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Short Communications

ON THE ACHIEVEMENT OF UNIFORM PACKING FOR DTA SAMPLES

W. H. FLANK

Houdry Laboratories, Air Products and Chemicals, Inc., Linwood, Pa., U.S.A.

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A simple device is described for ensuring uniform sample packing in DTA.

Differential thermal analysis (DTA) is a physico-chemical characterization technique that has experienced a very large growth in popularity in recent years. This has been due, at least in part, to improvements in equipment and materials used in thermal analysis studies. One of the most widely used commercial instruments is the Du Pont Model 900 Differential Thermal Analyzer, as data from a recent round-robin study have shown [1].

In attempting to obtain reproducible results on a series of finely divided inorganic materials using the Model 900 accessory high temperature cell, it was found that sample size and the packing density of the sample were difficult to control when customary packing methods were used and different people performed the experiments. Lack of reproducibility and the significant effects of differences in sample mass, thermal conductivity and thermal diffusivity, due to differences in the bulk density of the packed sample, are well known in the field of thermal analysis (see, for example, [2]).

A simple sample packing device was fabricated to overcome these problems and is shown diagrammatically in Fig. 1. The base and the 3-legged sample cup holder (only two legs of which are shown) are made of brass. The legs are threaded brass rods screwed into the body and positioned with brass nuts to seat the holder in a level position in conical holes drilled into the base. The sample cup cavity is thus kept in a plumb position. Mounted on the base plate with an adjusting nut is a stainless steel threaded rod shaped into a rounded tip at the end. This tip makes an indentation in the bottom of the platinum foil liner of the 4 mm diam. platinum sample cup, as shown in the exploded view in Fig. 1, in the same way as does the thermocouple bead when the shank of the filled sample cup is mounted over the bead in the high temperature cell of the instrument. Thus, disturbance of the sample in subsequent handling is minimized. The stainless steel packing tool is shown in tamping position, packing the sample into the cup in increments by virtue of its 64 g mass. The other parts of the tool are for reshaping the platinum cups and for pushing the shank of the cup out of the holder for easier removal after packing.

Data showing the degree of reproducibility attainable were obtained for a series of similar samples whose decomposition temperature and recrystallization temperature were being determined. Other details of this study have been published elsewhere [3]. The mean packed weight for the 13 samples in the series for which such data were obtained was 64.9 mg, with a standard deviation of 3.8. For repeat runs with the same sample, the standard deviation in the weights was below 1 mg. Weight loss data obtained after a number of replicate runs showed a standard deviation of 0.1 wt. percent.

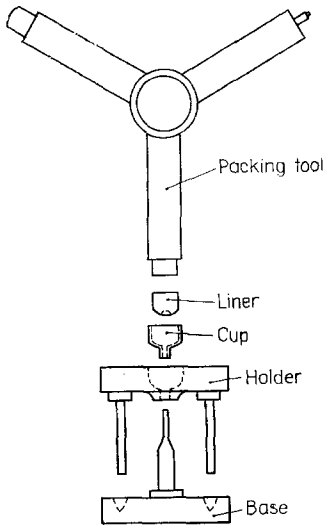

Fig. 1. DTA sample packing device

For the decomposition and recrystallization temperatures measured in the range 800–1100° on the several samples, the standard deviations were found to be less than 7° in all cases. A sample run without the aid of the packing device gave a result more than seven standard deviations from the mean, and another gave a result more than three standard deviations from the mean in the opposite direction. These and numerous other data obtained over a period of several years amply demonstrate the improvement in reliability of DTA data obtainable with the use of such a device for uniform sample packing.

References

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