National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2225

Temperature and Enthalpy of Fusion - Mercury

Standard Reference Material (SRM) 2225 is intended for use in calibrating differential scanning calorimeters, differential thermal analyzers, and similar instruments. This SRM consists of 2.5 gm of very high purity mercury. It is packaged in a vial with a septum through which a sample of mercury may be withdrawn by a syringe.

The certified values of the fusion temperature and enthalpy of fusion of SRM 2225 were determined by adiabatic calorimetry. The certified values are:

Fusion Temperature (K)	Enthalpy of Fusion (J/g)
234.30 ± 0.03	11.469 ± 0.008

The certified fusion temperature is the average of the midpoints of the results of three separate equilibrium measurements. The uncertainty is expressed as the range of fluctuation of the temperature about the midpoint over an extended period of time at the equilibrium condition. The certified enthalpy value is the average of 5 enthalpic determinations and the listed uncertainty is three times the standard error of the measured value. Systematic error in temperature is within the variability indicated for temperature; the systematic error in enthalpy is estimated to be less than 0.003 J/g. Details of the measurement procedures for both the adiabatic and differential scanning calorimetric measurements are reported in the literature [1].

The adiabatic calorimetric measurements were performed by J.E. Callanan of NIST in the laboratory of E. F. Westrum, Jr. at the University of Michigan.

The differential scanning calorimetry measurements were made by K.M. McDermott under the technical direction of J.E. Callanan, both of the NIST Engineering Science Division.

Statistical design and analysis of the scanning calorimetric measurements were provided by D.F. Vecchia of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.L. McKenzie.

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Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(Over)

Material Specifications: The mercury used for SRM 2225 is from the same stock as that for SRM 743, "Mercury, Triple Point on the International Practical Temperature Scale (1968), -38.841 °C" (234.309 K) [2]. The mercury used for SRM 743 is of exceptional purity, with the total of other elements estimated to be less than 20 parts per billion (ng/g) by emission spectrographic analysis. The fusion temperature determined in this study by heating into and out of the melting transition at 0.01 K/min was 234.32 ± 0.03 K where the uncertainty is expressed in terms of the range of values. The melting point reported by Busey and Giauque, when converted to the IPTS-68 scale, is 234.30 K; the enthalpy of fusion is reported to be 548.6 cal. $deg.^{-1}$ $mole^{-1}$ (11.44 J/g), which represents the average of three values with a standard deviation of a single value of 0.1[3].

Additional Information: The temperature and enthalpy of fusion were also measured by differential scanning calorimetry (dsc) and the results are presented as noncertified supplemental information. These values are presented because, though they are not certified, they may be of use in the application of SRM 2225 for calibration of dsc measurements.

Sixteen specimens of mercury with masses ranging from 13.983 to 67.971 mg were sealed in a nitrogen atmosphere and scanned at 2.5 K/min. The samples were prepared in uncoated aluminum pans. To verify that the mercury was not forming an amalgam with the pan, a sample of mercury was placed in a preweighed pan in an ambient atmosphere for several days, then the mercury was removed and the pan reweighed. There was no detectable increase in weight of the pan. Samples have also been rerun a number of times and continue to give the same results, indicating that the integrity of the sample is preserved when prepared in nitrogen in uncoated aluminum pans.

The fusion temperature obtained by dsc measurements was 234.34 ± 0.36 K. The uncertainty is three times the standard error of the average value and includes contributions to variability from instrumental factors, operating procedure, and effects of remounting the specimens. The temperature-of-fusion values were obtained by the usual procedures and corrections [4,5].

Enthalpy-of-fusion measurements, also made for these sixteen specimens, all agreed within 0.1 J/g. Enthalpy-of-fusion values obtained by dsc are not absolute measurements and the values must be corrected by appropriate calibration or correction factors [5]. The results in this case could not be corrected by the usual procedure using bracketing materials due to the lack of suitable bracketing materials in this temperature range. The measurements do provide a good indication of the precision attainable, however. The standard deviation of the results of the enthalpy-of-fusion measurements about the mean was ± 0.03 J/g. This result demonstrates that mercury may be measured by dsc with good precision (relative standard deviation of 0.3%) and therefore may be used with confidence as a calibrant for subambient dsc measurements using the certified enthalpy value obtained by adiabatic calorimetry.

References:

1. Callanan, J.E., McDermott, K.M., and Westrum, E.F., Jr. "Fusion of mercury. A new certified standard for differential scanning calorimetric use." *J. Chem. Thermodynamics*, submitted for publication.
2. National Bureau of Standards Certificate for SRM 743. Gaithersburg, MD, April 22, 1976.
3. Busey, R.H., and Giauque., W.F. "The heat capacity of mercury from 15 to 330 K. Thermodynamic properties of solid, liquid and gas. Heat of fusion and vaporization." *J. Amer. Chem. Soc.*, 75: 806-809, 1953.
4. Callanan, J.E., Sullivan, S.A., and Vecchia, D.F. "Standards development for differential scanning calorimetry." J. *Res. Nat. Bur Stds. (U.S.)* 91(3): 123-129; 1986.
5. Callanan, J.E., and Sullivan, S.A. "Development of standard operating procedures for differential scanning calorimeters." *Rev. Sci. Instrum.* 57(10): 2584-2592, 1986.