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Note

Use of diphenyl ether as a differential scanning calorimetric calibrant

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To our knowledge, no standards have been proposed for DSC calibration near or below room temperature which have the conveniences of the indium, tin and lead used routinely for higher temperature calibration. These conveniences include availability, purity, ease of handling, and accurately known heats and temperatures of transition. In the widely used Perkin-Elmer DSC-1B system, temperature calibration is not linear throughout the instrument operating range owing to inherent non-linearities of the temperature sensing system. Thus temperature calibration using indium (m.p. = 154.6 °C) will lead[1] to indicated temperatures which are low by 5–10° at 50 °C. However, many other substances such as n-octane (m.p. = 56.5 °C) and benzene (m.p. = 5.5 °C) may be used as temperature calibrants. Although the heats of fusion of both n-octane and benzene are known (43.21 cal g^{-1} and 30.45 cal g^{-1}, respectively)[2], their volatility makes them inconvenient as power calibrants. Thus n-octane has a vapor pressure of 10 torr at 19 °C while benzene exhibits this vapor pressure at − 11.5 °C (ref. 2). Consequently, it is difficult to obtain weighed samples for calibration owing to evaporation, and this is particularly troublesome for benzene. Since power calibration is nearly independent of temperature range, use of one of these substances as a temperature calibrant and a different substance such as indium as a power calibrant is also possible. However, it is convenient to use the same sample for both types of calibration because both may be performed simultaneously. Gallium (m.p. = 29.8 °C), which might be thought to be useful as a calibrant, is unsatisfactory because of its tendency to alloy with aluminum, the material commonly used for sample pans.

We have found diphenyl ether to be highly useful for both temperature and power calibration near room temperature. This compound is readily obtained from commercial sources, is inexpensive, and can be purified readily[3]. It melts at 26.87 °C and has a heat of fusion[4] of 24.173 cal g^{-1}. The compound has a low volatility (v.p. = 1 torr at 66.1 °C)[2] and consequently can be handled easily without loss of sample. Nevertheless, in order to allow extended use of the diphenyl ether samples as power calibrants, in our studies we have mounted them in sealed sample plans using a Perkin-Elmer volatile sample sealer accessory. The calibration samples are then weighed periodically during periods of use to check for sample loss and stored in a refrigerator when not in use. Because diphenyl ether exhibits a great tendency to supercool, only the melting process was used for temperature calibration.

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REFERENCES

1 M. J. O'Neill and R. L. Fyans, presented at the *Eastern Analytical Symposium, New York, Nov. 1971*; Perkin-Elmer Reprint MA-9.
2 R. C. Weast (Ed.), *Handbook of Chemistry and Physics*, Chemical Rubber Company Press, Cleveland, Ohio, 54th ed., 1973.
3 See, for example, J. C. Cases, *J. Chem. Educ.*, 50 (1973) 420.
4 G. T. Furukawa, D. C. Ginnings, R. E. McCoskey and R. A. Nelson, *J. Res. Natl. Bur. Stand.*, 46 (1951) 195.