STANDARD REFERENCE MATERIALS OF THE MELTING POINT OF PURE ORGANIC SUBSTANCES IN THE RANGE FROM +40 TO +250 °C

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VNIIM has completed development and type approval of the sets of certified reference materials (CRMs) for phase transition temperatures in the range of +40 to +250 °C: 1st GSO 12820/12822-2025 and 2nd GSO 13150/13152-2025. The sets comprising several types of melting point CRMs based on high-purity organic substances: benzophenone, benzoic acid, caffeine (1st set) and acetanilide, biphenyl, benzoguanamine (2nd set).

Development of the new set of CRMs was necessary due to solve several issues with the current tools and procedures used for of melting point measurement of organic materials. Current state revealed the absence of a unified approach to determine the metrological characteristics of measuring instruments, including procedures for their verification and calibration. This issue appears from different interpretation of the melting temperature definition as a thermodynamic property of a substance when using different measurement methods. Significant discrepancies in experimental data obtained by various techniques (particularly at different heating rates) were also noted, along with difficulties in interpreting both the obtained data and the observed discrepancies.

These factors adversely affect measurement uniformity in this field. Consequently, a novel approach was implemented in the development of the CRMs, anticipated to reduce the effects of these shortcomings.

The certified value of the developed CRMs was determined through direct measurements using the State Working Standard of Temperature (Grade 2) in the range of +40 °C to +250 °C (3.1.ZZB.0453.2024), in accordance with Part 2 of the State Verification Scheme for Temperature Measuring Instruments. The standard setup consists of two precision thermometric systems: one implementing the melting process in a custom-designed cell under constant heat flux, and the other using a dry-block thermostat with direct contact between the temperature sensor and the substance.

In the author's opinion a generalized method- and instrument-independent definition of melting point should be used, described as the temperature at which thermodynamic equilibrium between the solid and liquid phases of a substance can be achieved, provided that the liquid phase retains its initial composition and the amount of solid-phase crystals tends to zero.

The certified property of the CRMs is a melting point. It was determined as the temperature corresponding to the local minimum of the first derivative (or the point where the second derivative changes sign) within the two-phase region (melting plateau) when the liquid fraction is getting closer to 1.

The experiments were conducted under near-equilibrium conditions, with a heating rate in the range (0.2 – 1.0) °C/min, and temperature measurements were performed using a temperature sensor in direct contact with the substance (traceable to ITS-90).