni(II)	0.052853	1.90	453.7	0.862	−16.31
	0.078727	3.00	435.0	1.305	−16.58
	0.085413	3.05	444.6	1.356	−15.87
	0.092624	3.35	439.7	1.473	−15.90
	0.13828	5.20	444.2	2.310	−16.70
			$\langle \Delta H(2) \rangle = -16.27 \pm 0.17$ kJ mol^{-1}		
Cu(II)	0.040222	4.10	445.9	1.828	45.46
	0.075100	7.40	453.8	3.358	44.75
	0.076725	7.60	442.1	3.360	43.79
	0.084514	8.60	437.4	3.761	44.50
	0.13671	13.60	447.3	6.083	44.50
			$\langle \Delta H(2) \rangle = 44.59 \pm 0.27$ kJ mol^{-1}		
Zn(II)	0.077097	2.50	485.3	1.213	−14.60
	0.079829	2.50	453.5	1.134	−14.20
	0.084781	2.70	452.5	1.222	−14.41
	0.096744	3.20	443.2	1.418	−14.66
	0.10952	3.50	453.1	1.586	−14.48
			$\Delta H(2) = -14.47 \pm 0.08$ kJ mol^{-1}		
Reaction (3): $HCl \cdot 11.618H_2O(l)$ + solution 1 = solution 2					
			$\langle \Delta H(3) \rangle = -20.13 \pm 0.02$ kJ mol^{-1} [a]		

TABLE 1 (continued)

Metal	n (mmol)	$\Delta\theta \times 10^3$ (K)	γ (J K^{-1})	Q (J)	$\Delta H(i)$ (kJ mol^{-1})
Reaction (4):	$MCl_2 \cdot nH_2O(s)$ + solvent = solution 3				
Ni(II)	0.052783	0.65	440.3	0.286	−5.42
	0.078780	1.00	451.9	0.452	−5.74
	0.085212	0.95	449.5	0.427	−5.01
	0.092701	1.10	437.4	0.481	−5.19
	0.13794	1.50	451.9	0.678	−4.91
			$\langle \Delta H(4) \rangle = -5.25 \pm 0.17$ kJ mol^{-1}		
Cu(II)	0.040254	0.45	467.7	0.210	−5.23
	0.075280	0.90	442.1	0.398	−5.28
	0.077005	0.90	444.4	0.400	−5.19
	0.084510	1.00	443.5	0.443	−5.25
	0.13655	1.65	441.2	0.728	−5.33
			$\langle \Delta H(4) \rangle = -5.26 \pm 0.04$ kJ mol^{-1}		
Zn(II)	0.077120	9.00	441.6	3.975	−51.54
	0.080025	9.30	440.9	4.100	−51.24
	0.085082	10.00	442.2	4.422	−51.98
	0.096644	11.10	444.4	4.933	−51.04
	0.11004	12.80	441.9	5.657	−51.40
			$\langle \Delta H(4) \rangle = -51.44 \pm 0.17$ kJ mol^{-1}		
Reaction (5):	HDPM(l) + solution 3 = solution 4				
			$\langle \Delta H(5) \rangle = 10.69 \pm 0.08$ kJ mol^{-1} [a]		
Reaction (6):	$H_2O(l)$ + solution 4 = solution 5				
			$\langle \Delta H(6) \rangle = -0.91 \pm 0.01$ kJ mol^{-1} [a]		
Reaction (7):	solution 2 = solution 5				
			$\langle \Delta H(7) \rangle = 0.00$		

[a] The enthalpies of these reactions are independent of the composition of solutions 1, 3 and 4. The detailed results of the calorimetric measurements were reported in Part I [2].

The enthalpies of reactions (8) and (9) are a measure of the mean bond dissociation energy $\langle D \rangle$(M–O) and of the mean coordinate bond dissociation energy $\langle D_{CB} \rangle$(M–O) respectively. Since the metal–oxygen bonds in the

TABLE 2

The enthalpies of reaction (1) and standard molar enthalpies of formation of complexes in the solid and gaseous phases

Complex	$\Delta H(1)$ (kJ mol^{-1})	$-\Delta H_f^0(s)$ (kJ mol^{-1})	$-\Delta H_f^0(g)$ (kJ mol^{-1})
$Ni(DPM)_2$	-56.98 ± 0.33	1195.7 ± 7.6	1050.5 ± 12.6
$Cu(DPM)_2$	7.53 ± 0.38	1094.8 ± 7.6	949.8 ± 21.4
$Zn(DPM)_2$	-3.53 ± 0.34	1263.8 ± 7.6	1118.8 ± 21.4

TABLE 3

The list of the known standard molar enthalpies of formation (kJ mol^{-1})

Complex	$-\Delta H_f^0(s)$ (original values)	Ref.	$-\Delta H_f^0(s)$ (recalculated values)	$-\Delta H_f^0(s)$ (this paper)
$Ni(DPM)_2$	1251.8 ± 8.4	10	1203.8 ± 8.4	1195.7 ± 7.6
	1185.2 ± 6.6	11	1137.1 ± 8.0	
$Cu(DPM)_2$	1158.1 ± 6.2	12	1110.1 ± 8.0	1094.8 ± 7.6

dipivaloylmethanates studied are equivalent [13], their energies were calculated from the relations:

$$\langle D\rangle(M-O) = 1/4\Delta H(8) = 1/4\Delta H_f^0(M(g)) + 1/2 D(DPM^{\bullet}-H, enol(g)) + 1/2\Delta H_f^0(HDPM, enol(g)) - 1/2\Delta H_f^0(H(g)) - 1/4\Delta H_f^0(M(DPM)_2(g)) \quad (10)$$

and

$$\langle D_{CB}\rangle(M-O) = 1/4\Delta H(9) = 1/4\Delta H_f^0(M^{2+}(g)) + 1/2\Delta H_f^0(DPM^-(g)) - 1/4\Delta H_f^0(M(DPM)_2(g)) \quad (11)$$

where $D(DPM^{\bullet}-H)$ is the O–H bond dissociation energy in the enol form of dipivaloylmethane, equal to the enthalpy of the process

$$HDPM, enol(g) = H(g) + DPM^{\bullet}(g) \quad (12)$$

The experimental data for this energy for any β-diketone are not yet available, therefore this value should be estimated in the most reliable way. In the literature reports it is proposed that the $D(\beta$-dik–O) values are equal to the average value of the O–H bond dissociation energy in alcohols and aliphatic acids [14] or are equal to D(O–H) in phenol [15]. In our previous paper we have assumed the $D(ACAC^{\bullet}-H) = D(O-H)$ in alcohols, i.e. 418 ± 20 kJ mol^{-1} (ACAC, acetylacetonate) [6]. On the basis of this evaluation, the dissociation energy of the $DPM^{\bullet}$–H bond should be compared with the O–H bond dissociation energy in the appropriate alcohol of the branched chain, i.e. *t*-butanol: $D(t\text{-}C_4H_9O-H) = 434$ kJ mol^{-1} instead of $D(n\text{-}C_4H_9O-H) = 423$ kJ mol^{-1} [16]. A comparison of these data revealed only a slight influence of the branching of the aliphatic chain on the O–H bond energy. Thus, this conclusion for dipivaloylmethane seems to be reasonable and leads to $D(DPM^{\bullet}-H) = D(ACAC^{\bullet}-H) = 418 \pm 20$ kJ mol^{-1}.

The other values necessary for the determination of the enthalpy of reactions (8) and (9) are the heats of sublimation of the complexes. This value has been determined experimentally only for nickel(II) dipivaloylmethanate as 145.2 ± 10.0 kJ mol^{-1} [17]. Because of their unusual volatility, transition metal β-diketonates have been the subject of broad investigations to determine their sublimation enthalpies. It should be emphasized, however, that the results obtained vary depending on the method of measurement used, and have been criticized recently [1]. The reliable estimation of the enthalpy of sublimation should be based on incontrovertible experimental data. The precise measurements by the Knudsen effusion technique produced almost identical sublimation enthalpy values of 132 ± 10 kJ mol^{-1}, 132 ± 17 kJ mol^{-1} and 133 ± 9 kJ mol^{-1} for $Ni(ACAC)_2$, $Pd(ACAC)_2$ and $Pt(ACAC)_2$ respectively [18]. The other measurements indicated that the tetrahedral beryllium(II) acetylacetonate has an almost identical sublimation enthalpy value as the nickel(II) acetylacetonate (planar in the gaseous phase) [19]. On the basis of these experimental facts, the same sublimation enthalpy value for the planar $Cu(DPM)_2$ [20] and for the tetrahedral $Zn(DPM)_2$ [21] was assumed as for $Ni(DPM)_2$, i.e. 145 ± 20 kJ mol^{-1}. For the calculation of the enthalpies of reactions (8) and (9) we have applied the following literature data for ΔH_f^0 (kJ mol^{-1}): Ni(g), 430.1; Ni^{2+}(g), 2930.5 ± 1.5; Cu(g), 338.9 ± 2.1; Cu^{2+}(g), 3054.5 ± 2.1; Zn(g), 129.1 ± 0.4; Zn^{2+}(g), 2781.0 ± 0.4 [22]; HDPM, enol(g), −528.5 ± 3.8 [2]; DPM^-(g), −361.5 ± 20.4 [2]; H(g), 218.00 ± 0.01 [23]. The results are presented in Table 4. A comparison of the data in Table 4 with the values for the acetylacetonates of the metals studied [6] revealed that the replacement of a methyl group by a *t*-butyl group in the chelate ring of the complex has no effect on the metal–oxygen bond strength. This statement is correct only for the proper evaluation of the dissociation energy of the O–H bond in the ligand and of the sublimation enthalpy of complexes.

For the complete characterization of the metal–ligand bond, the relation between its length and energy is interesting. The dependence of the dissociation enthalpies $\Delta H(8)$ and of the ionic radii on the d-orbital population is shown in Fig. 1. These relations are mutually opposite. According to the simple electrostatic model, the shorter metal–ligand bonds should be char-

TABLE 4

The enthalpy change $\Delta H(8)$ of the radical and the enthalpy change $\Delta H(9)$ of the ionic complex dissociation processes with metal-oxygen bond energies ($\langle D \rangle$(M–O), mean bond dissociation energy; $\langle D_{CB} \rangle$(M–O), mean coordinate bond dissociation energy)

Complex	$\Delta H(8)$ (kJ mol^{-1})	$\Delta H(9)$ (kJ mol^{-1})	$\langle D \rangle$(M–O) (kJ mol^{-1})	$\langle D_{CB} \rangle$(M–O) (kJ mol^{-1})
$Ni(DPM)_2$	824 ± 43	3258 ± 43	206 ± 11	814 ± 11
$Cu(DPM)_2$	632 ± 46	3281 ± 46	158 ± 12	820 ± 12
$Zn(DPM)_2$	591 ± 46	3177 ± 46	148 ± 12	794 ± 12
$Mn(DPM)_2$ [a]	806 ± 46	2980 ± 46	201 ± 12	745 ± 12
$Fe(DPM)_2$ [a]	866 ± 46	3136 ± 46	217 ± 12	784 ± 12
$Co(DPM)_2$ [a]	850 ± 46	3201 ± 46	212 ± 12	800 ± 12

[a] Values taken from ref. 2.

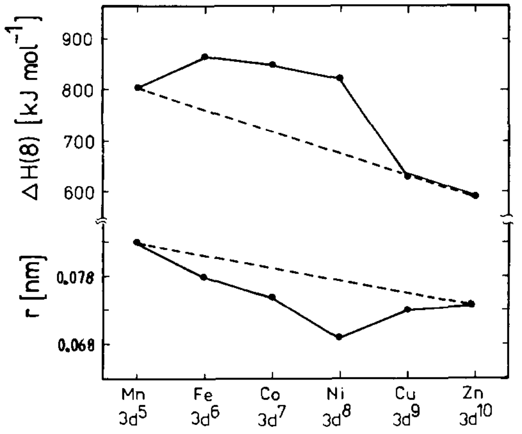

Fig. 1. Plot of the dissociation enthalpies $\Delta H(8)$ and ionic radii vs. the *d*-orbital population.

acterized by the higher enthalpy of formation, and, consequently, by the higher bond energy. This was confirmed in the dipivaloylmethanates studied, where the crystallographically determined bond lengths, i.e. r(Ni–O) = 0.1836 nm [24], r(Cu–O) = 0.1911 nm [20] and r(Zn–O) = 0.1962 nm [21] exhibit the opposite direction of change in their bond energy, $\langle D \rangle$(M–O). This is a smooth relationship (Fig. 2).

The ligand-field theory [25] predicts no stabilization for the high spin complexes of divalent manganese and zinc, but does predict additional stabilization for the high spin complexes of the divalent ions of iron, cobalt, nickel and copper. This is reflected in the relation of the enthalpy of dissociation of complexes of component ions in the gaseous phase, $\Delta H(9)$, with the electronic structure of the central ion (Fig. 3). From this relation the crystal-field stabilization energies (CFSE) were obtained graphically as

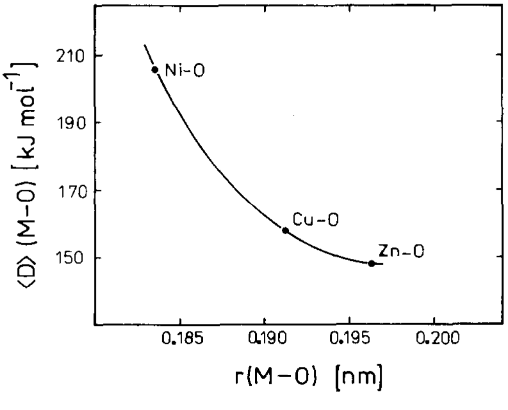

Fig. 2. Plot of $\langle D \rangle$(M–O) bond energies vs. M–O bond length.

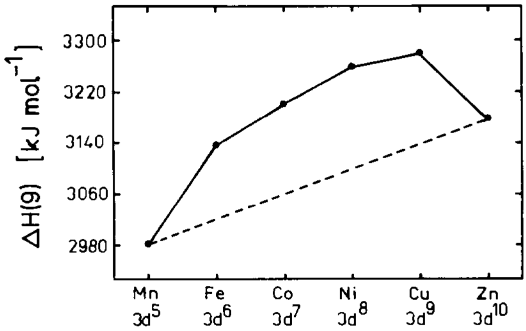

Fig. 3. Plot of the enthalpy changes of reaction (9) vs. the *d*-orbital population.

differences between the real and hypothetical values for the state without ligand-field influence (broken line between values for Mn(II)-d^5 and Zn(II)-d^{10}). The CFSE values obtained show the same trend as the CFSE values for acetylacetonates [6]: 117 (iron(II)) < 143 (cobalt(II)) $\leqslant$ 144 (copper(II)) < 160 (nickel(II)) kJ mol^{-1}.

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