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Note

THERMAL ANALYSIS OF *N-o*-CHLORO- AND *N-m*-CHLOROPHENYLHYDROXAMIC ACIDS

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The hydroxamic acids are versatile analytical reagents for the solvent extraction and gravimetric determination of several metal ions [1–5]. The *N-o*-chloro- and *N-m*-chlorophenylhydroxamic acids were synthesised [6] with a view to giving more selective and sensitive reagents for the determination of trace transition metals in the environment [7]. In the present paper, the thermal stability of these newly synthesised hydroxamic acids are described.

EXPERIMENTAL

The hydroxamic acids were synthesised as described elsewhere [6].

The TG and DTA curves 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 5–10 mg. Alumina was used as a reference material for the DTA.

The samples were characterised by X-ray powder diffraction on a Philips instrument (PW 1050) using CuK radiation.

RESULTS AND DISCUSSION

The DTA curves of the hydroxamic acids are reproduced in Fig. 1. The DTA and TGA data are given in Table 1.

The thermograms of the hydroxamic acids heated up to 1000°C in flowing air shows no change in weight around 100 ± 45°C, indicating that the hydroxamic acids contained no water of crystallization. The DTA curves show two exothermic peaks, one very sharp around 100 ± 45°C and another broad around 150 ± 50°C with weight loss.

The major products of these hydroxamic acids were analysed by UV, IR and X-ray analysis to be their fatty acids and carboxylic acid, anilides and finally tars.

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At about 120 ± 40°C the hydroxamic acids melted and decomposed (exotherm I) to the corresponding acids and anilides with continuous weight loss. Further at about 200 ± 50°C the organic matter decomposed to tars (exotherm II).

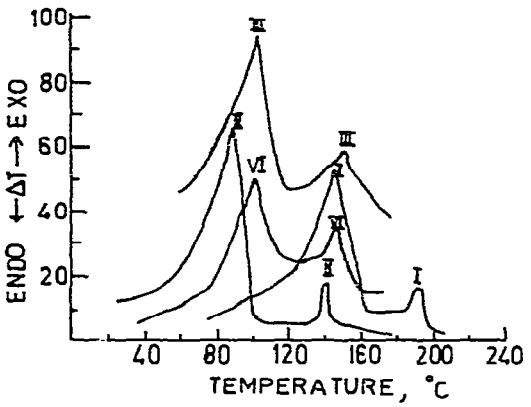

Fig. 1. DTA curves of the hydroxamic acids.

TABLE 1

Thermal analysis of *N*-arylhydroxamic acids

Compound No.	Hydroxamic acids	Mp (°C)	DTA (°C)		TG (wt. loss)	
			1st Isotherm	2nd Isotherm	°C [a]	°C [b]
I	*N*-*o*-Chlorophenyl-*o*-chlorobenzo-	144	145	190	190–210	210–255
II	*N*-*o*-Chlorophenyl-myristo-	86	88	135	136–180	180–240
III	*N*-*o*-Chlorophenyl-palmito	101	103	150	155–175	175–220
IV	*N*-*o*-Chlorophenyl-stearo-	96	95	140	141–170	170–215
V	*N*-*m*-Chlorophenyl-*o*-chlorobenzo-	136	140	175	176–200	200–250
VI	*N*-*m*-Chlorophenyl-myristo-	99	100	135	136–170	171–220
VII	*N*-*m*-Chlorophenyl-palmito-	100	100	150	151–175	175–215
VIII	*N*-*m*-Chlorophenyl-stearo-	95	95	135	136–175	175–210

[a] Decomposition.

[b] Complete decomposition.

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