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THERMAL ANALYSIS OF RARE EARTH COMPLEXES OF *N*-PHENYLBENZOHYDROXAMATE

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ABSTRACT

The thermal dissociation of rare earth complexes of *N*-phenylbenzohydroxamate were studied by differential thermal analysis (DTA) and thermogravimetric analysis (TGA). In general the DTA curves showed two exothermic peaks caused by melting and/or decomposition of the compounds.

INTRODUCTION

N-Phenylbenzohydroxamic acid (PBHA) is widely used as an analytical reagent for gravimetric determination and solvent extraction of various metal ions [1—6]. The preparation and properties of rare earth complexes of PBHA have been discussed earlier [7].

In the present investigation, the thermal analysis of La(III), Ce(III), Pr(III), Nd(III), and Sm(III) complexes of PBHA is discussed.

EXPERIMENTAL

The TG and DTA curves of the complexes were recorded on a Mettler thermal analyser maintaining the following instrumental factors in all the experiments: TG range 1 mg full scale sensitivity; DTA range 50 V, heating rate 5° min^{-1}; gas flow rate 100 ml min^{-1}; and mass of the sample 10 mg.

The samples were characterised by X-ray powder diffraction on a Philips instrument (PW 1050) using CuK radiation. The infrared spectra were recorded on a Perkin Elmer Model 221 Spectrophotometer using KBr discs. The solid complexes of La^{3+}, Ce^{3+}, Pr^{3+}, Nd^{3+}, and Sm^{3+}—PBHA were synthesised as described earlier [7].

RESULTS AND DISCUSSION

The DTA curves of rare earths complexes of PBHA are reproduced in Fig. 1. The DTA and TGA data are given in Table 1.

The thermograms for the complexes heated up to 1000°C in flowing air

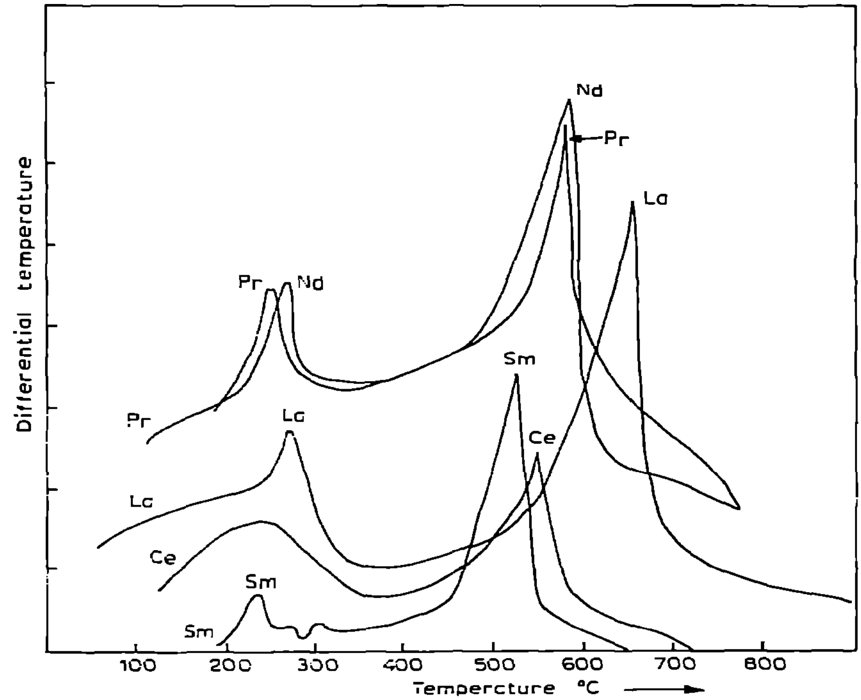

Fig. 1. DTA curves of the rare earth complexes of PBHA.

(Table 1) show no change in weight around 100—180°C indicating that the complexes contained no water of crystallisation. The complexes were stable up to 230°C. The curves have a number of similar factors. All of these

TABLE 1

DTA and TGA analysis of rare earth *N*-phenylbenzohydroxamates

Sample no.	Property	Rare earth				
		La	Ce	Pr	Nd	Sm
1	Colour of complexes	White	Reddish-yellow	White	White	White
2	M.p. (°C)	250	230	240	245	225
3	Analysis (%)					
	Calcd. N	5.42	5.41	5.40	5.34	5.39
	M	17.92	18.04	18.12	18.47	19.12
	Found N	5.62	5.39	5.46	5.39	5.29
	M	18.00	18.32	18.65	18.23	18.99
4	DTA					
	1st exotherm	265	246	250	255	235
	2nd exotherm	600	550	570	580	523
	Residue	La_2O_3	Ce_2O_3	Pr_2O_3	Nd_2O_3	Sm_2O_3
5	TGA					
	Weight loss (%)					
	Calcd.	82.16	81.96	81.98	81.53	80.90
	Found	83.00	80.53	82.00	81.88	80.00

chelates show an exothermic decomposition which appears as a peak in the DTA curves shown in Fig. 1 and as a weight loss in the TGA curves. The first sharp exotherm was around 235—250°C with a gradual weight loss and another broad one around 520—560°C with further weight loss (Table 1), presumably due to the burning of organic matter and oxidation of rare earths to their oxides.

The major products of the thermal decomposition of the complexes were analysed by IR, UV and X-ray analysis. The results indicate that the complexes were decomposed into rare earth benzoates, *N*-phenylbenzamide, and finally rare earth oxides and tar. At 235—250°C (first exotherm), the vaporisation of *N*-phenylbenzamide with gradual weight loss and around 520—560°C (second exotherm), the decomposition of rare earth benzoates and formation of oxides occurred.

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