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Note

STUDIES ON ARSENIC OXIDES: THERMAL ANALYSIS OF ARSENIC(III) OXIDE IN AIR, NITROGEN AND ARGON

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A survey of the current literature reveals that studies on the physico-chemical properties of the oxides of arsenic are scanty [1,2]. So far no attempt has been made of the thermogravimetric (TG) and differential thermal analysis (DTA) of As_2O_3 and related oxides of arsenic. In the present investigation the results, obtained from the TG and DTA of cubic As_2O_3 in air, nitrogen and argon, are discussed.

EXPERIMENTAL

Thermal analysis

The TG and DTA curves of As_2O_3, in different atmospheres, were recorded on a MOM derivatograph (Type 3427T) maintaining the following instrumental conditions in all the experiments: TG range, 5 mg full-scale sensitivity; DTA range, 50 μV; heating rate, 5°C min^{-1}; gas flow rate, 100 ml min^{-1}; mass of sample, 200 mg. The results are shown in Figs. 1—3.

X-Ray studies

The products, obtained after the thermal analyser runs as well as those obtained by the isothermal heating of As_2O_3 in air and nitrogen at various pre-determined temperatures (from TG curves), were characterised by X-ray powder diffraction method on a Philips instrument (PW 1104) using CuK_α radiation.

Preparation of the sample

Arsenic(III) oxide used in this study was 99.999% pure as supplied by Merck. The sample was a white crystalline solid and gave a sharp peak in X-ray diffraction patterns.

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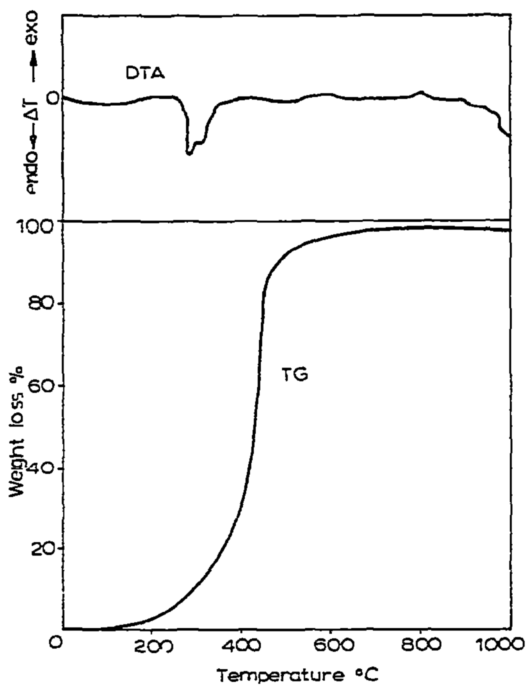

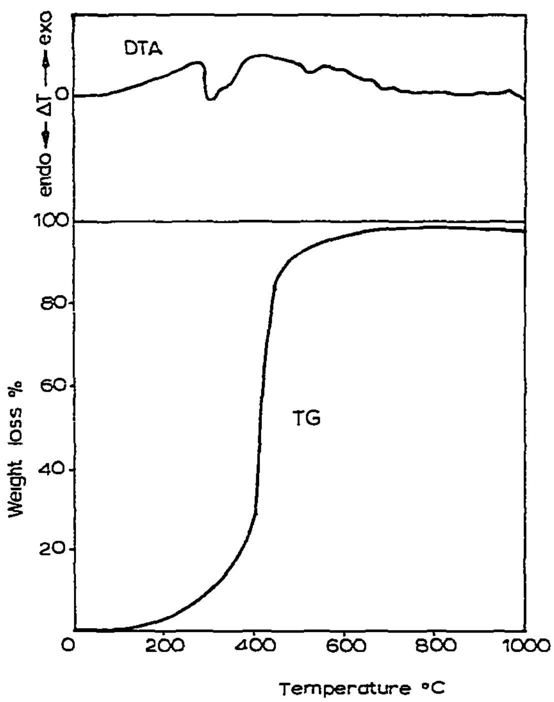

Fig. 1. TG and DTA curves for As_2O_3 in air.

Fig. 2 TG and DTA curves for As_2O_3 in nitrogen.

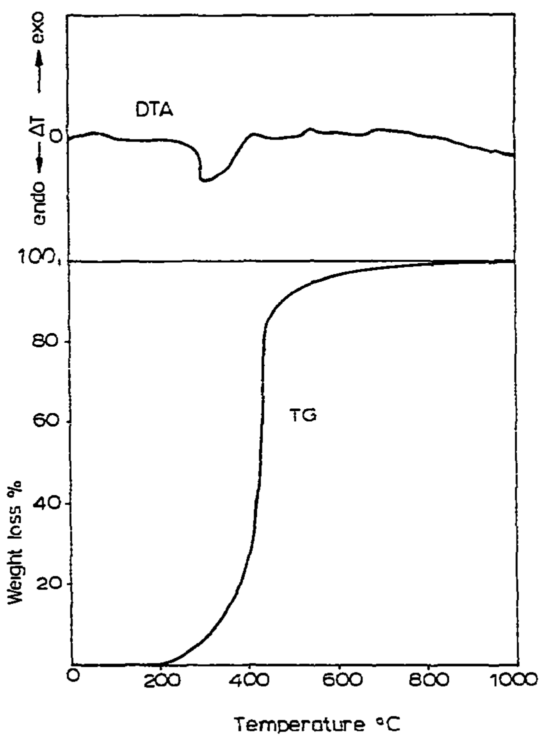

Fig. 3. TG and DTA curves for As_2O_3 in argon.

TABLE 1

The thermal characteristics (TG and DTA) of As_2O_3 in air, nitrogen and argon

Atmosphere	Temp (°C)	Time (h)	X-Ray analysis
Air	200	6	As_2O_3 Cubic
Air	280	6	As_2O_3 Monoclinic
Air	600	6	As_2O_3 Cubic sublimate, Monoclinic residue
Nitrogen	200	6	As_2O_3 Cubic
Nitrogen	280	6	As_2O_3 Cubic sublimate, Monoclinic residue
Argon	280	6	As_2O_3 Cubic sublimate, Monoclinic residue

For isothermal studies a known mass of the sample As_2O_3 was heated in a tubular furnace at the desired temperature for about 12 h and thereafter cooled in the furnace. The products were later characterised by X-ray analysis.

RESULTS AND DISCUSSION

Thermal analysis in air

The TG and DTA curves of As_2O_3 in air are shown in Fig. 1. Arsenic(III) oxide is stable in air up to 200°C, but starts to sublimate between 200 and 300°C. No weight gain is observed, instead there is a continuous weight loss. The corresponding DTA curve shows an endothermic peak. The final product (residue) was claudelite, as revealed by X-ray pattern.

Thermal analysis in nitrogen and argon

The thermal characteristics (TG and DTA) of As_2O_3 in nitrogen and argon are more or less the same as observed in air (see Figs. 2 and 3). The results shown in Table 1 indicate that As_2O_3 is not oxidised to As_2O_4 and As_2O_5 in air even when heated to 1000°C. At 280°C the cubic (arsenolite) form of As_2O_3 transforms to the stable monoclinic form (claudelite). The monoclinic form appears to be the more stable phase among the two modifications in nitrogen and argon. This conclusion comes from the fact that on heating in nitrogen or argon, the cubic form volatilizes at 280°C while the monoclinic form remains stable. Here also the partial volatilization of As_2O_3 occurs due to the simultaneous transformation to the monoclinic form above 285°C.

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