DEVELOPMENT OF METROLOGICAL ASSURANCE IN THE FIELD OF THERMAL ANALYSIS AND CALORIMETRY

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As stated in a lately accepted definition, "Thermal analysis (TA) is the study of the relationship between a sample property and its temperature as the sample is heated or cooled in a controlled manner" [1]. Due to the diversity of the types of studied samples and their measurable properties, a broad range of experimental techniques is currently employed in TA, with new methods continuously developing alongside scientific and technological progress.

Obtaining reliable measurement results, that are suitable for reasonable interpretation, requires performance evaluation for both methods and instrumentation. From a metrological perspective, it requires a well-defined traceability chain, established procedures, and appropriate reference standards. However, such means of metrological assurance are not available for some TA and calorimetric techniques, particularly for recently developed or rapidly evolving methods.

The goal of this study is to evaluate the current state of metrological assurance in TA and calorimetry, highlighting known issues, perspectives for development and available alternative tools in modern knowledge-intensive fields of TA application.

An overview of TA and calorimetric techniques is presented, covering both classical methods, such as thermogravimetric analysis (TG), bomb calorimetry, and differential scanning calorimetry (DSC), and more recent or specialized approaches.

As expected, largest deficits in metrological assurance are observed for newer techniques (i.e. Chip Calorimetry). Metrological assurance for classical methods is mostly well established, although for some well-known techniques such as Thermomechanical Analysis (TMA) and Dynamic Mechanical Analysis (DMA) a shortage of suitable reference materials and standardized procedures is observed. Similar issues are reported for classical methods operating near the limits of measurement ranges (e.g., low-temperature DSC, microcalorimetry, high-temperature dilatometry) [2,3]. Some cases where reliability of measurement results is negatively affected by the lack of metrological assurance are presented in detail.

Based on the analysis, general recommendations are formulated for performance evaluation in thermal analysis and calorimetry. Where available, official means of metrological assurance are presented, provided by metrology institutes in Russia and internationally. In addition, non-certified reliable sources, such as IUPAC recommendations, are considered when relevant. Current limitations in metrological assurance are summarized and identified as priority areas requiring further development.

1. Lever T. et al., *Pure Appl Chem.*, 2014, 86(4), 545–553
2. Ashton G. et. al,, *Thermochimica Acta*, 2021, 698
3. Medoz Z. et. al., *Analytical Biochemistry*, 2024, 694