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Note

Constant sensitivity differential thermal analysis

Some instrumentation variables

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The technique of constant sensitivity differential thermal analysis was introduced by Williams and Wendlandt[1] in 1971. In this technique, the ΔT parameter of the system is maintained invariant with temperature resulting in a constant calibration coefficient. Such a system is useful in quantitative DTA studies because it eliminates multipoint calibration and also simplifies calculations. Although the system has been described in detail[1], we wish to report here the effect of two additional instrumental parameters—the composition of the thermosleeves and the sample mass.

EXPERIMENTAL

The apparatus employed has been described in detail elsewhere[1,2].

RESULTS AND DISCUSSION

Effect of thermosleeve composition

Since the samples are contained in 1.6–1.8 mm diameter glass capillary tubes, their temperatures are detected by the ΔT thermocouples via metal thermosleeves. The capillary tubes are inserted into the sleeves which are positioned over and make contact with the thermojunctions. Obviously, the heat conductivity and the mass of

TABLE 1

EFFECT OF THERMOSLEEVE COMPOSITION ON INDIUM FUSION PEAK AREA AND HEIGHT

Thermosleeve composition	*Peak area* (mm^2)	*Peak height* (*mm*)
Aluminum	871	194
Silver	826	188
Gold	794	154

the thermosleeve will affect the ΔT sensitivity and hence the curve peak area. Using sleeves constructed from aluminum, silver and gold, the peak parameters obtained for the fusion of equal masses of indium metal are shown in Table 1. As can be observed, the aluminum sleeve gave the highest ΔT sensitivity, followed by silver and then gold. From these results, aluminum was selected for the apparatus, as previously described[1].

Effect of sample mass

To be useful for quantitative DTA studies, the curve peak area for a given sample must be directly proportional to the sample mass. The effect of sample mass on peak area (for the fusion of sulfur, S_8) is shown in Fig. 1. As can be seen from the

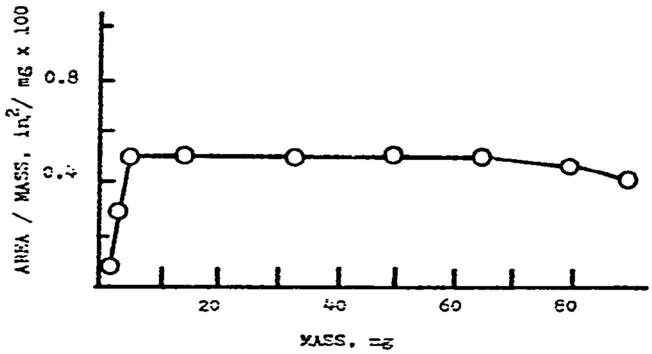

Fig. 1. Effect of sample mass on the curve peak area: mass ratio for the fusion of sulfur.

curve, the peak area to mass ratio was not independent of sample mass below about 8 mg nor above 70 mg. The low results at small sample sizes are due to the heat sink effect of the glass capillary tube and thermosleeve. At the larger sample sizes, inefficient heat transfer to the thermosleeve results in lower peak areas. Thus, sample sizes must be chosen in the constant *area:mass* ratio range for the sample under investigation.

ACKNOWLEDGMENT

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REFERENCES

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