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Preparation and Properties of Yttrium, Lanthanum, and Lanthanide *m*-Nitrobenzoates

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The conditions of the formation of rare earth element *m*-nitrobenzoates were studied and their quantitative composition and solubilities in water and benzene at 298 K were determined (their solubilities are in the orders of $10^{-3}\,\mathrm{mol\,dm^{-3}}$ and $10^{-4}\,\mathrm{mol\,dm^{-3}}$, respectively). From the values of solubilities in water the solubility products were established (they are in the orders of $10^{-10}\,\mathrm{mol^4\,dm^{-12}}$).

The IR and X-ray spectra for the prepared complexes and the dehydrated rare earth *m*-nitrobenzoates were recorded. All obtained complexes were found to be crystalline compounds. The conditions of thermal decomposition for complexes were also studied. It was found that on heating above 573 K the complexes decompose explosively and undergo a melting process at the same time. Therefore the thermal decomposition was carried out in the temperature range 273–573 K. The obtained results show that during dehydration process no transformation of the nitro group to nitrito occurs.

(Keywords: Complexes; Rare earth elements; Lanthanides)

Darstellung und Eigenschaften von Y, La *und Lanthaniden-meta-Nitrobenzoaten*

Die Bedingungen zur Darstellung von Y-, La- und Lanthanidenmeta-Nitrobenzoaten wurden untersucht. Ihre quantitative Zusammensetzung und ihre Wasserlöslichkeit bei 298 K wurden bestimmt (die Löslichkeit ist in der Größenordnung $10^{-3}\,\mathrm{mol\,dm^{-3}}$). Die Infrarot- und Röntgenspektren der erhaltenen Komplexe sowie der *m*-Nitrobenzoate der seltenen Erden nach der Dehydratisierung wurden gemessen und dabei festgestellt, daß es sich stets um kristalline Verbindungen handelt. Das thermische Verhalten der erhaltenen Komplexe wurde festgestellt: Sie zerfallen über 573 K explosiv und schmelzen zugleich. Der thermische Zerfall der erhaltenen *m*-Nitrobenzoate der seltenen Erden wurde im Temperaturbereich von 273–573 K untersucht. Es wurde festgestellt, daß die Y-, La- und Lanthaniden-*m*-Nitrobenzoate bei Temperaturzunahme oder im Dehydratisierungsprozeß keiner Umgruppierung in entsprechende Nitritoverbindungen unterliegen.

Introduction

m-Nitrobenzoic acid is cream coloured, crystalline solid, readily soluble in HCl and sparingly soluble in H_2O [1]. It crystallizes in a monoclinic system [2–5] and occurs in dimer form.

A survey of literature shows that the salts of *m*-nitrobenzoic acid with the following cations: NH_4^+, Na^+, K^+, Sc^{3+} [1, 6–9], Ba^{2+} [10], Ca^{2+} [11], Tl^+ [12–14], Cu^{2+} [15–24], La^{3+} [9, 25], Ce^{3+}, Pr^{3+}, Nd^{3+}, Sm^{3+} [25] and Th^{4+} [26] have been isolated in the solid state and some of their properties studied. There are no reports on all of the rare earth element *m*-nitrobenzoates; the aim of this work was to obtain them in solid state (with a metal to ligand ratio of 1:3) and to examine some of their physico-chemical properties.

Experimental

m-Nitrobenzoates of yttrium, lanthanum and lanthanides from Ce(III) to Lu were prepared by adding the equivalent quantities of 0.2 *M* ammonium *m*-nitrobenzoate (*pH* ~ 5) to a hot solution containing the rare earth element nitrates and crystallizing at 293 K. The solid formed were filtered off, washed with hot water to remove ammonium ions and dried at 303 K to constant mass.

Carbon, hydrogen and nitrogen microanalysis data for these complexes were determined by elemental analysis with V_2O_5 as oxidizing agent. The rare earth element contents were established by the oxalic acid method (Table 1). The content of crystallization water molecules was determined from the curve of TG and by isothermal heating these complexes at 573 K.

Table 1. *Analytical data*

Compound $L = C_7H_4NO_4$	% C		% N		% H		% Me	
	Calcd.	Found	Calcd.	Found	Calcd.	Found	Calcd.	Found
$YL_3 \cdot 2\,H_2O$	40.45	40.60	6.74	6.54	2.56	2.98	14.27	14.16
$LaL_3 \cdot 2.5\,H_2O$	36.95	36.93	6.15	6.13	2.49	2.35	20.37	20.44
$CeL_3 \cdot 2.5\,H_2O$	36.88	36.70	6.14	6.16	2.48	2.43	20.51	20.41
$PrL_3 \cdot 2\,H_2O$	37.33	37.47	6.22	6.39	2.37	2.57	20.87	21.01
$NdL_3 \cdot 2\,H_2O$	37.15	37.59	6.19	6.12	2.35	2.51	21.12	21.25
$SmL_3 \cdot 2\,H_2O$	36.82	36.53	6.13	6.23	2.33	2.55	21.95	22.00
$EuL_3 \cdot 2\,H_2O$	36.73	36.79	6.12	6.14	2.33	2.35	22.14	22.33
$GdL_3 \cdot 2\,H_2O$	36.45	36.34	6.07	6.04	2.31	2.32	22.74	22.69
$TbL_3 \cdot 2\,H_2O$	36.15	36.32	6.06	6.04	2.30	2.26	22.93	23.00
$DyL_3 \cdot 2\,H_2O$	36.18	36.14	6.03	6.09	2.29	2.23	23.33	23.30
$HoL_3 \cdot 2\,H_2O$	36.05	36.04	6.00	5.96	2.28	2.21	23.59	23.89
$ErL_3 \cdot 2\,H_2O$	35.93	35.77	5.98	5.95	2.28	2.53	23.85	23.59
$TmL_3 \cdot 2\,H_2O$	35.82	35.86	5.97	5.71	2.27	2.22	24.07	24.17
$YbL_3 \cdot 2\,H_2O$	35.64	35.54	5.94	5.84	2.26	2.21	24.47	24.47
$LuL_3 \cdot 2\,H_2O$	35.54	35.44	5.92	5.98	2.25	2.26	24.67	24.57

IR spectra for the prepared complexes, the spectra for *m*-nitrobenzoic acid, sodium *m*-nitrobenzoate, the dehydrated *m*-nitrobenzoates of yttrium, lanthanum and lanthanides and for sodium nitrite were recorded as KBr discs on a UR-20 spectrophotometer (range 4 000–400 cm^{-1}). Some of the experimental data are presented in Table 2.

The X-ray diffraction patterns were taken on a DRON-2 diffractometer using Ni filtered CuK_α radiation. The measurements were made within the range $2\theta = 4$–80° by means of the *Debye-Scherrer* method.

The thermal stability of the complexes was studied by the use of TG, DTA and DTG techniques. The measurements were made with a Q-1500 D derivatograph at a heating rate of 2.5 deg min^{-1}. Test samples were heated at a sensitivity TG—50 mg, DTG—500 μV, DTA—500 μV (Table 3).

The solubilities of *m*-nitrobenzoates of Y, La and lanthanides in water and benzene at 298 K were determined by measuring the concentration of Ln^{3+} ions in saturated solutions by the oxalate method (Table 4).

Results and Discussion

m-Nitrobenzoates of rare earth elements were obtained in crystalline form with the colour characteristic for Ln^{3+} ions. They were found to form hydrated salts with a metal to ligand ratio of 1 : 3 and the general formula: $Ln(C_7H_4NO_4)_3 \cdot nH_2O$, where $n = 2$ for Ln = Y, Pr-Lu and $n = 2.5$ for La, Ce.

The infrared spectrum for *m*-nitrobenzoic acid shows the following absorption bands: sharp absorption bands of the OH group between 3 400–2 400 cm^{-1}, a strong band of COOH at 1 685 cm^{-1}, the bands assigned to asymmetric and symmetric vibrations of the NO_2 group occuring at 1 525 cm^{-1} and 1 350 cm^{-1}, respectively, the deformation vibration bands of the aromatic ring at around 1 480 cm^{-1} and 1 445 cm^{-1} and the band arising from the stretching vibration of C—O group at 1 410 cm^{-1}. The band of C—H deformation vibration appears at 1 290 cm^{-1}. The bands at 830 cm^{-1} and 810 cm^{-1} are attributed to the C—N streching vibration and the bands at 725 cm^{-1} and 705 cm^{-1} are indicative of out-of-plane deformation vibration of C—H group.

The IR spectra for all the complexes are quite similar and their analysis confirms the formulations of *m*-nitrobenzoates obtained as nitro complexes. The water leads to the characteristic absorptions with the maxima in the 3 450–3 420 cm^{-1} range. When the acid is transformed to the salt a change in the spectra appears. All the spectra for *m*-nitrobenzoates of rare earth elements show no bands at 1 685 cm^{-1} characteristic for COOH, which is indicative of the absence of the free COOH groups in the complexes. The bands due to asymmetric and symmetric vibrations of the COO^- group are observed at 1 605–1 590 cm^{-1} and 1 425–1 405 cm^{-1}, respectively. The bands occuring at 1 545–1 525 cm^{-1} and 1 355–1 345 cm^{-1} are assigned to asymmetric and symmetric vibration of the NO_2 group, respectively. The bands due to stretching vibrations of the C—H group for *meta*-disubstituted compounds are observed at 1 085–1 075 cm^{-1} and the bands arising from the stretching vibration of the C—N group appear at 845–835 cm^{-1}. The bands at 725 cm^{-1} are indicative of vibrations of the neighbouring hydrogen atoms in the benzene ring substituted in 1 : 3 position. The absorption bands at 425–410 cm^{-1} confirm the ionic bond of metal-oxygen. For all *m*-nitrobenzoates of yttrium, lanthanum and lanthanides no shift changes of these bands are observed. Accordingly, it is possible to suggest that *m*-nitrobenzoic acid forms complexes of the same type and similar stabilities with rare earth elements.

Table 2. *Frequencies of maxima of the absorption bands of* COO^- *vibrations for m-nitrobenzoates of yttrium, lanthanum and lanthanides and for sodium m-nitrobenzoate*

Complex $L = C_7H_4NO_4$	$\tilde{\nu}_{asym}$ OCO (cm^{-1})	$\tilde{\nu}_{sym}$ OCO (cm^{-1})	$\Delta\tilde{\nu}$
$YL_3 \cdot 2\,H_2O$	1 590	1 415	175
$LaL_3 \cdot 2.5\,H_2O$	1 590	1 405	185
$CeL_3 \cdot 2.5\,H_2O$	1 590	1 405	185
$PrL_3 \cdot 2\,H_2O$	1 590	1 405	185
$NdL_3 \cdot 2\,H_2O$	1 590	1 410	180
$SmL_3 \cdot 2\,H_2O$	1 590	1 410	180
$EuL_3 \cdot 2\,H_2O$	1 590	1 410	180
$GdL_3 \cdot 2\,H_2O$	1 590	1 410	180
$TbL_3 \cdot 2\,H_2O$	1 590	1 410	180
$DyL_3 \cdot 2\,H_2O$	1 595	1 415	180
$HoL_3 \cdot 2\,H_2O$	1 595	1 415	180
$ErL_3 \cdot 2\,H_2O$	1 600	1 420	180
$TmL_3 \cdot 2\,H_2O$	1 600	1 420	180
$YbL_3 \cdot 2\,H_2O$	1 600	1 420	180
$LuL_3 \cdot 2\,H_2O$	1 600	1 425	175
NaL	1 605	1 390	215

The magnitudes of separation $\Delta\bar{\nu}$ between the frequencies due to $\bar{\nu}_{asym}$ OCO and $\bar{\nu}_{sym}$ OCO in the complexes are lower than in the sodium salt, which indicates a smaller degree of ionic bond in these complexes compared to that of sodium one (Table 2). Having a greater ionic potential than sodium ion, lanthanide ions influence the ligand more strongly causing its greater deformation. The same values of $\Delta\bar{\nu}$ reveal the similar degree of ionic bond in the complexes. The directions of shifts in the frequencies due to $\bar{\nu}_{asym}$ OCO and $\bar{\nu}_{sym}$ OCO are lower (or remain almost the same) and higher, respectively, relative to those in sodium *m*-

nitrobenzoate. Therefore it is possible to assume that the carboxylate ion is a chelating, bidentate ligand. In the IR spectra for all *m*-nitrobenzoates of Y, La and lanthanides the relatively lower intensities of the bands at 725 cm^{-1} compared to that for *m*-nitrobenzoic acid indicate the weak interaction of the benzene ring with lanthanide ions.

The small values of the displacements in the positions of ν_{asym} (NO_2) and ν_{sym} (NO_2) bands for *m*-nitrobenzoates of rare earth elements relative to those in *m*-nitrobenzoic acid are indicative of the weak coordination of NO_2 group to lanthanide ions.

As monodentate ligand the nitrite ion may bond either through nitrogen atom forming the nitro complex or through one of the oxygen atom yielding a nitrito one. The general effect of coordination via nitrogen is to raise the frequencies of both ν_{asym} (NO_2) and ν_{sym} (NO_2) from the free ion values of ~ 1 328 cm^{-1} and 1 260 cm^{-1}, respectively [29]. To make sure if the obtained rare earth element *m*-nitrobenzoates do not convert to nitrito isomers (during precipitation or on heating to 573 K [28, 29]) the IR spectra for hydrated and dehydrated *m*-nitrobenzoates of Y, La and lanthanides and for sodium nitrite were recorded. The results show that the *m*-nitrobenzoates of rare earth elements isolated as solid form nitro complexes and also no transformation process of the nitro group to nitrito takes place with rising temperature.

In order to examine the crystal structure of hydrated and dehydrated *m*-nitrobenzoates of Y, La and lanthanides and oxides obtained from ignition of *m*-nitrobenzoates and oxalates of rare earth elements the X-ray spectra were recorded. *m*-Nitrobenzoates of rare earth elements were found to be crystalline compounds characterized by low symmetry and the large sizes of unit cells. The *m*-nitrobenzoate of praseodymium has the same structure as the neodymium one. Similarly, *m*-nitrobenzoates of Sm, Gd and Tb appear to be isostructural compounds. X-ray spectra reveal that ytterbium and lutetium *m*-nitrobenzoates of the remaining rare earth elements do not show such properties (Figs. 1, 2). The dehydrated *m*-nitrobenzoates are also crystalline compounds but they have a less degree of crystallinity than the hydrated ones. No changes of their structures relative to the hydrated salts were observed. Accordingly, it is possible to assume that when heated in the temperature range 273–573 K all *m*-nitrobenzoates of rare earth elements do not form the nitrito complexes and the loss of crystallization water molecules does not modify their crystalline structures. The structures of all rare earth element oxides (with the exception of La_2O_3) remain the same irrespective of the way of their formation. Only the structure of La_2O_3 formed by roasting lanthanum *m*-nitrobenzoate differs essentially from the hexagonal structure of La_2O_3 obtained from the ignition of lanthanum oxalate.

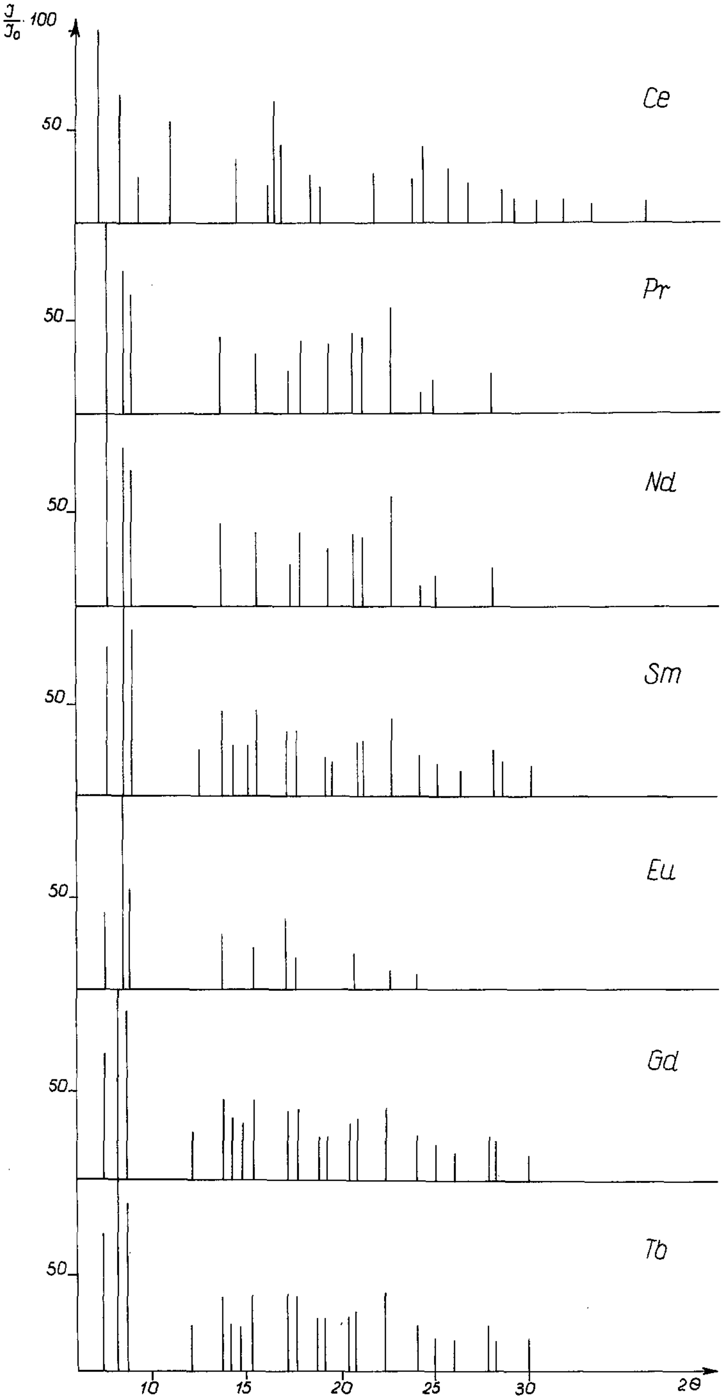

Fig. 1. Radiograms of *m*-nitrobenzoates of light lanthanides, Gd and Tb

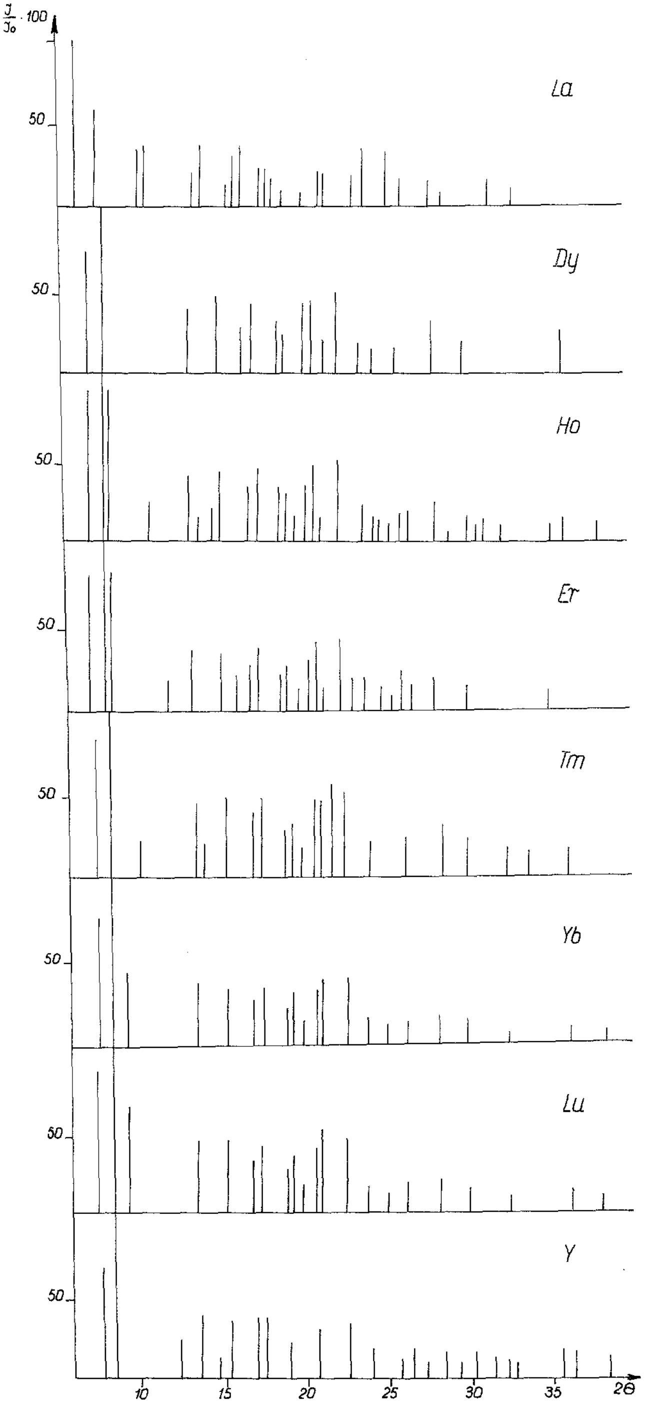

Fig. 2. Radiograms of *m*-nitrobenzoates of La, heavy lanthanides and Y

The hydrates of *m*-nitrobenzoates of rare earth elements are stable up to 573 K, after which they are explosively decomposed at the melting point. Therefore the thermal decomposition study for these complexes was carried out in the temperature range 273–573 K. The temperature data for dehydration process and the values of dehydration energies determined for those complexes are collected in Table 3. Figure 3 exhibits the derivatogram of yttrium *m*-nitrobenzoate. From the obtained results it appears that *m*-nitrobenzoates of Y, La and lanthanides are dehydrated in one step. The dehydration process connected with the strong endothermic effect proceeds in the temperature range 293–445 K. The least thermally stable complexes are the hydrates of cerium and lanthanum which start to lose the crystallization water at 293 K and 296 K, respectively. The most thermally stable are the samarium and terbium *m*-nitrobenzoates that release crystallization water at temperatures of 345 K and 347 K, respectively. In the temperature range from 433 K (for Er) to 480 K (for Ho) the anhydrous salts of rare earth element *m*-nitrobenzoates are formed.

Table 3. *Temperature ranges of the dehydration process of m-nitrobenzoates of Y, La and lanthanides*

Complex $L = C_7H_4NO_4$	ΔT_1[a] (K)	Loss of weight %		$n H_2O$	T_{max}[b] (K)	T_k(K)[c]	E(kJ m^{-1})
		Calcd.	Found				
$YL_3 \cdot 2H_2O$	333–426	5.77	5.80	2	388	450	48.50
$LaL_3 \cdot 2.5H_2O$	296–434	6.59	6.60	2.5	400	467	21.44
$CeL_3 \cdot 2.5H_2O$	293–421	6.58	6.40	2.5	386	467	36.42
$PrL_3 \cdot 2H_2O$	303–433	5.33	5.20	2	395	447	56.37
$NdL_3 \cdot 2H_2O$	345–432	5.30	5.40	2	393	459	48.13
$SmL_3 \cdot 2H_2O$	345–436	5.20	5.20	2	396	458	46.15
$EuL_3 \cdot 2H_2O$	334–445	5.24	5.40	2	398	478	50.27
$GdL_3 \cdot 2H_2O$	313–429	5.20	5.20	2	391	429	45.87
$TbL_3 \cdot 2H_2O$	347–427	5.19	5.20	2	387	463	49.50
$DyL_3 \cdot 2H_2O$	333–419	5.16	5.20	2	384	461	57.15
$HoL_3 \cdot 2H_2O$	321–407	5.15	5.00	2	381	480	65.58
$ErL_3 \cdot 2H_2O$	307–407	5.13	5.23	2	374	433	47.21
$TmL_3 \cdot 2H_2O$	322–397	5.11	5.20	2	374	451	51.14
$YbL_3 \cdot 2H_2O$	315–406	5.09	5.00	2	367	443	31.38
$LuL_3 \cdot 2H_2O$	313–390	5.07	5.20	2	365	440	41.52

[a] ΔT_1: temperature range corresponding to the endothermic loss of molecules of crystallization water

[b] T_{max}: temperature of the maximum of the endothermic effect in the DTA curve

[c] T_k: temperature of anhydrous salt formation

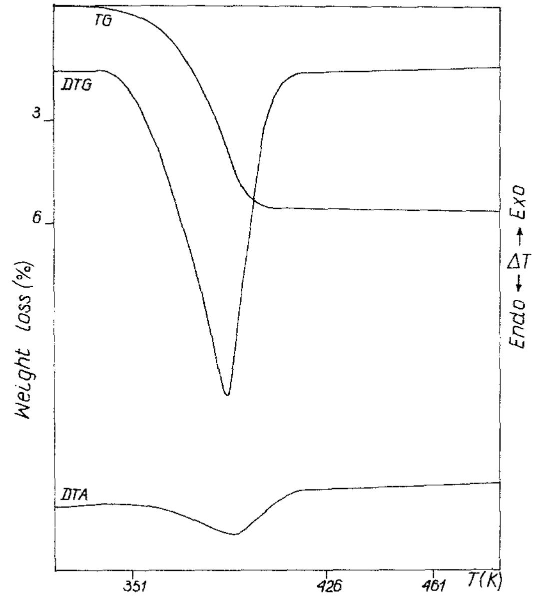

Fig. 3. TG, DTG and DTA curves of: $Y(C_7H_4NO_4)_3 \cdot 2\,H_2O$

Considering the temperature and the course of the dehydration process it is possible to suppose that the molecules of water in *m*-nitrobenzoates of rare earth elements are similarly bound. The small values of the dehydration temperatures and the loss of crystallization water molecules in one step indicate that it is probably outer sphere water. The low initial temperatures of dehydration for *m*-nitrobenzoates of La, Ce, Pr and Er reveal that crystallization water molecules are weakly bonded in these complexes whereas its highest values for terbium, samarium and neodymium *m*-nitrobenzoates suggest that crystallization water molecules are the most strongly bonded ones in these compounds. From TG and DTA curves the activation energies of dehydration reactions were calculated by means of the *Fatieev* and *Pletniew* method [30]. The values of the activation energy of dehydration reactions for all *m*-nitrobenzoates of rare earth elements (with the exception of lanthanum and cerium *m*-nitrobenzoates) are connected with the loss of two crystallization water molecules and the differences between them may suggest that molecules of outer sphere-water are probably coordinated to lanthanide ions with different force. The smallest values of activation energy for lanthanum and ytterbium *m*-nitrobenzoates may indicate that water molecules are the most weakly coordinated ones in these complexes whereas its highest value for holmium *m*-nitrobenzoates may show that they are presumably most strongly coordinated to holmium ion.

The solubilities of *m*-nitrobenzoates of Y, La and lanthanides in water and benzene at 298 K were measured. From the values of solubilities in water the solubility products were determined (Table 4). The solubilities of *m*-nitrobenzoates of rare earth elements in water and benzene are in the order of 10^{-3} mol dm^{-3} and 10^{-4} mol dm^{-3}, respectively and the solubility products of 10^{-10} mol^4 dm^{-12}. The *m*-nitrobenzoate of La is the most soluble salt while the *m*-nitrobenzoate of Er is the least soluble one. *m*-Nitrobenzoates of Y, La and lanthanides are better soluble in polar solvents. The values of solubility of *m*-nitrobenzoates of heavy lanthanides in benzene decrease gradually with the atomic number whereas for light lanthanides such a regularity is not observed. The *m*-nitrobenzoate of La is the best soluble salt in benzene while *m*-nitrobenzoate of Y is the least soluble one.

Table 4. *Solubility of m-nitrobenzoates of rare earth elements in water and benzene at 293 K and their solubility products in water*

Complex $L = C_7H_4NO_4$	Solubility as anhydrous salt mol dm^{-3}		Solubility products $\times 10^{-10}$ mol^4 dm^{-12}
	in H_2O $\times 10^{-3}$	in C_6H_6 $\times 10^{-4}$	
$YL_3 \cdot 2H_2O$	1.86	1.77	3.23
$LaL_3 \cdot 2.5H_2O$	2.40	5.52	8.95
$CeL_3 \cdot 2.5H_2O$	2.28	1.80	7.29
$PrL_3 \cdot 2H_2O$	1.93	3.52	3.80
$NdL_3 \cdot 2H_2O$	1.90	3.60	3.54
$SmL_3 \cdot 2H_2O$	1.45	4.00	1.22
$EuL_3 \cdot 2H_2O$	1.34	5.00	0.89
$GdL_3 \cdot 2H_2O$	1.57	4.41	1.66
$TbL_3 \cdot 2H_2O$	1.57	4.20	1.66
$DyL_3 \cdot 2H_2O$	1.38	4.10	0.97
$HoL_3 \cdot 2H_2O$	1.56	3.80	1.59
$ErL_3 \cdot 2H_2O$	1.13	3.50	0.44
$TmL_3 \cdot 2H_2O$	1.48	2.90	1.29
$YbL_3 \cdot 2H_2O$	1.70	2.50	2.28
$LuL_3 \cdot 2H_2O$	1.91	2.20	3.66

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