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Note

Sealed tube differential thermal analysis studies on the $CuSO_4 \cdot 3H_2O \rightarrow CuSO_4 \cdot H_2O$ transition

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Wendlandt[1] has previously summarized the detection of $CuSO_4 \cdot 3H_2O$ in the TG and DTA studies of $CuSO_4 \cdot 5H_2O$. Using the technique of sealed tube DTA, he observed two endothermic peaks for the dehydration of $CuSO_4 \cdot 5H_2O$—one peak was due to the $CuSO_4 \cdot 5H_2O \rightarrow CuSO_4 \cdot 3H_2O$ transition plus liquid water; the other was to originate from the $CuSO_4 \cdot 3H_2O \rightarrow CuSO_4 \cdot H_2O$ transition. We wish to report here additional information concerning the above latter transition.

EXPERIMENTAL

The sealed tube DTA apparatus has been previously described[2]. Experimental conditions employed were identical to those described earlier.

The $CuSO_4 \cdot 5H_2O$ employed was obtained from a commercially available source (Fisher Scientific Co., Pittsburgh, Pa.); a sample particle size corresponding to about 100 mesh was used. The $CuSO_4 \cdot 3H_2O$ sample was prepared from the 5-hydrate by use of a thermobalance. At the appropriate temperature and mass-loss, the TG run was terminated and the sample of the 3-hydrate removed and stored in a sealed glass capillary tube.

RESULTS AND DISCUSSION

The open and sealed tube DTA curves are given in Fig. 1.

In the sealed tube DTA curves, the curve peaks are due to water–copper(II) ion bond breaking and/or bond making reactions. Large thermal processes, such as those due to the vaporizations of water are not observed, hence, sealed tubes are very useful for dehydration (deaquation) studies of metal salt hydrates. Also, the experimental technique is much simpler than that required for high pressure DTA studies[3].

Curves (A) and (B) are those for the sealed and open tube studies of $CuSO_4 \cdot 5H_2O$, respectively, and are the same as those previously described[1]. A rerun of the sample from curve (A), after cooling for 2 h, is given in curve (C); after standing an additional 24 h, curve (D) was obtained. As can be seen, curve (D) is similar to

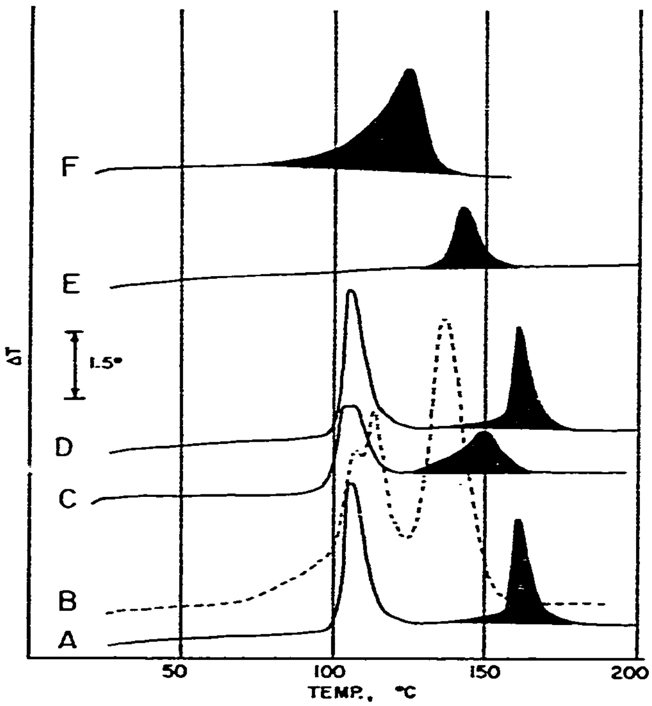

Fig. 1. Open and sealed tube DTA curves of $CuSO_4 \cdot 5H_2O$ and $CuSO_4 \cdot 3H_2O$ (upward direction of peaks indicate endothermic reactions). The shaded peak is for the $CuSO_4 \cdot 3H_2O \rightarrow CuSO_4 \cdot H_2O$ transition. (A) $CuSO_4 \cdot 5H_2O$, sealed tube; (B) $CuSO_4 \cdot 5H_2O$, open tube; (C) Sample (A), 2 h later; (D) Sample (A), 24 h later; (E) $CuSO_4 \cdot 3H_2O$, sealed tube; (F) $CuSO_4 \cdot 3H_2O$, open tube.

curve (A) indicating that rehydration of the sample occurred in the sealed tube on standing. Apparently, standing for 2 h is not sufficient for complete rehydration to take place.

In the case of $CuSO_4 \cdot 3H_2O$, the sealed and open tube results are shown in curves (E) and (F), respectively. The peak maximum occurs at a lower temperature (142°C) for the 3-hydrate than that for the 5-hydrate (161°C), which is probably due to the absence of an excess of water vapor in the former. Because of the free diffusion of the water vapor from the sample area, the peak maximum is shifted to even lower temperatures for the open tube curve (curve F).

The previous interpretation[1] that the second curve peak in the sealed tube DTA studies is due to the dehydration of $CuSO_4 \cdot 3H_2O$ is apparently correct. If the 3-hydrate is initially employed, the peak is shifted to lower temperatures due to the absence of excess water vapor in the sealed tube. If the initial compound is the 5-hydrate, the second peak is shifted to higher temperatures, due to the effect of water vapor from the previous dehydration reaction.

ACKNOWLEDGMENT

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