Journal of Thermal Analysis and Calorimetry, Vol. 89 (2007) 2, 573–578

THERMAL DECOMPOSITION OF $[Cd(NH_3)_6](NO_3)_2$

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Thermal decomposition of $[Cd(NH_3)_6](NO_3)_2$ was studied by thermogravimetry (TG) with simultaneous differential thermal analysis (SDTA) for two samples and at two different sets of measurement parameters. The gaseous products of the decomposition were on-line identified by evolved gas analysis (EGA) with a quadruple mass spectrometer (QMS). The decomposition of the title compound proceeds, for both cases, in the three main stages. In the first stage, deammination of $[Cd(NH_3)_6](NO_3)_2$ to $[Cd(NH_3)](NO_3)_2$ undergoes by three steps and 5/6 of all NH_3 molecules are liberated. At second stage the liberation of residual $1/6NH_3$ molecules and the formation of $Cd(NO_3)_2$ undergoes. However, during this process simultaneously a two-step oxidation of a part of ammonia molecules also takes place. In a first step as a result a mixture of ammonia, water vapour and nitrogen is formatted. At the second step, subsequent oxidation of a next part of NH_3 molecules undergoes. As a result, a mixture of nitrogen oxide, nitrogen and water vapour is formatted, what for these both steps clearly indicates the EGA analysis. The third stage of the thermal decomposition is connected with the melting and subsequent decomposition of residual $Cd(NO_3)_2$ to oxygen, nitrogen dioxide and solid CdO. Additionally, third sample was measured by differential scanning calorimetry (DSC) and the results are fully consistent with those obtained by TG.

Keywords: *DSC, EGA, hexaamminecadmium(II) nitrate(V), SDTA, TG, thermal decomposition*

Introduction

Thermal decompositions of the compounds of the type: $[M(NH_3)_6](NO_3)_2$ and $[M(H_2O)_6](NO_3)_2$, where M=Ni and Mg, were the subjects of our former studies [1, 2]. The same subject matter was studied by Wendlandt and Smith [3] for $[Co(NH_3)_6](NO_3)_2$ and by other authors [4–13] for various metal nitrates(V) and their hydrates and aqua complexes. From among all these, the mechanism of thermal decomposition of $[Cd(H_2O)_4](NO_3)_2$ was studied by Wojciechowski and Małecki [10]. As a natural extension of these studies we present here the results for $[Cd(NH_3)_6](NO_3)_2$.

The room temperature structure of the title compound is built of octahedral $[Cd(NH_3)_6]^{2+}$ cations and NO_3^- anions, with a network of very weak hydrogen bonds joined them with one another in a regular (cubic) lattice, space group $Fm\overline{3}m$. However, unfortunately the crystal lattice parameters and the structure of the low temperature phase are unknown.

Hexaamminecadmium(II) nitrate(V) has two solid phases between 100 and 300 K. The phase transitions temperatures values, at heating T_C^h=211.9 K and at cooling T_C^c=205.3 K, were obtained by us [14] from DSC measurements, by extrapolating of the corresponding T_{Cpeak}^h and T_{Cpeak}^c values *vs*. the heating and cooling rate dependence, respectively, to the scanning rate value equal zero.

Activation energy for the picoseconds reorientational motions of the NH_3 ligands through the 120° jumps around three-fold axis: Cd–N ($E_a(120°)$= 7.7 kJ mol^{-1}) was also obtained by us for the title compound from temperature dependence of FWHM of the infrared band connected with the rocking mode of NH_3 [14]. Similar activation energies: $E_a(120°)$=7.1 kJ mol^{-1} and additionally E_a(tumbl.)= 9.2 kJ mol^{-1}, for the tumbling motions of the whole $[Cd(NH_3)_6]^{2+}$ cations, were estimated from ^1H NMR measurements by Piślewski [15].

The general aim of this study was to gain a better understanding of the mechanism of thermal decomposition of the hexaamminecadmium(II) nitrate(V), particularly of the deammination process, in order to find eventual correlations with a molecular reorientational motions of NH_3 molecules and also to compare the obtained results with those obtained earlier for $[Mg(NH_3)_6](NO_3)_2$, $[Ni(NH_3)_6](NO_3)_2$ and $[Co(NH_3)_6](NO_3)_2$. Additionally, we checked out an influence of experimental conditions on the obtained results.

Experimental

The examined compound was obtained from $[Cd(H_2O)_4](NO_3)_2$. The tetraaquacadmium(II) nitrate(V) was synthesized by a reaction of the corre-

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1388–6150/$20.00

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sponding carbonate with diluted HNO_3 and re-crystallized several times from distilled water. After drying it for several days over BaO in a desiccator, it was placed in a quartz vessel and put in a glass tube through which dry gaseous ammonia was blown and the tube was placed inside an oven. First the tube was heated for several hours at about 370 K until all the water from the hexaaquacomplex had been lost. Then the hexaamminecomplex that formed was cooled to room temperature and kept at this temperature for several days. Before the measurements, the composition of the compound was determined on the basis of ammonia content with titration by means of HCl. The average content of NH_3 was found to be equal to the theoretical value within the error limited of ca. 1%. The infrared and Raman spectra also identified the title compound [14].

Thermogravimetry (TG/DTG) with simultaneous differential thermal analyses (SDTA) measurements were performed using a Mettler-Toledo 851^e apparatus. Sample A of mass equal to 13.0709 mg was placed in a 150 μL open platinum crucible and sample B of mass equal to 19.4637 mg was placed in a 150 μL open corundum crucible. High purity (99.999%) dry argon was used as the purge gas and the oxygen concentration in the system was below 0.001%. Both measurements were made within temperature range 300–873 K in a flow of argon (100 mL min^{-1}), but with a constant heating rate equal to: 5 K min^{-1} for sample A and 10 K min^{-1} for sample B. Each time before beginning of the experiments, the TA chamber has been purged over 5 min. The simultaneous evolved gas analysis (EGA) was performed during the experiments by a joined on-line quadruple mass spectrometer (QMS) using a Thermostar-Balzers apparatus with an ionization energy equal to 1400 eV. The temperature was measured by a Pt–Pt/Rh thermocouple with an accuracy of ±0.5 K.

Differential scanning calorimetry (DSC) measurements were performed using a Mettler-Toledo 821^e calorimeter for a sample C of mass equal to 2.85 mg in 40 μL aluminum closed crucibles with hole 0.5–1.0 mm diameter under the constant flow of argon (80 mL min^{-1}) in temperature range 300–873 K, with the heating rate equals to 5 K min^{-1}.

Results and discussion

As was specified above, two different experimental conditions were chosen in order to determine whether the observed thermal decomposition depends on: a sample mass (13.0709 mg – called sample A and 19.4639 mg – called sample B), a heating rate (5 and 10 K min^{-1}) and a material of crucibles (corundum and platinum). Below we present results of thermal decomposition obtained for sample A and sample B, in succession.

Figure 1 shows TG, DTG, QMS and SDTA curves recorded for sample A of $[Cd(NH_3)](NO_3)_2$ in platinum crucible at a constant heating rate of 5 K min^{-1} at the temperature range of 300–873 K. During the TG experiment, the QMS spectra of masses were followed from m/e=1 to 100, however, for reasons of graphic readability, only the masses of m/e=17, 18, 28, 30, 32, 44 and 46 – representing: NH_3, H_2O, N_2, NO, O_2, N_2O and NO_2 are shown. The QMS spectrum of mass m/e=17 represents, besides NH_3, also the OH fragment of H_2O fragmentation, which was taken into account in the determination of NH_3 concentration. The temperatures, percentage mass losses and the products of the decomposition at particular stages for sample A are presented in Table 1.

The TG, DTG and QMS curves show that the decomposition of the sample A proceeds in three main stages (I, II and III). It can be observed that the stage I involves the releasing of 5/6 of all NH_3 molecules in the three steps (Ia, Ib, Ic). The stage II is connected with the liberation of 1/6 of all ammonia molecules and with the formatting of $Cd(NO_3)_2$. However, the oxidation of a part of evolved NH_3 molecules concurrently take place, with formatting in the first step (IIa) of wa-

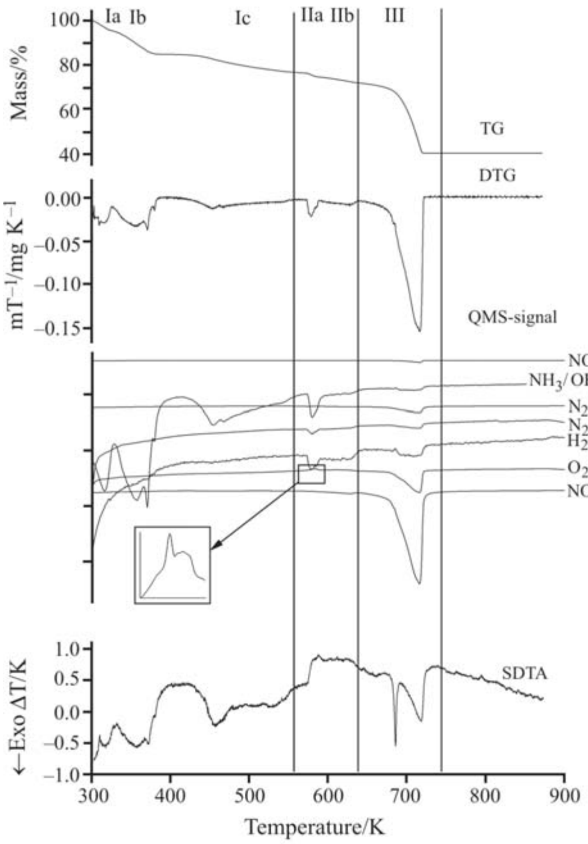

Fig. 1 TG, DTG, QMS and SDTA curves for $[Cd(NH_3)_6](NO_3)_2$ at the range of 300–873 K and at a constant heating rate of 5 K min^{-1} in a flow of argon (100 mL min^{-1}). Sample A in platinum crucible

Table 1 Parameters of $[Cd(NH_3)_6](NO_3)_2$ thermal analysis – sample A in platinum crucible

Sample mass/mg	Stage number	Temperature range/K	Mass loss at the stage/%	Mass after decomposition/%	Calculated values/%	Products of the decomposition
13.0709	Ia	302–327	4.5		5.0	NH_3
	Ib	327–398	10.6		10.1	$2NH_3$
	Ic	398–572	8.5		10.1	$2NH_3$
	IIa,b	572–640	4.5		5.0	NH_3
	III	640–727	31.5		31.9	$1/2O_2$+$2NO_2$
				40.4	37.9	CdO

ter vapour and nitrogen, and at the second step (IIb) of water vapour, nitrogen and NO, what is clearly seen as the minima on appropriate QMS curves. Note that in the case of the QMS curve which corresponds to the oxygen (m/e=32) we can see the maximum (see an insertion in Fig. 1). It can be explained as a sudden consumption of oxygen due to ammonia oxidation. The ammonia oxidation observed during stage IIa of the sample decomposition may be explained in terms of catalytic effect of platinum crucible, well known as a good catalyst for oxidation. The oxygen necessary for ammonia oxidation is supplied in our opinion in sufficient amount by the purge gas. If this assumption is true, we should not observe this effect changing platinum crucible on corundum one. Really, as it will be indicated latter, in corundum crucible the ammonia oxidation practically does not exist. The stage III, which hold just after the melting point of $Cd(NO_3)_2$, is connected with the total decomposition of $Cd(NO_3)_2$ to nitrogen dioxide, oxygen and solid CdO. 40.4% of the initial mass of the sample remained after stage III of the thermal decomposition and this quite well corresponds to the theoretical amount of cadmium oxide (Table 1).

The profile of the SDTA curve of $[Cd(NH_3)_6](NO_3)_2$ shows five endothermic and two exothermic peaks. Jointly with TG+QMS results presented above, the first three endothermic peaks can be explained by the three steps of the deammination process. The first endothermic peak corresponding to liberation of one-sixth of all NH_3 molecules and the second to liberation of two-sixth of NH_3 molecules and the latter was undergone immediately after the first one. The third endothermic peak corresponds to liberation of two-sixth of NH_3 molecules at the third step. As the result of such three-step deammination of $[Cd(NH_3)_6](NO_3)_2$ the penta-, tri- and mono-amminecadmium(II) nitrate(V) are formatting, according to the following reactions:

$$\text{Ia}\quad [Cd(NH_3)_6](NO_3)_2 \rightarrow [Cd(NH_3)_5](NO_3)_2 + NH_3$$

$$\text{Ib}\quad [Cd(NH_3)_5](NO_3)_2 \rightarrow [Cd(NH_3)_3](NO_3)_2 + 2NH_3 \quad (1)$$

$$\text{Ic}\quad [Cd(NH_3)_3](NO_3)_2 \rightarrow [Cd(NH_3)](NO_3)_2 + 2NH_3$$

The first small exothermic peak on SDTA curve (at ca. 580 K) can be explained as connected simultaneously with both processes: 1) endothermic – decomposition of monoamminecadmium(II) nitrate(V) to $Cd(NO_3)_2$ and NH_3, according to the reaction for stage II and 2) exothermic – oxidation of a part of liberated ammonia molecules, according to the reaction for step IIa. As a result of this oxidation a mixture of non-oxidized ammonia, nitrogen and water vapour is formatted, what indicates the EGA analysis. The second small exothermic peak (at ca. 625 K) is connected with a subsequent oxidation of the next part of non-oxidized yet NH_3 molecules (step IIb). As a result a mixture of the rest of non-oxidized ammonia, nitrogen oxide, nitrogen and water vapour is formatted, what clearly indicates EGA analysis (Fig. 1). Thus, the stage II and steps: IIa and IIb of the thermal decomposition of the title compound can be described, for example, by the following reactions:

$$\text{II}\quad [Cd(NH_3)](NO_3)_2 \rightarrow Cd(NO_3)_2 + NH_3 \quad (2)$$

$$\text{IIa}\quad xNH_3 + 1/4xO_2 \rightarrow 2/3xNH_3 + 1/6xN_2 + 1/2xH_2O$$

and (3)

$$\text{IIb}\quad yNH_3 + 1/2yO_2 \rightarrow 1/2yNH_3 + 1/4yNO + 1/8yN_2 + 3/4yH_2O$$

Similar effect was observed by us in the case of thermal decomposition of $[Ni(NH_3)_6](NO_3)_2$ [1] and lately also by Rodrigues and Monteiro in thermal decomposition of $[Pt(NH_3)_4]^{2+}$ in NaX zeolite [16].

The fourth big and narrow endothermic peak presents in SDTA curve at T_m=686 K is connected with the melting of the $Cd(NO_3)_2$, what is consistent with presented below DSC results. The last fifth endothermic big and broad peak (at 737 K) can be explained as the $Cd(NO_3)_2$ decomposition in the liquid phase, according to one of the following probable reactions:

$$\text{III}\quad Cd(NO_3)_2 \rightarrow CdO + 1/2O_2 + 2NO_2 \quad (4)$$

Both, the temperature of the melting and the mechanism of thermal decomposition of $Cd(NO_3)_2$ are fully consistent with the results presented by Wojciechowski and Małecki [9], who investigated thermal decomposition of $[Cd(H_2O)_4(NO_3)_2$. However, due to high value energy of ionization in our QMS spectrometer, and in consequence of the strong fragmentation of the decomposition products, relatively low intensity of NO_2 peak is observed. Moreover, according to our previous results [1, 2], we suppose that in the gaseous phase of the decomposition products the following byproduct reactions may also take place:

$$N_2O+2O_2 \leftrightarrow NO_2+NO+O_2 \leftrightarrow 3/2NO_2+1/4N_2+O_2 \leftrightarrow 2NO+3/2O_2 \quad (5)$$

For example, Małecka [13] describing a thermal decomposition of several nitrates(V) of aqua complexes of transition metals distinguishes as the byproducts, beside NO_2 and O_2, also NO and N_2O.

In order to carry out the careful comparison of the obtained results with those obtained earlier for $[Mg(NH_3)_6](NO_3)_2$ and $[Ni(NH_3)_6](NO_3)_2$ we decided to repeat the experiment for $[Cd(NH_3)_6](NO_3)_2$ with exactly the same experimental conditions (sample B) as were realised by us in the case of the hexaamminemagnesium and hexaamminenickel(II) nitrates(V) [1]. Figure 2 shows TG, DTG, QMS and SDTA curves recorded for sample B of $[Cd(NH_3)](NO_3)_2$ in corundum crucible at a constant heating rate of 10 K min^{-1} at the temperature range of 300–900 K. During the TG experiment, the QMS spectra of masses were registered, exactly like in the case of sample A. The temperatures, percentage mass losses and the products of the decomposition at particular stages for sample B are presented in Table 2.

The TG, DTG and QMS curves show that the decomposition of the sample B proceeds in three main stages (I, II and III). It can be observed that the stage I involves the freeing of five-sixth of all NH_3 molecules in the three steps (Ia, Ib, Ic). In steps Ia and Ib one-half of NH_3 molecules is freeing. In a case of the sample B step Ic and stage II were summed up because the obtained results were not very precise, due to experimental conditions, and it is difficult to specify these two processes. The stage II is connected with the liberation of 1/6 of all ammonia molecules and with the formatting of solid $Cd(NO_3)_2$, thus altogether in this summered stages (Ic+IIa,b) the second one-half of all NH_3 molecules are evolved. At the stage II of sample B decomposition in a corundum crucible, contrary to the previous experiment, we do not observe a consumption of oxygen, thus the oxidation of the evolved NH_3 has no place. It proves our hypothesis about catalytic effect of platinum crucible. The stage III is connected, similarly to the previous experiment, with the decomposition of $Cd(NO_3)_2$ to nitrogen dioxide, oxygen and solid CdO. 39.4% of the initial mass of the sample remained after the stage III of the decomposition and this quite well corresponds to the theoretical amount of cadmium oxide (Table 2).

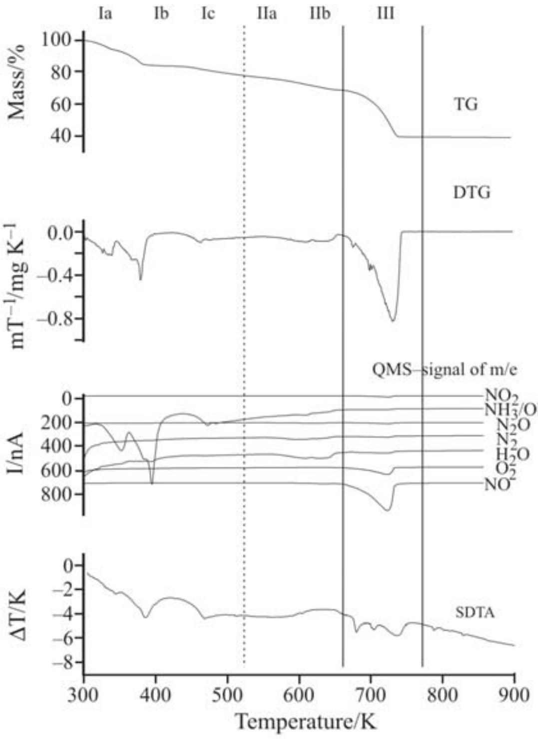

Fig. 2 TG, DTG, QMS and SDTA curves for $[Cd(NH3)_6](NO_3)_2$ at the range of 300–900 K and at a constant heating rate of 10 K min^{-1} in a flow of argon (100 mL min^{-1}). Sample B in corundum crucible

The profile of the SDTA curve for sample B of $[Cd(NH_3)_6](NO_3)_2$ is not exactly the same as the one registered for sample A. Nevertheless, it clearly indicates that the mechanism of thermal decomposition for sample B is nearly the same as for sample A.

Table 2 Parameters of $[Cd(NH_3)_6](NO_3)_2$ thermal analysis – sample B in corundum crucible

Sample mass/mg	Stage number	Temperature range/K	Mass loss at the stage/%	Mass after decomposition/%	Calculated values/%	Products of the decomposition
19.4637	Ia	302–348	6.0		5.0	NH_3
	Ib	348–420	10.1		10.1	$2NH_3$
	Ic+IIa,b	420–660	15.0		15.1	$3NH_3$
	III	660–756	29.5		31.9	$1/2O_2+2NO_2$
				39.4	37.9	CdO

Only small differences were observed in the third step of deammination (Ic), which can not be sequestered from the second stage (II) of [Cd(NH$_3$)$_6$](NO$_3$)$_2$ decomposition, probably because of worse thermal conductivity of the corundum crucible.

The above presented mechanism of thermal decomposition of [Cd(NH$_3$)$_6$](NO$_3$)$_2$ is well consistent with the results which we obtained from DSC measurement. The DSC curve obtained for the title compound at heating the sample C is presented in Fig. 3. The profile of DSC curve shows five endothermic and two exothermic peaks. The first three endothermic peaks can be explained by the three stages of the deammination resulting in the formation of the penta-, tri- and monoammine, according to reaction (1). Next, two small exothermic peaks can be explained as a superposition of two effects: the first is related to the final deammination (endothermic process) and the second one is related to ammonia oxidation (exothermic process). Competitive exothermic effect connected with a structural rearrangement of the sample is also possible but less probable because Cd(NO$_3$)$_2$ above T_C=433 K undergoes the phase transition from orthorhombic to cubic lattice [17]. The remaining two big endothermic peaks are connected with the melting and decomposition of Cd(NO$_3$)$_2$, respectively. The melting point is connected with entropy change ΔS=66.6 J mol^{-1} K^{-1}.

The thermal decomposition of [Cd(NH$_3$)$_6$](NO$_3$)$_2$ is somewhat different than that of such isostructural complex compounds as: [Ni(NH$_3$)$_6$](NO$_3$)$_2$, [Mg(NH$_3$)$_6$](NO$_3$)$_2$ and [Co(NH$_3$)$_6$](NO$_3$)$_2$. Firstly, in the case of [Ni(NH$_3$)$_6$](NO$_3$)$_2$ and [Co(NH$_3$)$_6$](NO$_3$)$_2$ the whole thermal decomposition proceeds in two main stages and in the case of [Mg(NH$_3$)$_6$](NO$_3$)$_2$ it undergoes in three main stages. From that point of view, thermal decomposition of [Cd(NH$_3$)$_6$](NO$_3$)$_2$ is more similar to that for [Mg(NH$_3$)$_6$](NO$_3$)$_2$. Secondly, the deammination process of [Ni(NH$_3$)$_6$](NO$_3$)$_2$ undergoes in three steps and only two-third (2/6+1/6+1/6=2/3) of all NH$_3$ molecules are initially liberated, in the case of [Mg(NH$_3$)$_6$](NO$_3$)$_2$ the deammination undergoes in two steps and two-third (2/6+2/6=2/3) of all NH$_3$ molecules are liberated and in the case of [Co(NH$_3$)$_6$](NO$_3$)$_2$ the deammination undergoes in one step and 1/3 of all NH$_3$ molecules are liberated. In turn, the deammination process of [Cd(NH$_3$)$_6$](NO$_3$)$_2$, for sample B all (1/6+2/6+3/6=1) NH$_3$ molecules are freeing, although it undergoes in three steps, as in the case of [Ni(NH$_3$)$_6$](NO$_3$)$_2$, though it is quite different from those for all the other aforementioned compounds. The mechanism of further thermal deammination and decomposition of these compounds is also different. In all cases they are connected with the reduction-oxidation processes, but in the case of [Mg(NH$_3$)$_6$](NO$_3$)$_2$, contrary to the [Ni(NH$_3$)$_6$](NO$_3$)$_2$, at the second stage, besides the liberation of 1/3 of all NH$_3$, undergoes the formation of Mg(NO$_3$)$_2$, which next in turn partially decomposes with creating O$_2$ (besides MgO and NO), which in turn oxidizes NH$_3$, what in consequence leads to forming also N$_2$, NO and H$_2$O. Total decomposition of magnesium nitrate(V) is undergone just in the third stage. This third stage is not present in the case of [Ni(NH$_3$)$_6$](NO$_3$)$_2$, and the decomposition of diamminenickel(II) nitrate(V) is undergone directly to the final products (H$_2$O, NO, N$_2$O and N$_2$), without the formation of Ni(NO$_3$)$_2$, because of the autocatalytic effect of the formed NiO. In the case of the cobalt compound the tetraamminecobalt(II) nitrate(V) is decomposed very rapidly with formations of H$_2$O, N$_2$, and Co$_3$O$_4$, besides NH$_3$. Summing up this comparison, we can notice that although the thermal decomposition of [Cd(NH$_3$)$_6$](NO$_3$)$_2$ is somewhat different from that for other aforementioned compounds, it is thought the most similar to the thermal decomposition of [Mg(NH$_3$)$_6$](NO$_3$)$_2$.

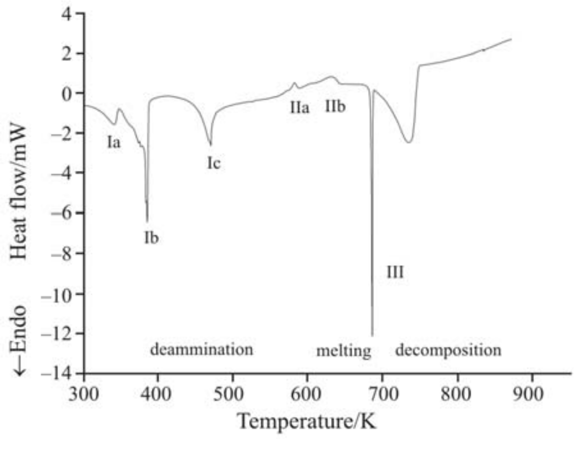

Fig. 3 DSC curve for [Cd(NH$_3$)$_6$](NO$_3$)$_2$ at the range of 300–873 K and at heating rate of 5 K min^{-1} in a flow of argon (80 mL min^{-1}). Sample C in an aluminium crucible

Conclusions

From the present study it can be concluded that:

- The thermal decomposition of the title compound proceeds in three main stages. In stage I deammination of [Cd(NH$_3$)$_6$](NO$_3$)$_2$ to [Cd(NH$_3$)](NO$_3$)$_2$ is undergone in three steps and five-sixth (1/6+2/6+2/6=5/6) of the all NH$_3$ molecules are liberated. Stage II undergoes the liberation of 1/6 of all NH$_3$ and the formation of Cd(NO$_3$)$_2$. Simultaneously, in a platinum crucible, a part of evolved NH$_3$ molecules are oxidized in two steps: at 580 and at 625 K. Stage III of thermal decomposition, which take place after melting of the cadmium nitrate(V), is connected with the decomposition of Cd(NO$_3$)$_2$ to oxygen, nitrogen dioxide and solid CdO.

- The ammonia oxidation process, which take place at the stage II of the $[Cd(NH_3)_6](NO_3)_2$ thermal decomposition in platinum crucible, practically does not undergoes in a corundum crucible.
- Beside the ammonia oxidation process, we did not notice significant differences between the mechanism of thermal decomposition of $[Cd(NH_3)_6](NO_3)_2$ obtained at two different experimental conditions. Small differences were connected only with a different heating rate, mass of a sample, thermal conductivity and catalytic properties of particular crucible.
- The thermal deammination processes and the mechanism of further thermal decomposition of similar complex compounds of the type: $[M(NH_3)_6](NO_3)_2$, where M=Mg, Ni, Co and Cd, are slightly different. However thermal decomposition of $[Cd(NH_3)_6](NO_3)_2$ is more similar to that of $[Mg(NH_3)_6](NO_3)_2$ than to those of the others aforementioned compounds.

Acknowledgements

Our thanks are due to Associated Professor A. Migdał-Mikuli and Ł. Skoczylas MSc. from the Faculty of Chemistry of the Jagiellonian University for stimulating the discussion.

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Received: March 23, 2006
Accepted: June 7, 2006
OnlineFirst: October 20, 2006

DOI: 10.1007/s10973-006-7610-6