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## Note

# CALIBRATION OF THERMOBALANCE TEMPERATURE SCALES

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Elder [1] has noted the quite large spans in temperature, and consequent large standard deviations, reported for bottom-loaded thermobalances in the certification program carried out by the International Confederation for Thermal Analysis [2]. In a system calibrated using another set of reference materials, he found that the set provided measured temperatures in a subsequent run that obeyed "a reasonably well-defined linear relationship" when the deviation of the given value was plotted against temperature whereas the ICTA Certified Reference Materials "deviate randomly and markedly from this linearity." There is, of course, a misunderstanding of the data provided in the Certificate for the ICTA materials. (These materials are certified by the ICTA and marketed by the U.S. National Bureau of Standards as GM 761.)

The data on the individual temperature values are given in the Certificate principally to inform the user of the magnitude of variation in measured temperatures that can be found in different instruments. It is important for a user to know that a weight change for which he has measured a temperature fifteen degrees different from that reported in another laboratory might still be the same weight change occurring at the same temperature. The Certified Reference Materials provide a means of relating the measurements. The major source of variation is the relation between the temperature sensor and the sample, both in position and distance, and these parameters are generally fixed by the thermobalance design.

The average values calculated from the eighteen reports are not advanced as "best values". They may, however, be used as values from which deviations can be ascertained and reported. In this they serve an important function. They cannot serve the function for which Elder had wished to use them, viz. the setting of a known sample temperature during a chemical process by setting a control system calibrated in the absence of any heat effect. It is worth noting that the deviations found by Elder were within the range reported by users of bottom-loaded thermobalances [2,3].

* The author is responding as Chairman of the International Confederation for Thermal Analysis' Committee on Standardization. Other current members of the Committee are: E.L. Charsley, P.K. Gallagher, K. Heide, G. Lombardi, R.C. Mackenzie, E. Marti, H.G. McAdie, F. Paulik, J.P. Redfern, O.T. Sørensen and R.H. Still.

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Accurate knowledge or control of temperature of the sample during a heat-evolving or heat-absorbing process can be obtained in two ways, by use of a sensor in direct contact with the sample or by separate differential methods. The first is impractical in the very sensitive system chosen by Elder. The alternative, though, is feasible. In a separate experiment without actual weight change measurements, a thermocouple can be put in contact with the sample and the temperature difference between that thermocouple and the instrument thermocouple measured while the chemical process is going on [4].

This kind of measurement would have to be repeated for each set of substantially-varying experimental conditions but the more-certain knowledge of the actual sample temperature will generally warrant the effort. Reporting of the indicated temperatures for the Certified Reference Materials would still be a service to other experimenters trying to duplicate or evaluate the work.

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