Thermochimica Acta, 207 (1992) 337–339
Elsevier Science Publishers B.V., Amsterdam

Note

The decomposition of copper oxalate to metallic copper is well suited for checking the inert working conditions of thermal analysis equipment

J. Mullens, A. Vos, R. Carleer, J. Yperman and L.C. Van Poucke

Limburg University Centre, Laboratory of Inorganic and Physical Chemistry, B-3590 Diepenbeek (Belgium)

(Received 11 December 1991)

Thermal analysis (TA) techniques require careful control of some experimental parameters. One of these parameters is the gaseous atmosphere in which the experiment is carried out. Particularly at high temperatures, even very small amounts of oxygen can drastically influence the experiment so that totally different results are obtained compared with those in a completely inert atmosphere.

The conduction of inert gases such as argon or (below 800°C) nitrogen over an oxygen absorbent before entering the TA equipment is a necessary but not sufficient condition for carrying out the experiment in a totally oxygen-free atmosphere. One has to take care that the TA equipment is completely free of the oxygen that is inevitably inside the equipment after loading. Even flushing with the inert gas for 30 min at a flow of 50 ml min^{-1} before starting the experiment might be insufficient to remove all the residual air if the gas inlet and gas outlet are not placed in the right positions. This is shown in the following experiment.

Figure 1 shows the decomposition of 30.543 mg copper oxalate; the equipment was flushed with argon for 30 min at a rate of 50 ml min^{-1} before starting the experiment and the flushing was continued during the experiment at the same flow-rate. In an inert atmosphere the decomposition goes to metallic copper (theoretical remaining weight, 41.9%). As shown by the weight increase between 300 and 600°C, there is an oxidation of copper (theoretical remaining weight is 52.5% if 100% CuO)! The

Correspondence to: J. Mullens, Limburg University Centre, Laboratory of Inorganic and Physical Chemistry, B-3590 Diepenbeek, Belgium.

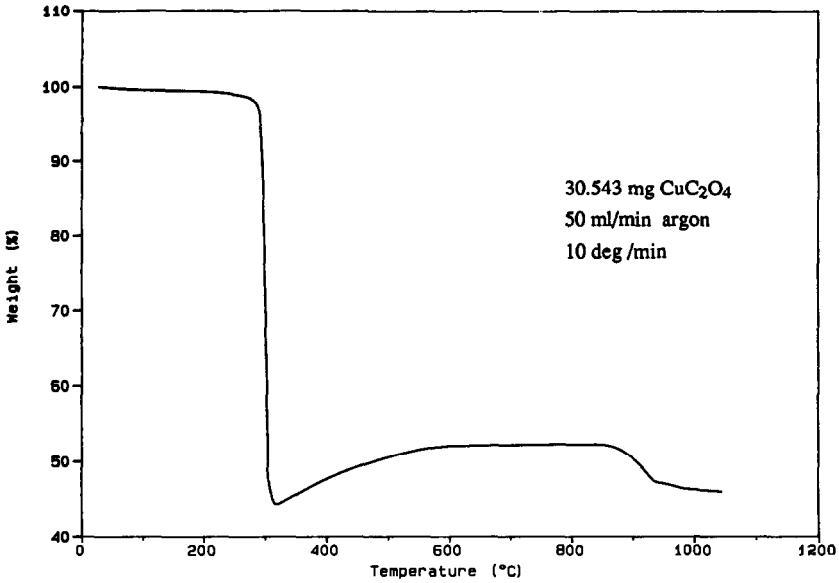

Fig. 1. The decomposition of copper oxalate following flushing with argon for 30 min.

explanation of this behaviour is that the dead volume at the left side of the balance was not oxygen free in spite of a flushing time of about 1 h (30 min flushing before starting and 30 min during the experiment at a heating rate of 10°C min^{-1}). The cause of all the trouble is the unsuitable place that the manufacturer has provided for the gas inlet (Fig. 2). To overcome this problem, we closed the original inlet and replaced it with a new one (a small job for a glassblower); in addition, a cap was welded on the inside to obtain a better spread of the gas and to prevent the swing of the tare weights. As shown in Fig. 3, the results of this small surgery were spectacular. Even after a flushing time of only 10 min before starting the experi-

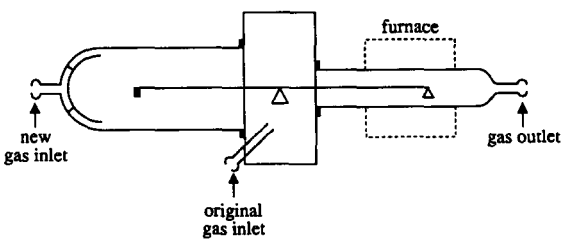

Fig. 2. Diagram of the TA equipment showing the positions of the original and new gas inlets.

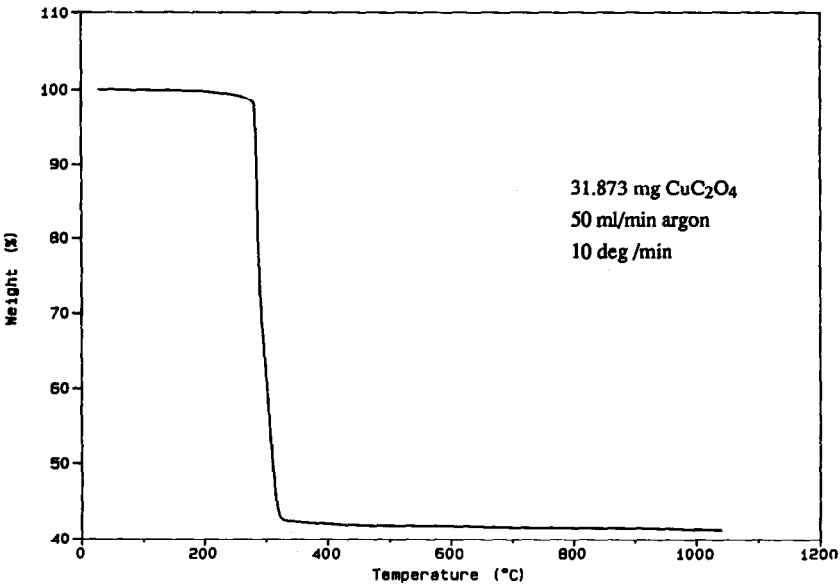

Fig. 3. The decomposition of copper oxalate after modification of the TA equipment and following flushing with argon for 10 min.

ment, the equipment was completely free of oxygen. In addition, one has of course always to take care that all the removable parts fit correctly in order to prevent sucking in of air.

The decomposition of copper oxalate is a well-suited very sensitive reaction for checking all kinds of TA equipment.