A New In-Situ Electrical Calibration System for High Temperature Calvet Calorimeters

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Thermal analysis and calorimetry are used in many academic and industrial laboratories to study the thermal behavior of materials for research or quality control. The existing international standards recommend a calibration of the calorimeters and DSCs (Differential Scanning Analyzers) with respect to temperature and heat by using the melting points of standard reference materials. These reference materials are usually those used to define the international temperature scale, namely gallium, indium, tin, zinc, aluminum and silver. However there are few certified reference materials above 400 °C, while the operating range of some thermal analyzers and calorimeters exceeds 1000 °C. Moreover, there is a strong need for metrological traceability in the field of calorimetry due to the increasing implementation of quality insurance systems. To address these requirements, LCM-LNE has been working in the development of a metrological standard facility. The final aim of this study is to be able to certify reference materials in terms of specific heat and heat of fusion over the temperature range [23 °C, 1000 °C]. The metrological approach is based on the modification of a commercial Calvet calorimeter and of the procedures implemented for calibration and measurement, so as to get measurement uncertainties sufficiently low to fulfill the objectives of the certification of reference materials. A high temperature in-situ calibration system (constituted by a resistance wire wound around the crucible containing the material sample) was integrated into the calorimeter to perform the calibration by electrical substitution. The system allows both calibration and measurement without modification of the apparatus, so that the experimental conditions during both steps remain unchanged. This paper describes in details the design of the calibration system as well as the calibration procedure implemented. First results obtained on calibration of the calorimeter and determination of the enthalpy of fusion of tin are presented and discussed.