Journal of Thermal Analysis, Vol. 28 (1983) 359–370

THE THERMAL CHARACTERISTICS OF POTASSIUM OXALATO LANTHANATES: $K_3LnOx_3 \cdot n\,H_2O$ (Ln = La – Tb) AND $K_8Ln_2Ox_7 \cdot 14\,H_2O$ (Ln = Tb – Yb, Y)

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(Received March 24, 1983; in revised form April 21, 1983)

The title compounds were studied by TG, DTA, DSC, IR and absorption spectroscopy. The complexes go through dehydration (70–200°C), an irreversible exothermic process (in air or N_2 atmosphere, 250–300 °C) and decomposition to a mixture of oxides and carbonates (385–700 °C). The exothermic process occurs without weight loss and corresponding heats of reaction fall in the range 0–26 kJ/mol. The absorption spectrum of the Nd complex in the range 5000–6000 Å was employed to monitor perturbations in the coordination sphere of Nd^{3+} arising from the exothermic process. Involvement of the Nd^{3+} cation is implied and the heats of reaction show a close relationship to the radii of Ln^{3+}. The interpretation of these data was made with the aid of valuable structural information obtained previously.

Recently we reported a successful synthesis of potassium oxalato lanthanates: $K_3LnOx_3 \cdot n\,H_2O$ ($n = 6$ for Ln = La, $n = 5.5$ for Ln = Ce, Pr and $n = 3$ for Ln = Nd, Sm, Eu, Gd, Tb) and $K_8Ln_2Ox_7 \cdot 14\,H_2O$ for Ln = Tb, Dy, Ho, Er, Yb and Y [1]. In that report the detailed crystal structures of the complexes of Nd, Gd, Er and Y were described and cell parameters of the complexes of Sm, Eu, Tb, Dy, Yb were given. The complexes of Nd, Sm, Eu, Gd are triclinic with space group $P\bar{1}$ and those of Dy, Ho, Er, Yb and Y are monoclinic with space group $C2/c$. Tb forms a monoclinic complex $K_8Tb_2Ox_7 \cdot 14\,H_2O$ and a triclinic complex $K_3TbOx_3 \cdot 3\,H_2O$. The complexes of La, Ce and Pr have a different crystal structure which is currently being investigated.

In a further investigation of these complexes we have studied their thermal characteristics by differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermogravimetry (TG) and absorption spectroscopy. The complexes show dehydration and finally decomposition into a mixture of oxides and carbonates in the range 50–200 and 385–700° (Ce complex decomposes at 320–330°) respectively. In addition to these endothermic stages an irreversible exothermic peak is observed without weight loss at 250–300° and DSC studies indicate energies involved to be in the range 0–26 kJ/mol of Ln^{3+}.

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Having obtained valuable information from the structural investigations it has been possible to put forward reasonable proposals to explain the dehydration behavior of these complexes, which is found to be very different from that of lanthanide oxalates; and to explain the origin of the irreversible exothermic peak which is apparently peculiar to these complexes. We have also discounted an oxalate decomposition mechanism in which an initial separation into individual oxalate components is postulated for lanthanide and actinide oxalato complexes [2].

Experimental

All the compounds were prepared according to the procedure described previously [1]. The DTA and DSC data were obtained using a Dupont 900 instrument while the TG data were obtained using a Dupont 960 thermal balance. Unless otherwise stated the DTA, DSC and TG data were obtained in a nitrogen atmosphere. For TG the samples were packed in a platinum boat; the crystals weighing 13–20 mg were not crushed and the heating rate was 20 degree/min. Temperatures were measured with a chromel-alumel thermocouple placed near the platinum boat and were corrected for nonlinearity with the tables supplied by Dupont Co. In the case of DTA and DSC the thermal couples were placed under a raised platform carrying the reference alumina. Except in the case of the exothermic peak at about 250–300° the temperatures quoted are derived from the TG curves. In DTA and DSC studies the heating rate was maintained at 10 degree/minute and the sample holders were identical aluminium crucibles for measurements in the range 0–500°. For DSC investigations a standard sample of metallic tin supplied by Dupont Co. was used for calibrations and a fusion enthalpy of 59.892 mJ/mg was assumed. Sample weights were 15–18 mg for $Ln =$ La – Pr and 20–32 mg for $Ln =$ Nd – Yb, Y. The gaseous flow rates were 0.34 liters per minute.

The absorption spectra were obtained using a Cary 14 spectrophotometer and samples were thoroughly mulled in Nujol. IR spectra were obtained from a Perkin–Elmer 621 spectrophotometer with samples also mulled in Nujol.

Results and discussion

The onset dehydration and decomposition temperatures along with the corresponding weight losses expressed as:

$$\frac{\text{Weight loss in dehydration (or decomposition)}}{\text{Total weight lost in the range 24–900 °C}} \times 100$$

are given in Table 1. The heats of reaction are also given in Table 1. Sample DTA and TG curves are given in Figs 1 and 2.

Crystal structure investigations revealed that one water molecule is coordinated to the Ln^{3+} cation in complexes of Nd and Gd [1] and the other two water molecules

Table 1 Thermoanalytical data in N_2 atmosphere and Ln^{3+} radii

Complex and mol · wt.	Dehydration			Decomposition			ΔH,	Radius
	Temp., °C	Wt. loss, %[+] Obs.	Calc.	Temp., °C	Wt. loss, %[+] Obs.	Calc.	− kJ/mol	of Ln^{3+} (Cn 8)Å
$K_3LaOx_3 \cdot 6\,H_2O$ (628.21)	60–70	41.0	41.9	410–400	59.0	58.1	26	1.18
$K_3CeOx_3 \cdot 5.5\,H_2O$ (620.42)	55–60	40.8	39.8	320, 390*	59.2	58.9	22	1.14
$K_3PrOx_3 \cdot 5.5\,H_2O$ (616.85)	50–60	38.9	39.8	400–410	61.0	60.2	22	1.14
$K_3NdOx_3 \cdot 3\,H_2O$ (579.54)	100–110	27.4	26.5	390–400	72.6	73.5	13	1.12
$K_2SmOx_3 \cdot 3\,H_2O$ (585.7)	120–130	26.4	26.5	390–400	73.6	73.5	10	1.09
$K_3EuOx_3 \cdot 3\,H_2O$ (587.3)	110–120	27.2	26.5	390–400	72.8	73.5	7	1.07
$K_3GdOx_3 \cdot 3\,H_2O$ (592.05)	110–120	26.7	26.5	400–410	73.3	73.5	13	1.06
$K_3TbOx_3 \cdot 3\,H_2O$ (594.2)	100	27.8	26.5	390	72.2	73.5	0.80	1.04
$K_8Tb_2Ox_7 \cdot 14\,H_2O$ (1498.6)	60–70	41.8	43.4	400–410	58.2	56.6	0	1.04
$K_8Dy_2Ox_7 \cdot 14\,H_2O$ (1507.8)	60–70	41.8	43.4	400–410	58.2	56.6	10	1.02
$K_8Ho_2Ox_7 \cdot 14\,H_2O$ (1510.8)	50–60	43.9	43.4	385–395	56.1	56.6	–	1.02
$K_8Er_3Ox_7 \cdot 14\,H_2O$ (1515.32)	50–60	41.7	43.4	385–395	58.3	56.6	17	1.00
$K_8Yb_2Ox_7 \cdot 14\,H_2O$ (1526.8)	50–60	41.1	43.4	400–410	59.5	56.6	18	0.98
$K_8Y_2Ox_7 \cdot 14\,H_2O$ (1360.6)	50–60	41.5	43.4	400–410	58.5	56.6	10	1.02
$Cs_3PrOx_3 \cdot 7.6\,H_2O$ (940.4)	60–70	48.5	48.6	380–385	51.5	51.4	33.5	1.14
$Cs_3ErOx_3 \cdot 5\,H_2O$ (920.0)	45.50	38.5	37.5	400–410	61.5	62.5	0	1.00

* 320° (TG); 390° (DTA-cell construction enables N_2 atmosphere to be thoroughly maintained) product taken as CeO_2 rather than Ce_2O_3.

[+] *Assuming the reactions:*

$$2K_3LnOx_3 \cdot nH_2O \xrightarrow{-2nH_2O} 2K_3LnOx_3 \rightarrow 3K_2CO_3 + Ln_3O_3 + 6CO + 3CO_2$$

and

$$K_8Ln_2Ox_7 \cdot 14H_2O \xrightarrow{-14H_2O} K_8Ln_2Ox_7 \rightarrow 4K_2CO_3 + Ln_2O_3 + 7CO + 3CO_2$$

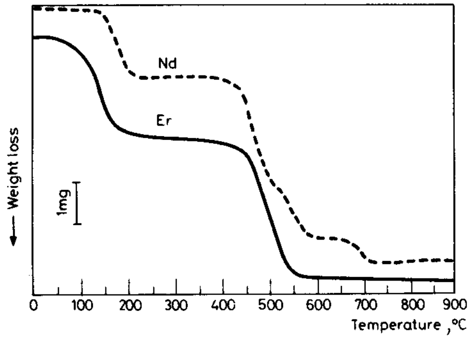


Fig. 1 TG curves for $K_3NdOx_3 \cdot 3\,H_2O$ (Nd) (16 mg) and $K_8Er_2Ox_7 \cdot 14\,H_2O$ (Er) (14 mg) obtained in N_2 atmosphere

coordinate to the K^+ ion. It is therefore expected that one water molecule would be lost at a relatively higher temperature that the other two because of obvious differences in their bonding energies to Ln^{3+} and K^+ respectively. The two stage water loss is endeed observed in $K_3GdOx_3 \cdot 3\,H_2O$ but no distinct separation of the dehydration stages is indicated on TG curves even at a heating rate of 10 degree/minute. Furthermore the oxalate ligands coordinate through the oxygens forming the longest C—O bonds (i.e. the more negatively charged oxygens). One expects these pronounced cation-anion interactions to weaken the $Ln^{3+}{-}OH_2$ (cation-dipole) interaction and therefore lead to an early loss of the coordinated water molecule. For instance the last two water molecules in $Gd_2Ox_3 \cdot 10\,H_2O$ are lost beginning at about 200° in contrast to total dehydration at nearly the same temperature in $K_3GdOx_3 \cdot 3\,H_2O$. The La, Ce and Pr compounds appear to have additional zeolitic water which is lost at low temperature (50°) but no structural information is yet available to confirm this.

The monoclinic complexes of Tb, Dy, Ho, Er, Yb and Y lose all their water in a single stage and dehydration is complete upon heating to 200° (complete dehydration indicated by absence of water absorption peaks in the IR spectrum). This behavior contrasts sharply with that of $Ln_2Ox_3 \cdot n\,H_2O$ in which the last water molecules are lost at much higher temperatures (300—340°). This was expected from our structure determination in which no water molecules were found coordinated to the Ln^{3+} (Ln = Tb, Dy, Ho, Er, Yb and Y) cations [1]; the dodecahedral geometry around these Ln^{3+} cations was found to be made up of oxalate oxygens only.

Decomposition of the oxalate ligand starts at about 385° with an endotherm in both N_2 and air atmospheres (except for the Ce complex, which decomposes at 260° in air). The endotherm is accompanied by weight loss. In N_2 atmosphere this rather weak endotherm is followed by a large endotherm (which is split into two peaks for

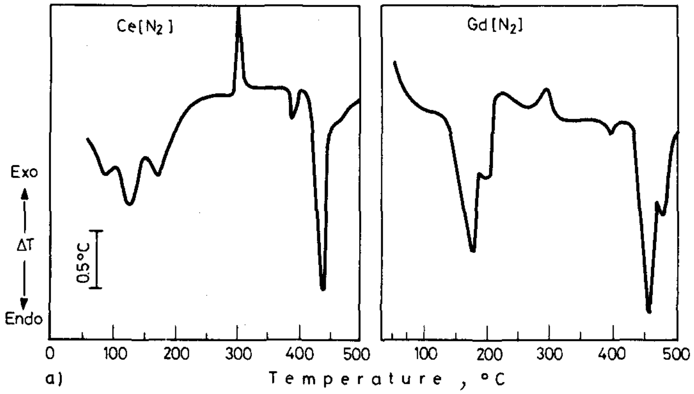
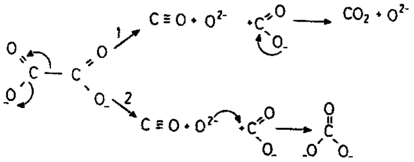


Fig. 2 (a) DTA curves for $K_3CeOx_3 \cdot 5.5\ H_2O$ (Ce) (14 mg) and $K_3GdOx_3 \cdot 3\ H_2O$ (Gd) (19 mg) obtained in N_2 atmosphere

the monoclinic complexes), while in air the endotherm is followed by a large exotherm (which is also split in the case of monoclinic complexes). The complexes of La, Pr, Nd, Sm and Eu form stable oxycarbonates $Ln_2O_2CO_3$ indicated by a clear horizontal plateau on the TG curves. The decomposition of these intermediate oxycarbonates results in a product with a stoichiometry approximating to a 3 K_2CO_3/Ln_2O_3 mixture. Oxycarbonate intermediates are well known in the thermal decomposition of lanthanide oxalates [3–5]. The Ce complex decomposes to a more complex mixture of oxides and carbonates without going through any intermediate, in both air and N_2 atmospheres. The monoclinic complexes of Tb, Dy, Ho, Er, Yb and Y decompose to a mixture of carbonates and oxides approximating to 4 K_2CO_3/Ln_2O_3. The presence of carbonate was confirmed by IR and its characteristic reaction with acids. Further weight losses were observed at 950° but no investigations were carried beyond this temperature.

In another experiment, the Ce complex was heated in the DTA cell under N_2 atmosphere until the exothermic process at 250–300° was complete. The heated sample was then transferred to the TG system which was then flushed with air for 10 minutes before heating was started. Weight loss was noticeable at about 260° and was complete at about 480° without any indication of intermediate products. It was therefore established that the exothermic process can not be associated with a split of the complex into component oxalates (K_2Ox and Ce_2Ox_3) since the decomposition temperatures of the two oxalates in air, 260 and 475° respectively, are very different; i.e. the separate decomposition of Ce_2Ox_3 and K_2Ox should be readily detected on the DTA and TG curves. It is difficult to be certain that a split of the complexes into individual component oxalates does not occur before oxalate decomposition by looking at the TG and DTA curves of the other materials studied here since oxalate decomposition takes off slowly and overlaps cannot be discounted. But the TG curves of the complexes K_4UOx_4 and Cs_4UOx_4 reported by Awasth et al. [2(a)] indicate complete oxalate decomposition into a mixture of oxides and carbonates in the neighborhood of 300°. Similar studies by the same investigators were done on the complexes Ba_2UOx_4 and Sr_2UOx_4 [2(b)] and oxalate decomposition was found to be complete at about 210 and 380°, respectively. It appears to us that both our work and that of Awasth et al. indicate that the thermal stability of the oxalate anions is critically influenced by the cations Ln^{3+} (in the lanthanide complexes) and U^{4+} (in the uranium complexes). The charge balancing cations K^+ or Cs^+ and Ba^{2+} or Sr^{2+} appear to be playing a much less significant role in determining the fate of the oxalate anion. X-ray structure determination revealed that the coordination sphere of the Ln^{3+} cation is made up of bridging and non-bridging oxalates which means the oxalate groups are experiencing different levels of interaction with the Ln^{3+} cations [1]. This may explain why the exothermic or endothermic oxalate decomposition peaks are split (Fig. 1). The exothermic peak observed by Awasth et al. at 440° in the decomposition of M_2UOx_4 (M = Ba, Sr) complexes in air could be associated with the combustion of carbon deposited by the disproportionation of carbon monoxide (a typical feature of oxalate decomposition [3, 6]), rather than the decomposition of strontium or barium oxalate as suggested by Awasth et al. [2]. As stated earlier the oxalate ligands bond to Ln^{3+} via the oxygens carrying the largest negative charge, one therefore expects the electrostatic interaction between the oxalate anions and K^+ to be weak thereby making a split of the complexes into component oxalate salts thermodynamically unfavourable because strong Ln^{3+}-oxalate bonds have to be broken to form weak K^+-oxalate bonds.

Employing the oxalate decomposition mechanism which we reported earlier [7]:



the rupture of the C—O bond may be thought of as being decisively determined by the interaction between Ln^{3+} and the oxalate oxygens. The formation of potassium carbonate may therefore result from secondary electronic reorganization processes occurring after the rupture of the C—O bond and does not necessarily indicate the prior formation of potassium oxalate.

The exothermic peak at 250—300° (340—390° for Cs_3PrOx_3) is not found in dioxalato lanthanates $KLnOx_2$ or lanthanide oxalates Ln_2Ox_3. To probe the possibility for the involvement of the Ln^{3+} coordination sphere, we have investigated the influence of this process on the nature of the hypersensitive peak in the absorption spectrum of the neodymium complex. This peak corresponds to the transition $^4I_{9/2} \rightarrow {}^4G_{5/2}$ and occurs at about 5780 Å; another peak known to be hypersensitive corresponds to the transition $^4I_{9/2} \rightarrow {}^4G_{7/2}$ and occurs at about 5200 Å. While ignoring the unresolved controversy regarding the origin of hypersensitivity in the absorption and emission spectra of lanthanide compounds [8–11], researchers have utilized the phenomena to obtain detailed information regarding the coordination sphere of Ln^{3+} in solutions of their complexes [12–14]. Extensive thermodynamic data and stability constants [14] and in some cases the Ln^{3+} site symmetry [12, 13] have been obtained from the oscillator strength, shifts and shape of the hypersensitive peaks. In Fig. 3–5 we present parts of the absorption spectra of $K_3NdOx_3 \cdot 3\,H_2O$ (Fig. 3), K_3NdOx_3 (preheated to 225°) (Fig. 4) and K_3NdOx_3 (preheated to 325°) (Fig. 5), showing the two hypersensitive peaks. Clearly the exothermic process affects the coordination sphere of the Nd^{3+} cation since it effects modifications in the definition of the transitions associated with the hypersensitive absorption band.

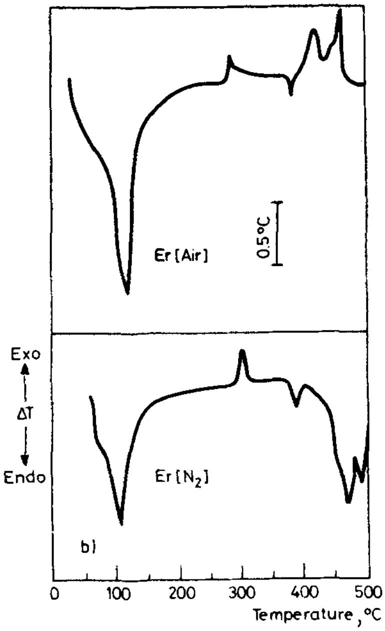


Fig. 2 (b) DTA curves for $K_8Er_2Ox_7 \cdot 16\,H_2O$ (15–16 mg) obtained in N_2 and air atmospheres

We have also investigated the trend followed by the magnitude of the enthalpy function, ΔH, when the radius of Ln^{3+} is varied. A plot of ΔH *vs.* r (the Ln^{3+} radius in octacoordination [15]) is shown in Fig. 6 where good correlation was obtained. The ΔH values are an average of two or three values differing by not more than 3 kJ/mol. The plot predicts positive ΔH values for most of the trioxalato complexes of heavy lanthanides and indeed no transition is observed for the compound Cs_3ErOx_3

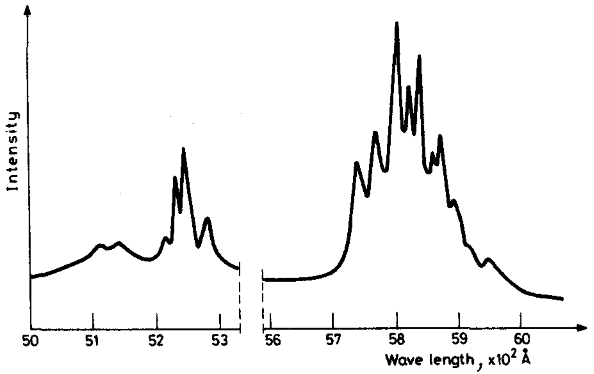


Fig. 3 Absorption spectrum of $K_3NdOx_3 \cdot 3\,H_2O$ in Nujol mull at room temperature

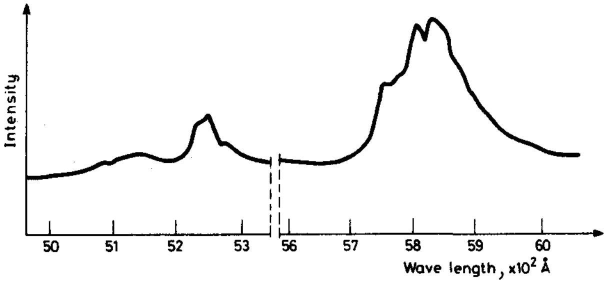


Fig. 4 Absorption spectrum of K_3NdOx_3 (preheated to 225°) in Nujol mull at room temperature

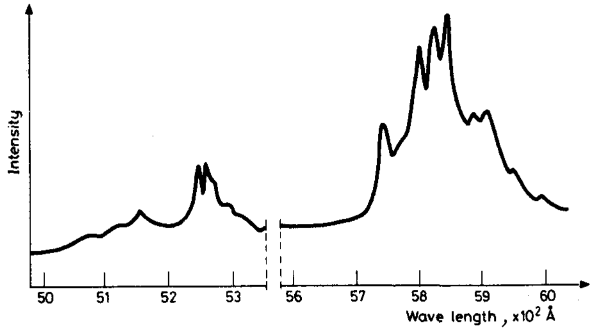


Fig. 5 Absorption spectrum of K_3NdOx_3 (preheated to 325°) in Nujol mull at room temperature

while a prominent peak is found on the DTA curve of Cs_3PrOx_3. The exothermic peak for 27.2 mg of $K_3TbOx_3 \cdot 3\,H_2O$ was small and no exotherm was observed in the region 200–380° with 34.8 mg of $K_8Tb_2Ox_7 \cdot 14\,H_2O$.

In view of the above facts the most satisfactory explanation of this exothermic process is the formation of more Ln–O bonds. This is possible through the utilization of suitably placed oxygen atoms of non-bridging oxalates in one chain (or dinuclear species for complexes of heavy lanthanides) by a Ln^{3+} cation in an adjacent chain (or dinuclear species). X-ray structure determination reveals that the complexes $K_3LnOx_3 \cdot 3\,H_2)$ have chains with $\ldots Ox(LnOx_2 \cdot OH_2)Ox \ldots$ units while the $K_8Ln_2Ox_7 \cdot 14\,H_2O$ have dinuclear species $Ox_3LnOxLnOx_3^{8-}$ [1]. The formation of these new bonds will lead to at least coordination number nine around the Ln^{3+} and there are no steric or radii ratio restrictions which make this possibility unlikely since nonacoordination is known in many room temperature structures of $Ln_3Ox_3 \cdot n\,H_2O$ and $LnOx_2^-$ systems [16–20]. The reason why Ln_2Ox_3 and $MLnOx_2$ (M = alkaline metal) would not form more bonds like the complexes studied here is that there are no unbridged oxalate ligands in those compounds to facilitate the process. All oxalate anions in those compounds are rigidly bonded in an extensive bridging structure [16–20]. Actually even in the compounds K_3LnOx_3 where each Ln^{3+} cation is bridged to two other Ln^{3+} cations via oxalate ligands, structural rigidity (more pronounced in complexes of smaller Ln^{3+}) appears to impose restrictions on the degree of new Ln–O contacts which is reflected in the diminishing magnitude of ΔH (Fig. 6). On the other hand the complexes $K_8Ln_2Ox_7$ with one bridge per Ln^{3+} cation should suffer less structural rigidity and accordingly the Ln–O bond strength (for new Ln–O contacts) increases with increasing ionic potential (q/r) (Fig. 6). It seems that the formation of new Ln–O contacts in $K_8Ln_2Ox_7$ complexes requires that q/r of Ln^{3+} be greater than q/r of Tb^{3+}. As noted earlier the prerequisite for

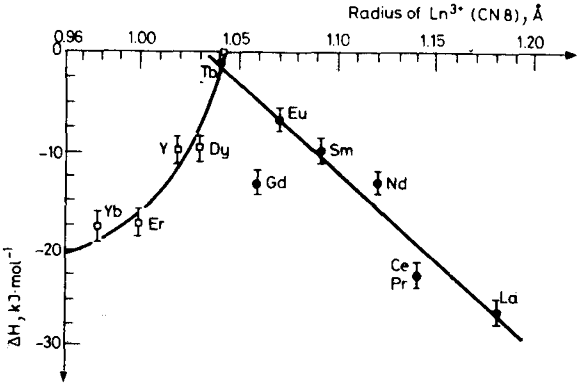


Fig. 6 *The heats of reaction (at 250–300°) vs. the ionic radius of Ln^{3+} in octacoordination* (see Table 1). Filled circles K_3LnOx_3; squares $K_8Ln_2Ox_7$

the stability of these dinuclear species is that $q/r \geqslant q/r$ for Tb^{3+} and Tb^{3+} keeps a low coordination number to ensure the largest possible q/r. Observation of the exotherm at a rather high temperature in Cs_3LnOx_3 is understood if we appreciate the fact that replacement of K^+ with Cs^+ increases the separation between the adjacent chains. If this is true the activation energy for the process should also increase and the manifestation of this increase could be a shift of the exotherm to a higher temperature.

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We thank the USA government for a Fulbright scholarship to I. A. Kahwa.

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Zusammenfassung — TG, DTA, DSC, IR und Absorptionsspektrometrie wurden zur Untersuchung der Titelverbindungen herangezogen. Die Komplexe werden dehydratisiert (70–200 °C), gehen dann eine irreversible exotherme Reaktion ein (in Luft oder Stickstoffatmosphäre, 250–300 °C) und werden schliesslich zu einer Mischung von Oxiden und Carbonaten zersetzt (385–700 °C). Der exotherme Prozess geht nicht mit einem Gewichtsverlust einher, und die entsprechenden Reaktionswärmen liegen im Bereich von 7–26 kJ/mol. Das Absorptionsspektrum des Nd-Komplexes im Bereich von 5000–6000 Å wurde zur Beobachtung von durch den exothermen Prozess verursachten Störungen in der Koordinationssphäre von Nd^{3+} herangezogen. Die Reaktionswärmen weisen einen engen Zusammenhang mit den Radien von Ln^{3+} auf. Zur Interpretation dieser Daten wurde auf früher erhaltene strukturelle Informationen zurückgegriffen.

Резюме — Методами ТГ, ДТА, ДСК, ИК- и электронной спектроскопией изучены лантаноксалатокалиевые комплексы. Разложение комплексов протекает через стадию дегидратации (70–200°С), необратимый эксотермический процесс (на воздухе и в атмосфере азота при 250-300° С) и конечный процесс с образованием смесей окислов и карбонатов (385–700° С). Эксотермический процесс не сопровождается потерей веса и соответствующие теплоты реакции находтся в области 7–26 кдж/моль. Электронный спектр поглощения неодимиевого комплекса в области 5000–6000 А был использован для определения возмущений в координационной сфере Nd^{3+}, возникающих вследствии экзотермического процесса. Принята во внимание сложность катиона Nd^{3+}. Теплоты реакции показывают тесную взаимосвязь с ионными радиусами лантанидов. Интерпретация полученных данных проведена на основе полученной ранее структурной информации.