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Note

A remark on "a simple method for recording correct sample temperature in thermogravimetry"

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We would like to confirm the importance and support the general applicability of the note by Wendlandt[1], who recommended the use of two geometrically similar crucibles for a more precise temperature mesaurement in all cases where a direct contact between the sample and a temperature sensor is impossible. This arrangement is suitable, for example, for thermogravimetry or thermomagnetometry, where the investigated sample is placed on a crucible suspended below the balance shoulder while the temperature is measured on the geometrically similar crucible supported by a temperature sensor (and/or control) wiring and mounted just underneath the upper sample crucible. From our own experience, particularly when this is applied to IR heating[2–4], we can say that the agreement between the sample and reference temperatures depends upon

(1) the identity of shape, thermal inertia, etc.;

(2) the properties of the surfaces (colour, reflectivity, etc.);

(3) the weight of the investigated sample and its even distribution;

(4) the mutual position of both crucibles with regard to the temperature gradient caused by heating;

(5) the amount of heat conducted out by supporting wires. Points (1) and (2) are usually solved by a special construction[2], point (3) is best worked out by spreading the sample on the internal (unheated) surface of both crucibles in the form of a mechanical suspension in a suitable liquid[4], while points (4) and (5) can only be satisfactorily solved by calibration.

It is recommended that both crucibles be removable in order to facilitate the cleaning of the rest of the reacted sample, preferably by placing the crucibles on fixed discs to assure good heat conductivity.

If the temperature distribution is homogeneous enough, the accuracy of such temperature measurements is in the order of magnitude of one degree Kelvin[3], while the ordinary measurements by naked thermocouple junction can produce errors of tens of degrees Kelvin[5].

Further improvements can be achieved by calibration, where measurement of the temperature difference between the sample and the reference crucibles is made prior to the entire experimentation. Practically, it is accomplished by suspending the upper crucible on two thermocouple wires and measuring the temperature difference as a function of increasing temperature for the given heating rate. Additional accuracy can be attained by checking the absolute temperatures of at least three reference points; for example, optically by placing a few crystals of weight stable substances on the upper crucible and observing their melting points, e.g. dinitrobenzene (90 °C), KNO_3 (333 °C), Ag_2SO_4 (652 °C) (but be careful of later evaporation changes), or magnetometrically, by addition of substances of known Curie points as ICTA recommended alloys or, better, stable magnetic oxides such as $Y_3ScFe_4O_{12}$ (20°C), $Y_3Fe_5O_{12}$ (278°C) or $NiFe_2O_4$ (585°C) which exhibits particularly sharp transitions[6]. From the correlation of these absolute temperatures with the calibration graph, the accuracy of the temperature measurements can then be as good as $\pm$ 0.5 K.

Thus we shall be very glad to see the suggestion of Wendlandt[1] receive deserved attention by manufacturers of commercial equipment, as it was the case of our suggestion to use ribbed (multideck) crucibles to decrease the temperature gradient in the layer of a sample so placed on a relatively large contact surface[5,7,8], which was finally used in commercial accessory crucibles for the Derivatograph thermobalance[9].

NOTE ADDED IN PROOF

The analogy between the low-temperature IR radiation and normal radiation, decisive for the heat transfer mechanism at higher temperatures, is assumed in order to take the rules listed above to be generally valid in high-temperature thermogravimetry, etc.

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