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Note

A simple method for recording sample temperature in thermogravimetry

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In thermogravimetry (TG), many investigators prefer that the change of sample mass be recorded as a function of sample temperature using an X–Y function recording system[1]. With many recording microbalances, this goal is compromised in that the furnace temperature, T_f, which is a close approximation of the actual sample temperature, T_s, is the recorded temperature function. This is necessary, of course, due to the difficulties in attaching external electrical leads to a thermocouple in intimate contact with the sample.

We have found a simple solution to this problem which eliminates the attachment of connecting leads to a sensitive balance weighing mechanism. This arrangement, which is shown in Fig. 1, makes use of a second sample container (D) identical to the one suspended from the balance (C). This container, which has an attached thermocouple, is in a fixed position located as close as possible to the one attached to the

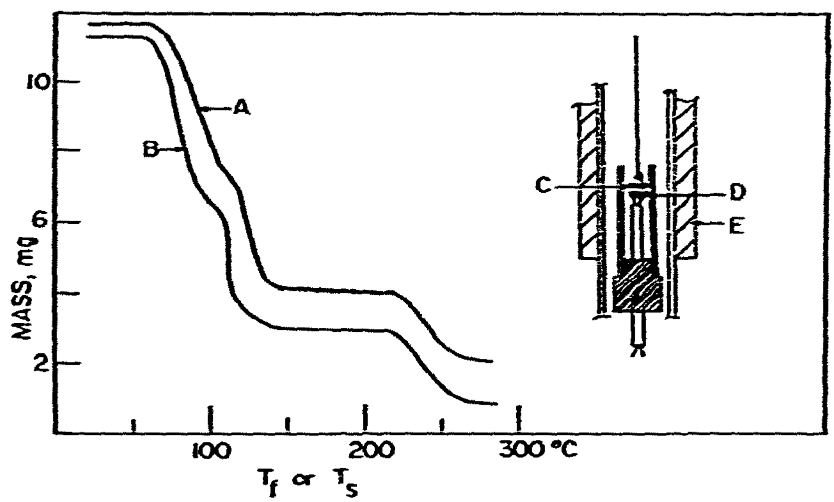

Fig. 1. TG curves and apparatus configuration for two-sample container system. (A) = TG curve of $CuSO_4 \cdot 5\,H_2O$ plotted as a function of T_f (10°C min^{-1} in N_2); (B) = TG curve of $CuSO_4 \cdot 5\,H_2O$ plotted as a function of T_s (same conditions). C = Balance sample container; D = fixed sample container with thermocouple; E = furnace.

balance. Obviously, two identical samples of approximately the same mass must be employed, one for the balance container and the other for the fixed container. The output from the fixed container thermocouple is connected in series to a 0 °C reference thermocouple and then recorded on the X-axis of an X–Y recorder. If the fixed container does not contain a sample, it detects the furnace rather than the sample temperature.

The TG curves of $CuSO_4 \cdot 5H_2O$ plotted against T_s and T_f are illustrated in Fig. 1. Curve A, which is the curve for the dehydration reactions, is a recording of the loss of mass as a function of furnace temperature, T_f, while curve B uses the sample temperature, T_s, as the recorded variable on the X-axis. Due to the large endothermic reaction that occurs during the 3 → 1 hydrate transition, the T_s curve is somewhat different in appearance from the T_f curve in this mass-loss region. However, there is little difference in the 1 → 0 hydrate transition region of the curve.

The two sample-two container system is convenient to use, although it does employ two separate samples. Most of the TG curves obtained by this method at least for hydrated salts, have increased "resolution" which makes it easier to detect intermediate compounds that may be formed during the thermal decomposition process.

ACKNOWLEDGEMENT

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REFERENCE

1 W. W. Wendlandt, *Thermal Methods of Analysis*, Wiley-Interscience, New York, 2nd ed., 1974, p. 79.