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Note

INDENTIFICATION OF THE CHROMIUM SALT OF STEARIC ACID

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It has been assumed [1—5] that the reaction between sodium stearate and a chromium(III) salt leads to the formation of chromium(III)tristearate ($CrSt_3$). Our experience has shown that the product from this reaction invariably has a chromium content less than the theoretical value (e.g. experiment 5.2%; theory 5.76%) and published data confirm this [1,3]. Extraction of this reaction product with ethanol causes the Cr content to increase from 5.2%, through 5.76% to a value closer to that for mono-hydroxy substituted distearate ($CrSt_2OH$) and the extract is found to contain stearic acid (HSt). This suggests that the original reaction product is either

(i) impure $CrSt_3$ in which one of the stearate chains is weakly held and ethanol extraction replaces this chain by an —OH group after removing the impurity (HSt), or

(ii) a mixture of substituted stearates [$CrSt_2OH$ and possibly $CrSt(OH)_2$] and stearic acid, and extraction with ethanol simply removes the acid from admixture.

Thermogravimetric evidence supports the latter.

EXPERIMENTAL

Materials

The chromium salt was prepared by adding a 1.5% (w/w) aqueous sodium stearate solution and a 1% aqueous chromic potassium sulphate (BDH,AR) solution simultaneously to a third vessel containing water, with vigorous stirring of the mixture maintained at 45 ± 5° C. The precipitate was filtered, washed with water (8 dm^3) and dried in a vacuum desiccator over phosphorus pentoxide at 50° C. This will be referred to as the metathetic product.

The extraction of this product was performed at room temperature by irradiating a 1.5% (w/w) mixture of metathetic product and ethanol in an ultrasonic bath for 20 min, decanting the supernatant liquid and repeating once. After filtration the product was dried in a vacuum desiccator at 50° C. This will be referred to as the extracted product.

Chemical analysis

The chromium contents of these products were determined by ignition, in a platinum crucible, at 700° C to constant weight, followed by fusion with sodium carbonate, acidification with sulphuric acid, addition of excess ferrous ammonium sulphate and back titration with fresh potassium permanganate.

Apparatus

A Stanton-Redcroft TG 750 thermobalance and Pt crucibles were used. Two mg samples were subjected to a heating rate of 20°C min^{-1} under a dynamic atmosphere of dried, white spot N_2 at a flow rate of 25 cm^3 min^{-1}.

RESULTS

Chemical analysis

From Table 1 it would appear that the extracted product is predominantly $CrSt_2(OH)$ with some $CrSt(OH)_2$. The presence of stearic acid was disproved by the absence of a 1705 cm^{-1} peak in the IR spectrum.

Thermal analysis

The TG and DTG curves for the metathetic product are shown in Fig. 1(a). Figure 1(b) shows the results for a mixture of extracted product and stearic acid, prepared so as to have the same % chromium content as the metathetic product. Percent weight losses for these two samples over four temperature ranges are compared in Table 2.

TABLE 1

Analysis of products

Product	Chromium (%)
Metathetic	5.23
Extracted	8.61
$CrSt_3$ (theoretical)	5.76
$CrSt_2(OH)$ (theoretical)	8.18
$CrSt(OH)_2$ (theoretical)	14.08

TABLE 2

Weight losses

Temperature range (°C)	Weight loss (%)	
	Metathetic product	Extracted product
188—275	25.4	25.8
275—360	19.3	19.8
360—483	24.8	24.8
483—end	19.0	18.9

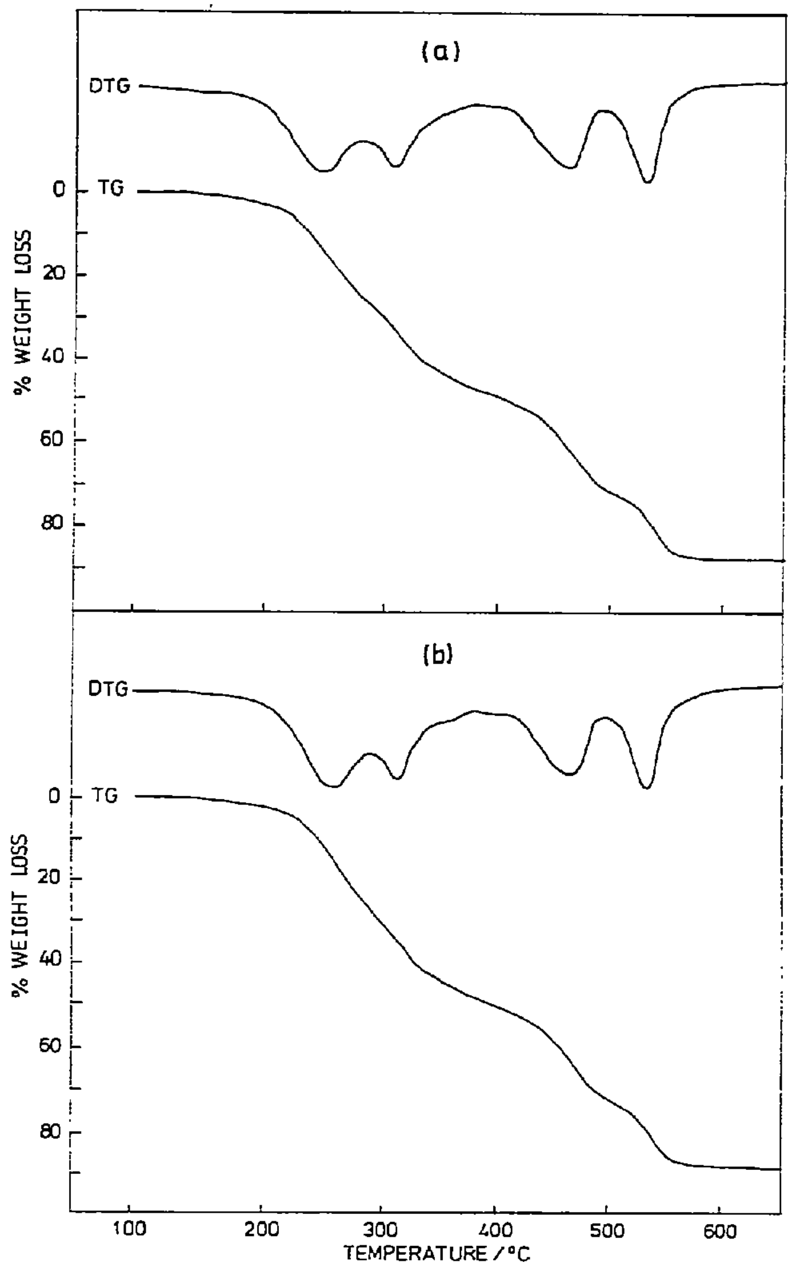

Fig. 1. TG and DTG curves. (a) Metathetic product; (b) mixture of extracted product and stearic acid.

CONCLUSION

The very close similarity of the TG and DTG curves, as demonstrated by Fig. 1 and Table 2, suggests that the product of the metathetic reaction between sodium stearate and a chromium salt is not chromium tristearate (contaminated with stearic acid) but a mixture of stearic acid and substituted chromium stearates, $CrSt_2(OH)$ and $CrSt(OH)_2$. Treatment of the metathetic product with ethanol removes the acid impurity.

It would thus appear that the tristearate of chromium like that of aluminium [6—8] requires special precautions to be taken during its preparation [9], and that the results quoted for previous studies [1—5] on chromium tristearate do not refer to $CrSt_3$ but to mixtures of stearic acid and hydroxy substituted stearates [$CrSt_2(OH)$ and $CrSt(OH)_2$].

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