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THERMAL DECOMPOSITION OF GALLIUM NITRATE HYDRATE $Ga(NO_3)_3 \cdot xH_2O$

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The thermal decomposition of gallium nitrate hydrate ($Ga(NO_3)_3 \cdot xH_2O$) to gallium oxide has been studied by TG/DTG and DSC measurements performed at different heating rates. It is concluded that 8 water molecules are present in the hydrate compound. The anhydrous gallium nitrate does not form at any temperature as the reaction consists of coupled dehydration/decomposition processes that occur with a mechanism dependent on heating rate. TG measurements performed with isothermal steps (between 31 and 115°C) indicate that $Ga(OH)_2NO_3$ forms in the first stage of the reaction. Such a compound undergoes further decomposition to $Ga(OH)_3$ and $Ga(NO_3)O$, compounds that then decompose respectively to Ga(OH)O and finally to Ga_2O_3 and directly to Ga_2O_3. Diffuse reflectance Fourier transform IR spectroscopy (DRIFTIR) has been of help in assessing that the reaction consists of parallel dehydration/decomposition processes.

Keywords: *dehydration, gallium nitrate hydrate, thermal decomposition, thermogravimetric analysis*

Introduction

Thermal decomposition of metal nitrates is currently used as the first step in the preparation of metal – dispersed catalysts: metal nitrates are dispersed on suitable porous supports and allowed to decompose usually to their oxides that are, in a subsequent step, reduced to metal by H_2 [1]. The thermal decomposition of metal nitrates has been recently addressed in some interesting papers [2–10] and in a review [11] where the issue of the thermal decomposition of rare earth nitrates was studied not only with respect to the thermodynamic and kinetic aspects of the decomposition in itself but also to some properties of the obtained rare earth oxides.

Quite recently we evaluated different gallium oxide precursors in order to synthesize lithium gallates ($LiGaO_2$ and $LiGa_5O_8$) [12]. So it seemed useful to study the different stages of the thermal decomposition of these precursors in order to individuate the temperature field and the thermal schedule for the preparation of lithium gallates. The present work reports the results obtained in this regard by thermoanalytical (TG-DSC) and spectroscopic (DRIFTIR) techniques.

Experimental

$Ga(NO_3)_3 \cdot xH_2O$ purchased by Aldrich (99.999% purity) has been used as received.

TG measurements have been performed (in duplicate on each sample) with a 951 thermogravimetric analyser (DuPont Inc. USA) connected to a TA2000 computer (by TA Instruments Inc. USA). Samples of about 15 mg were put in a Pt sample holder and subjected to different thermal schedules under nitrogen flow of 10 $dm^3\ h^{-1}$ (the maximum flow recommended by the manufacturer):

- continuous heating from 25 to 650°C with heating rates of 1, 2, 5, 10, 20, and 50 K min^{-1} ('dynamic TG measurements')
- heating at 50 K min^{-1} up to different temperatures, selected on the basis of the results obtained in the dynamic runs, with an isothermal stage appended at the end of the ramp (for isothermal temperatures and times see Table 1 – 'TG measurements with isothermal step')
- heating at 50 K min^{-1} up to different temperatures, selected on the basis of the results obtained in the dynamic runs, followed by an isothermal stage (3 h) and further heating (2 K min^{-1}) up to 600°C (for isothermal temperatures see Table 2 – 'TG measurements with isothermal step and subsequent heating ramp').

The mass values (in % of the initial mass) pertaining to the different reaction stages have been individuated on the basis of the DTG signal and are intended with a precision of ±40 μg that, in the case of our samples (≈15 mg), corresponds to a percentage of 0.25–0.30%.

A TG measurement has been carried out on a sample of pure gallium nitrate by connecting the thermobalance to a FT-IR spectrometer (Model 730 by

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Table 1 Mean value of the mass losses (ΔM_{tot}) recorded during TG measurements with isothermal steps performed at different temperatures (T_{iso}) for different times (t_{iso})

T_{iso}/°C	t_{iso}/h	ΔM_{tot}/%
31.5	67	52.72±0.21
36.5	32	50.45±0.25
41.2	31	56.99±0.19
45.5	12	53.62±0.32
49.9	17	55.69±0.23
55.1	19	57.93±0.25
59.1	7.5	58.68±0.18
64.3	6	58.95±0.31
68.7	8.5	59.96±0.24
73.4	7	59.94±0.26
78.1	5	60.39±0.30
95.9	15	58.61±0.25
113.9	5	59.24±0.27

Nicolet USA). This has been done to analyse the gases evolved during the dehydration/decomposition processes. The sample has been heated at 10 K min^{-1} up to 600°C. Nitrogen (4 dm^3 h^{-1}) has been used both as purging gas for the spectrometer and to sweep the gaseous products from the thermobalance into the FTIR gas cell (kept at 200°C throughout the run). The spectra of the evolved gases have been obtained by Fast Fourier transform of 16 coadded interferograms collected at 8 cm^{-1} resolution.

DSC measurements have been performed with a DSC 910 by DuPont Instruments Inc. USA at 2, 5, 10, 20 and 50 K min^{-1} (under a nitrogen flow of 2 dm^3 h^{-1}, the maximum flow recommended by the manufacturer) between room temperature and ≈600°C on gallium nitrate samples placed in open platinum cups. The DSC cell has been calibrated by using indium and zinc (both Aldrich, 99.99% purity) as calorimetric standards.

The FTIR measurements were recorded by the instrument described above and fitted with a diffuse reflectance attachment (Drift collector by Spectra Tech, UK). The spectra (4000–400 cm^{-1}) have been obtained by FFT of 512 coadded interferograms (resolution 2 cm^{-1}) collected at room temperature and with the spectrometer purged with a dry nitrogen flow of 15 dm^3 min^{-1}. The samples were prepared as physical mixtures of ≈5 mass/mass% gallium nitrate in potassium bromide. Pure potassium bromide was used as background. Spectra have been recorded on gallium nitrate as received and after 3 subsequent thermal treatments in oven (respectively of 15 h at 65°C, 24 h at 135°C and 3 h at 190°C).

Results and discussion

Dynamic TG measurements

The mean value of the final mass of all measurements is 22.93±0.32% which corresponds to 8.49 water molecules per gallium nitrate molecule if the hypothesis is made that gallium oxide is the residue obtained at the end of all the TG scans. Such a hypothesis is confirmed by X-ray powder diffraction (XRPD) of the samples recovered at the end of all runs that appear constituted only by nearly amorphous gallium oxide. Thus it can be concluded that gallium nitrate employed in this work is very likely $Ga(NO_3)_3 \cdot 8H_2O$ that takes on a little amount of surfacial, loosely bound water as it is demonstrated by the fact that the mass loss process starts at room temperature. None of the TG curves shows any plateau with mass values near to those expected for partial or complete dehydration. This point has been confirmed by performing a TG run (10 K min^{-1}) coupled with FTIR analysis of the evolved gas: from Fig. 1 it can be seen that, at ≈80°C, both H_2O (from dehydration) and N_2O_5 (from decomposition) are released during TG run. The same gases are released up to 260°C due to the decomposition of the intermediates $GaO(NO_3)$ and $Ga(OH)_3$ (see the following) to Ga_2O_3.

In the case of the run performed at 1 K min^{-1} (Fig. 2), from the total mass loss a hydration of 8.074 water molecules can be calculated. The first peak of

Table 2 Mean values of the mass obtained from TG measurements performed with 3 h isothermal steps at different temperatures (T_{iso}) followed by a heating ramp at 2 K min^{-1} up to 600°C. The mean mass values at the end of the isothermal step ($M_{fin,iso}$), at the end of the run (M_{fin}) and at two characteristic temperatures T_1 and T_2 (M_{T1} and M_{T2}, see text) are reported

T_{iso}/°C	$M_{fin,iso}$/%	T_1/°C	M_{T_1}/%	T_2/°C	M_{T_2}/%	M_{fin}/%
40	79.07±0.21	116	38.28±0.20	307	26.23±0.14	23.13±0.10
50	74.34±0.14	110 133	41.07±0.22 37.27±0.18	324	25.99±0.19	23.08±0.08
60	49.82±0.22	105	40.57±0.28	279	27.79±0.18	24.86±0.07
70	41.58±0.30	–	–	334	26.22±0.15	23.94±0.09
80	40.63±0.26	–	–	312	26.24±0.22	22.85±0.06
115	41.47±0.10	–	–	225	27.71±0.12	22.38±0.12

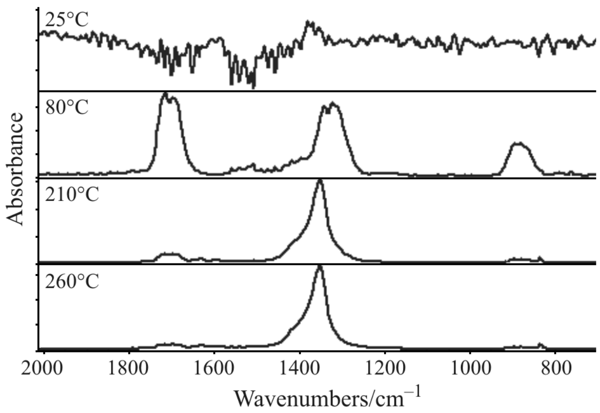

Fig. 1 IR spectra of the gases evolved at different temperatures during a TG run performed on $\mathrm{Ga(NO_3)_3{\cdot}}x\mathrm{H_2O}$ at 10 K min^{-1}

the DTG curve ends at ≈100°C where the mass reaches 41.99±0.18% of its initial value: such a value nearly coincides with the final mass it has to be expected (41.33%) if the following reaction takes place:

$$\mathrm{Ga(NO_3)_3{\cdot}\,8.07H_2O}(s)\rightarrow$$
$$\rightarrow\mathrm{Ga(OH)_2(NO_3)}(s)+\mathrm{N_2O_5}(g)+7.07\ \mathrm{H_2O}(g) \tag{1}$$

The decomposition of $\mathrm{Ga(OH)_2(NO_3)}$ occurs in 2 steps: the first, faster one, implies a mass loss that brings the mass down to 36.07±0.22% (at ≈136°C) and can be accounted for by the following reaction:

$$m\mathrm{Ga(OH)_2(NO_3)}(s)\rightarrow(m/2)\mathrm{Ga(OH)_3}(s)+$$
$$+(m/2)\mathrm{Ga(OH)O}(s)+(m/2)\ \mathrm{N_2O_5}(g) \tag{2}$$

From the mass loss between 100 and 136°C a value of m=0.4398 can be determined.

The second slower step is reflected by the DTG peak at ≈150°C and corresponds to a mass decreasing down to 28.26±0.16% (at 220°C). The mass loss during this step can be accounted for:

$$(1-m)\mathrm{Ga(OH)_2NO_3}(s)\rightarrow[(1-m)/2]\mathrm{Ga(OH)_3}(s)+$$
$$+[(1-m)/2]\mathrm{Ga(OH)O}(s)+[(1-m)/2]\mathrm{N_2O_5}(g) \tag{2'}$$

corresponding to an expected final mass value of 28.53% (*vs.* 28.26±0.21% experimental mass).

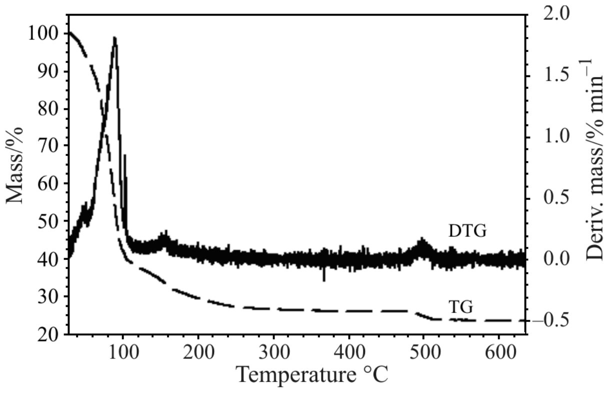

Fig. 2 TG and DTG curves of $\mathrm{Ga(NO_3)_3{\cdot}}x\mathrm{H_2O}$ recorded at 1 K min^{-1}

Then a continuous mass loss process occurs (≈2.33% by mass) up to 460°C, followed by a last mass loss step (see the DTG peak located at about 500°C) of 2.57±0.09%. It is very likely that these mass losses correspond to the decomposition of $\mathrm{Ga(OH)O}$ and $\mathrm{Ga(OH)_3}$ to $\mathrm{Ga_2O_3}$ according to the reaction:

$$(1/2)\mathrm{Ga(OH)_3}(s)+(1/2)\mathrm{Ga(OH)O}(s)\rightarrow$$
$$\rightarrow(1/2)\mathrm{Ga_2O_3}(s)+\mathrm{H_2O} \tag{3}$$

with ΔM=4.49% (experimental 4.90±0.32%).

As concerns the TG/DTG curves recorded at 2 K min^{-1} (Fig. 3a), the first DTG peak ends at ≈141°C with a corresponding mass value of 39.07±0.15% due to the reaction:

$$\mathrm{Ga(NO_3)_3{\cdot}\,8.50H_2O}(s)\rightarrow m\mathrm{Ga(OH)_2NO_3}(s)+$$
$$+(1-m)\mathrm{Ga(NO_3)O(s)}+\mathrm{N_2O_5}(g)+(8.50-m)\mathrm{H_2O}(g) \tag{4}$$

with m=0.6830.

Therefore, by increasing the heating rate, the first step of the decomposition proceeds with the formation of a mixture of $\mathrm{Ga(OH)_2NO_3}$ and $\mathrm{Ga(NO_3)O}$ instead of only $\mathrm{Ga(OH)_2NO_3}$.

Another stage of mass loss occurs that brings the mass at 30.25±0.19% (at ≈200°C). Such a stage can

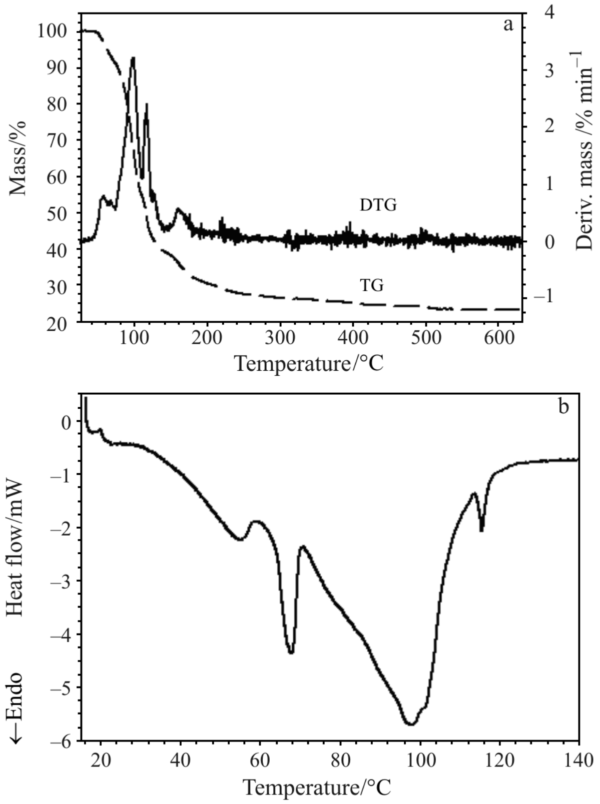

Fig. 3 a – TG and DTG curves of $\mathrm{Ga(NO_3)_3{\cdot}}x\mathrm{H_2O}$ recorded at 2 K min^{-1}; b – DSC curve of $\mathrm{Ga(NO_3)_3{\cdot}}x\mathrm{H_2O}$ recorded at 2 K min^{-1}

be accounted for by the reaction (2) (the expected mass value at the end of the reaction is 30.12%).

From ≈200°C a nearly continuous mass loss process (experimental 7.27±0.10%) starts that corresponds to the decomposition of $Ga(NO_3)O$, $Ga(OH)_3$ and $Ga(OH)O$ to Ga_2O_3 (expected 7.20%). A parallel DSC scan has been performed at 2 K min^{-1} (Fig. 3b): a multiple endothermic peak is present between 30 and 140°C with four maxima (at ≈50, 70, 100 and 115°C) and a total area yielding 284 kJ mol^{-1} gallium nitrate hydrate.

The run performed at 5 K min^{-1} (Fig. 4a) shows a DTG peak that ends at ≈185°C with M=31.70±0.17% i.e. well below the value expected (M≈40%, note that the hydration of the sample is 8.844 water molecules) for the formation of $Ga(OH)_2(NO_3)$. The observed mass value can be explained by admitting that the following reaction occurs:

$$Ga(NO_3)_3{\cdot}8.84H_2O(s)\rightarrow hGa(OH)_3(s)+ \\ +kGaO(NO_3)(s)+[(3-k)/2]N_2O_5(g)+ \\ +[(17.668-3h)/2]H_2O(g) \qquad (5)$$

with k=0.3952 and h=0.6048.

Therefore, by further increasing the heating rate, the mechanism of the decomposition proceeds with formation of $Ga(OH)_3$ and $GaO(NO_3)$ instead of a mixture of $Ga(OH)_2NO_3$ and of $Ga(NO_3)O$.

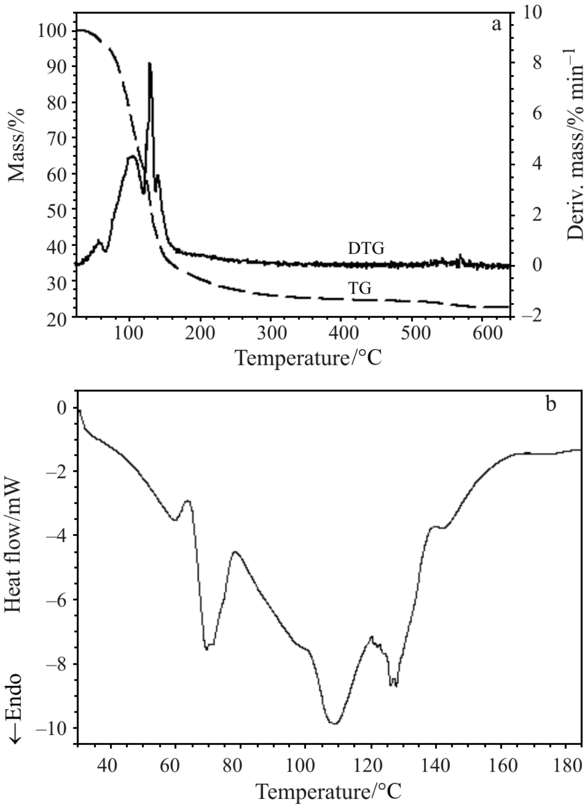

Fig. 4 a – TG and DTG curves of $Ga(NO_3)_3{\cdot}xH_2O$ recorded at 5 K min^{-1}; b – DSC curve of $Ga(NO_3)_3{\cdot}xH_2O$ recorded at 5 K min^{-1}

Then the mass further decreases down to M=24.10±0.12% (at 501°C), fairly similar to the value one would expect (M=23.93%) if the following reactions would occur:

$$hGa(OH)_3(s)\rightarrow hGa(OH)O(s)+hH_2O(g) \qquad (6)$$

$$kGaO(NO_3)(s)\rightarrow(k/2)Ga_2O_3(s)+(k/2)N_2O_5(g) \qquad (7)$$

The last step is very likely:

$$hGa(OH)O(s)\rightarrow(h/2)Ga_2O_3(s)+(h/2)H_2O(g) \qquad (8)$$

that should lead to a final mass value of 22.62% (*vs*. 22.58±0.06% experimental).

A parallel DSC scan performed at 5 K min^{-1} (Fig. 4b) shows a multiple endothermic peak within 40 and 180°C with five maxima (at ≈55, 70, 109, 128 and 140°C) and a total area yielding 278 kJ mol^{-1} gallium nitrate hydrate. The difference with respect to the enthalpy measured at 2 K min^{-1} is quite low so that it does not reflect the fact that different products are obtained at the end of the peak (a mixture of $Ga(OH)_2NO_3$ and $GaO(NO_3)$ at 2 K min^{-1}; a mixture of $GaO(NO_3)$ and $Ga(OH)_3$ at 5 K min^{-1}).

The run performed at 10 K min^{-1} (Fig. 5a) on a sample of $Ga(NO_3)_3{\cdot}8.84H_2O$ shows a DTG peak which ends at ≈210°C with M=30.76±0.11%. Such a mass loss corresponds to the reaction (5) with k=0.2567 and h=0.7433.

Thus, by increasing heating rate, the relative amount of $Ga(OH)_3$ produced increases with respect to that of $Ga(NO_3)O$. The reaction proceeds, as at 5 K min^{-1}, with the decomposition of $Ga(NO_3)O$ to Ga_2O_3 and of $Ga(OH)_3$ to $Ga(OH)O$. These reactions complete at 498°C with an experimental M=23.88±0.16% *vs*. (calculated) 24.19%. The final step corresponds to the thermal decomposition of $Ga(OH)O$ to Ga_2O_3 (experimental M=22.58±0.04% *vs*. calculated M=22.58%). The DSC endothermic peak (Fig. 5b) shows five maxima from 40 to 200°C (T≈65, 75, 111, 135 and 155°C) and the enthalpy of reaction is now 296 kJ mol^{-1} of gallium nitrate hydrate. The slightly higher value with respect to the peak recorded at 5 K min^{-1} can be justified by considering that the peak refers to the formation, under the peak, of different relative amounts of $GaO(NO_3)$ and $Ga(OH)_3$. The runs performed at 20 K min^{-1} ($Ga(NO_3)_3{\cdot}8.57H_2O$ – Fig. 6a) and at 50 K min^{-1} ($Ga(NO_3)_3{\cdot}8.26H_2O$ – Fig. 7a) show DTG peaks ending at ≈350°C with M≈25%, value that nearly corresponds to the formation of the unique intermediate $Ga(OH)O$ that fully decomposes, at high temperatures, to Ga_2O_3. In this cases the relevant DSC peaks (Figs 6b and 7b) yield, from 50 to 350°C, 350–380 kJ mol^{-1} gallium nitrate hydrate, values that now significantly reflect the larger extent of decomposition under the peaks.

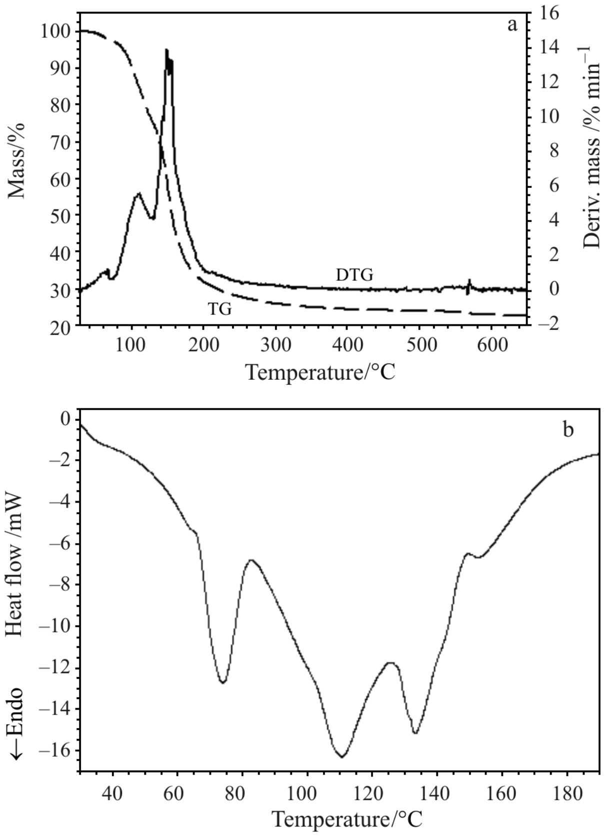

Fig. 5 a – TG and DTG curves of $Ga(NO_3)_3 \cdot xH_2O$ recorded at 10 K min^{-1}; b – DSC curve of $Ga(NO_3)_3 \cdot xH_2O$ recorded at 10 K min^{-1}

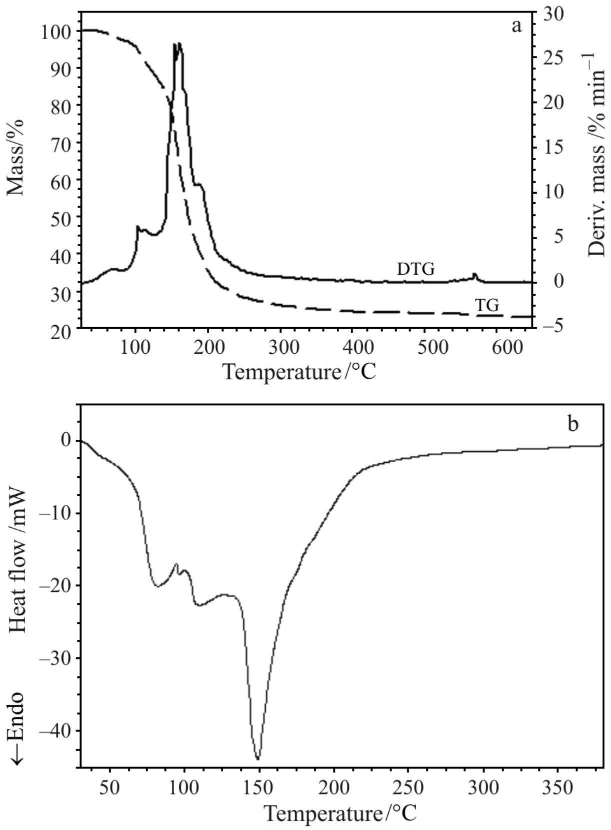

Fig. 6 a – TG and DTG curves of $Ga(NO_3)_3 \cdot xH_2O$ recorded at 20 K min^{-1}; b – DSC curve of $Ga(NO_3)_3 \cdot xH_2O$ recorded at 20 K min^{-1}

TG measurements with isothermal step and TG measurements with isothermal step and subsequent heating ramp

It has been shown that the mechanism of $Ga(NO_3)_3 \cdot xH_2O$ decomposition depends on heating rate. To try and assess what are the intermediates during the reaction course, a series of TG measurements has been performed where samples of $Ga(NO_3)_3 \cdot xH_2O$ have been heated quickly to temperatures in the range 30–115°C and maintained at these temperatures for different times (Table 1). In some cases, after 3 h isotherm, heating has been resumed at 2 K min^{-1} up to 650°C (Table 2).

By observing the mass losses reported in Table 1 it can be seen that they are fairly constant for isothermal temperatures between 55 and 115°C. The mean value of the residual mass is 40.79±0.83% that well agrees with the mass value (40.55%) expected for the formation of $Ga(OH)_2NO_3$. Therefore the TG measurements with isothermal step indicate that $Ga(OH)_2NO_3$ is the first intermediate compound in the course of the decomposition.

From the results reported in Table 2, the following remarks can be proposed:

- at isothermal temperatures of 70°C and higher, a nearly constant mass value close to the value expected for the formation of $Ga(OH)_2NO_3$ is attained (41.23±0.52%) at the end of the isothermal period;
- for isothermal temperatures of 50 and 60°C, mass values close to that expected for the formation of $Ga(OH)_2NO_3$ are attained only after heating is resumed up to respectively T_1=110°C and 105°C;
- in the case of the lowest isothermal temperature (40°C), a mass loss value is attained (38.28±0.20%) at T_1=116°C that is midway between the values expected for the formation of $Ga(OH)_2NO_3$ (40.55%) and of $Ga(NO_3)O$ (36.14%). A mass value (37.27±0.18%) more shifted towards that expected for the formation of $Ga(NO_3)O$ is reached at higher temperatures (T_1=133°C) when heating is resumed after the isothermal step at 50°C;
- all measurements show, at temperatures T_2 ranging from 225 to 334°C, a mass value M_{T2}=26.70±0.82% that fairly agrees with the mass value expected (27.33%) for the reaction

$$Ga(OH)_2NO_3(s) \rightarrow (2/3)Ga(OH)_3(s) + \\ +(1/6)Ga_2O_3(s) + (1/2)N_2O_5(g);$$

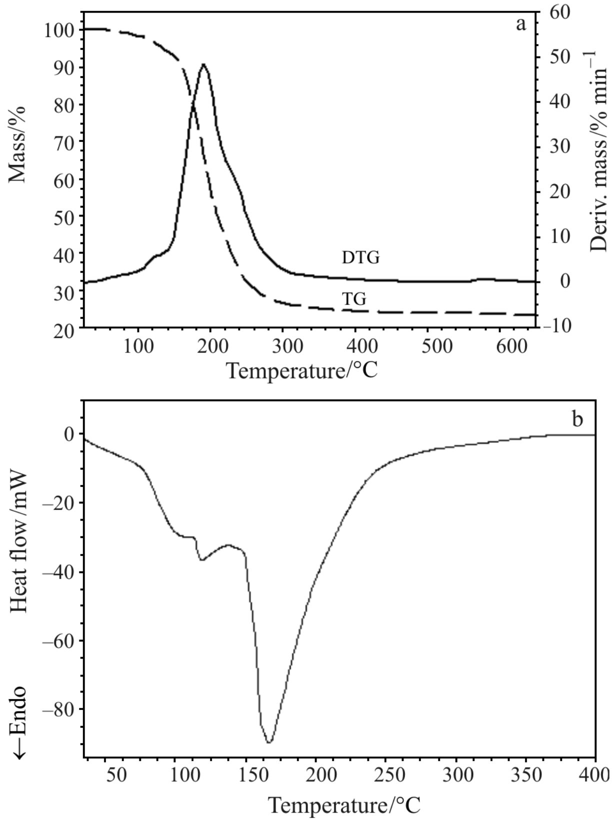

Fig. 7 a – TG and DTG curves of $Ga(NO_3)_3{\cdot}xH_2O$ recorded at 50 K min^{-1}; b – DSC curve of $Ga(NO_3)_3{\cdot}xH_2O$ recorded at 50 K min^{-1}

- for the runs with isotherms at 40, 50, 80 and 115°C, the mean final mass value (22.86±0.34%) is close to that expected (M=22.92%) for the reaction

$$(2/3)Ga(OH)_3(s)\rightarrow(1/3)Ga_2O_3(s)+H_2O(g);$$

- for the measurements with isotherms at 60 and 70°C, the mean value is 24.40±0.65%, that coincides with the one expected (M=24.40%) for the reaction

$$(2/3)Ga(OH)_3(s)\rightarrow(2/3)Ga(OH)O(s)+(2/3)H_2O(g).$$

Figure 8 reports the DRIFT-IR spectrum of commercial gallium nitrate (spectrum 1) along with those of samples that underwent different thermal treatments (spectra 2, 3 and 4): it can be seen that both hydroxyl (see the stretching broad band centered at about 3500 cm^{-1}) and nitrate (see the most intense stretching band located between 1390 and 1350 cm^{-1}) groups are present in samples of gallium nitrate heated up to 200°C for prolonged times. If attention is focussed on the free hydroxyl-stretching peak at ≈3750 cm^{-1} (due to the non H-bonded OH groups) it can be seen that its intensity increases by increasing the temperature of annealing (spectra 2, 3 and 4). As regards the stretching band of the nitrate group, its maximum frequency shifts towards higher wavenumbers (from ≈1360 to 1382 cm^{-1}) by treating the sample at 135°C i.e. at a temperature where, according to the TG evidence, $Ga(OH)_2NO_3$ is formed. On the other hand the two peaks at ≈1046 and ≈836 cm^{-1} (due to the nitrate groups) show an intensity that decreases by increasing the annealing temperature so allowing one to conclude that the intermediates containing OH groups [$Ga(OH)O$ and $Ga(OH)_3$] become prevailing over those containing NO_3^- groups [$Ga(OH)_2NO_3$ and $Ga(NO_3)O$]. The differences observed between the spectra near 2400 and 2750 cm^{-1} are due respectively to fluctuations of the carbon dioxide level in the spectrometer and to the decrease of the intensity of the overtone of the most intense stretching band of the nitrate (1350–1390 cm^{-1}).

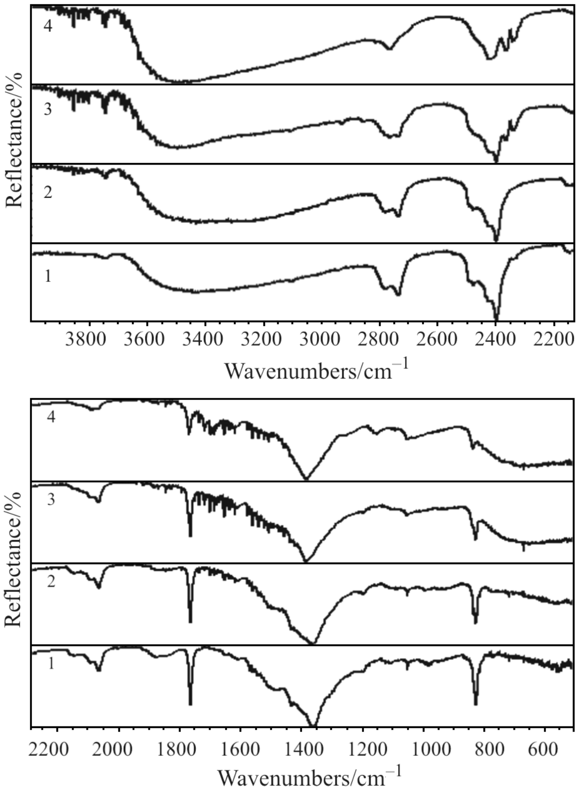

Fig. 8 DRIFT-IR spectra recorded on samples of commercial $Ga(NO_3)_3{\cdot}xH_2O$ (spectrum 1) subjected to subsequent thermal treatments: 15 h at 65°C (spectrum 2); 24 h at 135°C (spectrum 3); 3h at 190°C (spectrum 4)

Conclusions

- TG measurements performed at different heating rates allowed to conclude that 8 water molecules are present in the samples of gallium nitrate employed in this work $Ga(NO_3)_3{\cdot}8H_2O$;
- From TG/FTIR gas analysis it could be seen that both H_2O (from dehydration) and N_2O_5 (from decomposition) are released throughout the whole TG run. This means that anhydrous gallium nitrate is not thermodynamically stable;

- The dehydration/decomposition process occurs with a mechanism that depends on heating rate: $Ga(OH)_2NO_3$ is the first intermediate obtained at 1 K min^{-1}. By increasing heating rate (2 K min^{-1}) $GaONO_3$ also forms while, by still higher heating rates (5 and 10 K min^{-1}), $Ga(OH)_3$ is obtained, instead of $Ga(OH)_2NO_3$, together with $GaONO_3$. Such a dependence of the decomposition patterns on heating rate is confirmed by the DRIFT-IR spectra but it is evident only at high heating rates in the DSC runs;
- The intermediates $Ga(OH)_2NO_3$ and $Ga(OH)_3$ decompose to Ga(OH)O and finally to Ga_2O_3 whereas $GaONO_3$ decomposes directly to Ga_2O_3;
- At heating rates of 20 and 50 K min^{-1}, Ga(OH)O is the only intermediate leading to the formation of Ga_2O_3;
- From TG isothermal experiments it is confirmed that the first intermediate obtained is $Ga(OH)_2NO_3$. This compound forms if enough time (2–3 h) is elapsed at a temperature of at least 70°C or by heating at a moderate scan rate (2 K min^{-1}) up to 105–110°C after 2–3 h annealing at lower temperatures (50–60°C). By even lower annealing temperature (40°C) the formation of $Ga(OH)_2NO_3$ is nearly skipped and the anhydrous intermediate $Ga(NO_3)O$ forms.

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