Short Communications

ON THE PREPARATION OF COPPER SULPHATE TRIHYDRATE BY A DERIVATOGRAPH*

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(Received November 11, 1975)

Copper sulphate trihydrate was prepared by dehydration from the pentahydrate sulphate by quenching the latter compound from the Derivatograph from 90 °C to room temperature.

Copper sulphate trihydrate is known as a decomposition product of the pentahydrate sulphate by heating it to between 85°–100 °C[1]. The synthetic compound can also be prepared by adding 55 ml of concentrated sulphuric acid to 45 ml of a solution saturated in copper sulphate at room temperature. The trihydrate is precipitated by heating on a hot water bath at 60°–70° for about 48 hours [2]. It has also been found as the mineral bonattite by Jambor [2] and Garavelli [3]. The determination of the crystal structure was performed by Zahrobsky and Baur [4].

We prepared this compound by simultaneous thermogravimetric analysis and differential thermal analysis. The equipment used was a Paulik–Paulik Erdey-type Derivatograph model 1969 manufactured by MOM, Budapest.

The preparation of the trihydrate sulphate required several trial- and- error experiments where the heating rate and grain size were varied. The best resolution was obtained with a heating rate of 0.5°/min and grain size having diameter between 0.10 mm and 0.20 mm. The results are shown in Fig. 1 and in this case the trihydrate is stable in a temperature range of 5°.

Using the same heating rate and different grain sizes the range of stability of this compound becomes smaller. For the same grain size and for a heating rate of 1°/min the horizontal part of the TG curve disappears completely.

The monohydrate and anhydrous sulphate are stable over a large range of temperature and are not too sensitive to either grain size of heating rate.

In the use of the Derivatograph it is possible to follow the DTA curve by visual inspection. In this way the heating process can be stopped at any temperature of interest The trihydrate sulphate was prepared by quenching cooling of the Derivatograph at 90° to room temperature. This quench temperature was chosen because this point is the minimum of the DTA curve, which means that the

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* Work supported in part by Conselho Nacional de Pesquisas and the Banco Nacional de Desenvolvimento Econômico, Brasil.

Table 1

$CuSO_4 \cdot 3H_2O$, X-ray Powder Data

Synthetic material				Dehydration of $CuSO_4 \cdot 5H_2O$			
d, Å	I	d, Å	I	d, Å	I	d, Å	I
5.10	68	2.972	22	5.15	65	2.98	25
4.85	30	2.819	42	4.87	30	2.82	40
4.42	100	2.747	10	4.40	100	2.74	10
3.96	44	2.660	6	3.98	40	2.66	6
3.68	16	2.557	8	3.69	18	2.55	10
3.65	52	2.498	40	3.66	50	2.49	40
3.29	10	2.420	12	3.29	10	2.45	15
3.25	64	2.353	16	3.25	60	2.35	17
3.18	40	2.277	36	3.18	40	2.27	40
3.01	40			3.02	40		

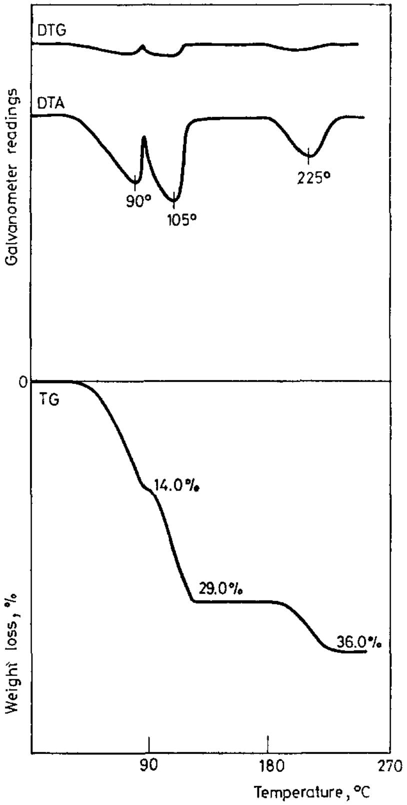

Fig. 1. TG, DTG and DTA curves with 5°min^{-1} heating rate for $CuSO_4 \cdot 5H_2O$. $CuSO_4 \cdot 3HO_2$ is formed at 90°. $CuSO_4 \cdot H_2O$ at 105° and $CuSO_4$ at 225°

decomposition of $CuSO_4 \cdot 5H_2O$ to $CuSO_4 \cdot 3H_2O$ is complete. A room temperature X-ray diffraction analysis was performed and the results are shown in Table 1 which also shows the X-ray data for the synthetic material [2]. The agreement between the two sets of data is excellent.

It is important to mention that heating rate and grain size depend on the brand of equipment used but we think that the technique employed in the preparation of this compound is of general application. Any other compound that is a product of a decomposition reaction can be prepared by the same technique, provided that horizontal line on the TG curve occurs, i.e., a stable compound is formed over a certain range of temperature.

On the other hand, for those decomposition reactions where overlap occurs, the process of preparation of compounds becomes more complicated. In this case the minimum of the DTG curve can be projected onto the TG curve [5] for the separation of the two compounds, although only approximately because of the overlapping process.

References

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