NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
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Standard Reference Material® 2232a

Indium for DSC Temperature and Enthalpy Calibration

CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are primarily intended for use in the temperature and enthalpy calibrations of differential scanning calorimeters (DSCs), differential thermal analyzers (DTAs), and similar instruments.

Description: A unit of SRM 2232a consists of an approximately 1 g ingot of indium metal sealed in plastic under an inert atmosphere.

Certified Values: The certified melting temperature and enthalpy of fusion for SRM 2232a are provided in Table 1. A NIST certified value is a value that is traceable to the International System of Units (SI) and for which NIST has the highest confidence in its accuracy [1]. Included in Table 1 are the estimated expanded uncertainties. The expanded uncertainty is calculated as $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor corresponding to approximately 95 % confidence [2,3]. For the certified values shown in Table 1, k = 2 and k = 1.93 for temperature and enthalpy, respectively.

Table 1. Certified Melting Temperature (t_m) and Enthalpy of Fusion (ΔH_{fus}) for SRM 2232a[(a)]

t_m	(156.5985	± 0.0003) °C
ΔH_{fus}	(28.58	± 0.17) J·g^{-1}

(a) Values are expressed as, $x \pm U(x)$ where x is the certified value and $U(x)$is the associated expanded uncertainty. The true value lies within the interval $x \pm U(x)$ with 95 % confidence.

Non-Certified Values: Non-certified heating rate dependent enthalpy of fusion values are provided in Appendix A.

Additional Information: Additional information is provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 2232a** are valid indefinitely within the measurement uncertainty specified. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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Safety: SRM 2232a is intended for research use. Consult the safety data sheet for additional information.

Storage: SRM 2232a should be stored in a sealed container at room temperature (20 °C ± 5 °C). Storage temperatures should never exceed 155 °C.

Use: A clean, sharp blade can be used to slice off samples of the appropriate size and shape for use in instrument calibration. Every possible effort should be made to avoid contamination during handling procedures.

Analysis: Value assignments for the melting temperature and enthalpy of fusion of SRM 2232a were based on a combination of measurement techniques, with all analyses performed at NIST [4]. Additional information is provided in Appendix B.

REFERENCES

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If you use this SRM in published work, please reference:

Fortin TJ, Herman TK, Koepke AA, Splett JD (2024) Certification of Standard Reference Material® 2232a: Indium for DSC Temperature and Enthalpy Calibration. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-242. https://doi.org/10.6028/NIST.SP.260-242

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APPENDIX A

Non-Certified Values: The non-certified heating rate dependent enthalpy of fusion values for SRM 2232a are provided in Table A1. A NIST non-certified value is defined as being suitable for use in method development, method harmonization, and process control but lacking either adequate accuracy, metrological traceability, or sufficient documentation [1]; however, in this instance, all three of these criteria have been met. The heating rate dependent enthalpy of fusion values are included as non-certified values because they are considered of limited use for calibration, as is discussed in Appendix B. They are included here simply for reference. Also included in Table A1 are the estimated expanded uncertainties. The expanded uncertainty is calculated as $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor corresponding to approximately 95 % confidence [2,3]. Corresponding k values are shown below.

Table A1. Non-Certified Heating Rate (β) Dependent Enthalpy of Fusion Values (ΔH_{fus}) for SRM 2232a

β[(a)] °C·min^{-1}	ΔH_{fus}[(b)] J·g^{-1}	k[(c)]
1	28.61 ± 0.16	1.94
3	28.67 ± 0.14	1.97
5	28.72 ± 0.15	1.96
10	28.87 ± 0.26	1.91

(a) Nominal heating rate.
(b) Values are expressed as $x \pm U(x)$ where x is the non-certified value and $U(x)$ is the associated expanded uncertainty. The true value lies within the interval $x \pm U(x)$ with 95 % confidence.
(c) Coverage factor corresponding to approximately 95 % confidence.

Period of Validity: These non-certified values are valid indefinitely within the measurement uncertainty specified. The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Value: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Certificate of Analysis and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Analysis: Value assignments for the enthalpy of fusion as a function of heating rate (Table A1 above) were based on DSC measurements performed at NIST. Details can be found in reference 4.

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Appendix B

Source and Preparation: The material for SRM 2232a was sourced from a single lot (lot # 135139138114-400) of high-purity indium metal purchased from Merelex Corporation, Los Angeles, CA. The material was packaged as approximately 1 g ingots sealed in plastic under an inert environment. The supplier's Glow Discharge Mass Spectrometry (GDMS) analysis results indicate a total impurity (on a metals basis) of approximately 0.1 parts per million (ppm, or $\mu g \cdot g^{-1}$). A purity of ≥99.99999 % (metals basis, by mass) was independently confirmed via GDMS analysis conducted by the National Research Council of Canada (NRC Canada, Ottawa, Ontario, Canada).

Melting Temperature Analysis: Determination of the melting temperature employed a fixed-point cell constructed from randomly selected SRM 2232a samples and included the evaluation of freezing and melting curves, as well as direct comparison with the laboratory reference cell used to realize the indium freezing point in the NIST Standard Platinum Resistance Thermometer Calibration Laboratory (SPRTCL). Additional details can be found in reference 4.

Enthalpy of Fusion Analysis: Determination of the enthalpy of fusion employed DSC measurements on 15 samples taken from seven randomly selected ingots of SRM 2232a. The DSC used for these measurements was calibrated for both temperature and enthalpy using certified reference materials procured from the national metrology institute of Germany (Physikalish-Technische Bundesanstalt (PTB)). Because DSC measurements are highly sensitive to experimental parameters such as heating rate (β) [5–7], measurements on the 15 SRM 2232a samples were made at four different heating rates (1 °C min^{-1}, 3 °C min^{-1}, 5 °C min^{-1}, and 10 °C min^{-1}), and a β-dependent enthalpy of fusion value determined for each (see Table A1). However, since individual instruments will presumably exhibit their own particular dependency, β-dependent information is only of limited use; for a certified reference material the enthalpy of fusion at thermal equilibrium is the value of interest. Therefore, the β-dependent values were extrapolated to determine the enthalpy of fusion at β = 0 °C min^{-1}. This value is what is reported as ΔH_{fus} in Table 1. Additional details can be found in reference 4.

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