National Institute of Standards & Technology

Certificate

Standard Reference Material 2222

Temperature and Enthalpy of Fusion - Biphenyl

Standard Reference Material (SRM) 2222 is intended for use in calibrating differential scanning calorimeters, differential thermal analyzers, and similar instruments. This SRM is made from a sample of high purity biphenyl.

The temperature and enthalpy of fusion of SRM 2222 were measured by differential scanning calorimetry. The measurement procedure used for certification followed the American Society for Testing and Materials (ASTM) recommended practices [1, 2]. The documentation for these procedures is given in NBS Special Publication 260-99 [3]. The certified values for SRM 2222 are:

Melting Temperature*	Enthalpy of Fusion
342.41 ± 0.27 K	120.41 ± 0.57 J/g

*Heating rate of 2.5 K/min

The certified values are averages of duplicate measurements made on each of eighteen specimens of the SRM. The uncertainty values are three times the estimated standard error of the certified values. These uncertainties include contributions to variability from instrumental factors, operating procedures, and effect of remounting the specimens [3].

The measurements were performed by K.M. McDermott under the technical direction of J.E. Callanan, both of the NIST Chemical Engineering Science Division.

Consultation on the statistical design of the experimental work and statistical analysis of the data was provided by J.M. Mulrow and D.F. Vecchia, both of the NIST Statistical Engineering Division.

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

Gaithersburg, MD 20899
June 1, 1989

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(Over)

Supplemental Information: The following information describes some aspects of the certification that may be useful to the user of SRM 2222. The values presented as supplemental information are not certified and are presented for information only.

SRM 2222 is made from biphenyl which was 99.984 percent pure as determined by gas chromatography. This material is in crystalline form and is easily pelletized. The SRM unit-of-issue is 1 gram which will provide approximately two hundred fifty 4-mg samples.

The SRM material was purified by zone refining (thirty-three passes at 2.54 cm/hr with L/1 = 10). The zone-refined material was determined to be 99.984 percent pure by gas chromatographic analysis with the sample in solution on a 25-m polymethylsiloxane capillary column. The gas chromatographic analysis was made using helium carrier gas, a flame ionization detector, and a column temperature of 523 K. The purification was performed by A.R. McGhie of the Laboratory for Research on the Structure of Matter, University of Pennsylvania. T.J. Bruno of the Center of Chemical Engineering analyzed the material to determine purity.

The specimens measured for the certification of the SRM were sealed under argon to retard any effects of exposure to air. The certified reference samples used as the bracketing standards for temperature and enthalpy determinations and the values assigned to these materials are listed in the following table:

Material	Source	Fusion Temp (K)	Enthalpy of Fusion (J/g)
Naphthalene	NPL (CRM M16-03)	353.39 ± 0.03 [4]	148.627 ± 0.312 [4]
Mercury	NIST (SRM 2225)	234.30 ± 0.03 [5]	11.469 ± 0.008 [5]

The observed temperatures were corrected by the two-point calibration procedure (linear interpolation) recommended by ASTM [1] and discussed in reference 3. Certification measurements were made at a heating rate of 2.5 K/min.

The correction procedure used for the enthalpy measurements was also a linear interpolation of the correction factors obtained by measuring two standards which bracket the anticipated value of the test sample [6].

Some temperature and enthalpy of fusion values for biphenyl that have been reported in the literature are listed below for the purposes of comparison:

Investigator	Year	Method	T(fus) K	ΔH(fus) J/g
Spaght, et. al. [7]	1932	Radiation calorimetry	341.45	120.92
Stull, D.R. [8]	1947	Vapor pressure	342.7	—
Ueberreiter & Orthmann [9]	1950	Nernst calorimeter	343	120.47
Plato & Glasgow [10]	1969	DSC	342.2	119.39
Wauchope & Getzen [11]	1972	DSC	—	122.64 ± 3.3

Shelf-Life: This SRM is anticipated to have an indefinite shelf-life under normal use and storage conditions. We recommend that the SRM be stored at or below ambient temperature, in the dark, protected from moisture. The SRM will be monitored from time to time at NIST; if the value of any parameter significantly changes from the certified value(s), the purchaser will be notified.

References:

1. ASTM Standard Practice for temperature calibration of differential scanning calorimeters and differential thermal analyzers E967-83. *Annual Book of Standards*, **14.02**: 782-787, 1984.
2. ASTM Standard Practice for heat flow calibration of differential scanning calorimeters E968-83. *Annual Book of Standards*, **14.02**: 788-794, 1984.
3. Callanan, J.E., Sullivan, S.A., and Vecchia, F.G. "Feasibility study for the development of standards using differential scanning calorimetry." U.S. National Bureau of Standards *Special Publication 260-99*, 1985, May.
4. NPL Certificate of Measurement, M16-01` and M16-03. National Physics Laboratory, Teddington, U.K.; 1977.
5. NIST Certificate of Analysis, SRM 2225, Mercury. Office of Standard Reference Materials, National Institute of Standards and Technology, Gaithersburg, MD, 20899.
6. Callanan, J.E., and Sullivan, S.A. "Development of standard operating procedures for differential scanning calorimeters." *Rev. Sci. Instrum.*, **57(10)**: 2584-2592, 1986.
7. Spaght, M.E., Thomas, S.B., and Parks, G.S. "Some heat-capacity data on organic compounds, obtained with a radiation calorimeter." *J. Phys. Chem.*, **36**: 882-888, 1932.
8. Stull, D.R. "Vapor pressure of pure substances. Organic Compounds." *Ind. Eng. Chem.*, **39**: 517-550, 1947.
9. Ueberreiter, K., and Orthmann, H.J. "Specific heat, specific volume, temperature- and thermal-conductivity of some disubstituted benzene and polycyclic systems." *Z. Naturforschung*, **5a**: 101-108, 1950.
10. Plato, C., and Glasgow, A.R., Jr. "Differential scanning calorimetry as a general method for determining the purity and heat of fusion of high-purity organic chemicals. Application to 95 compounds." *Anal. Chem.*, **41**: 330-336, 1969.
11. Wauchope, R.D., and Getzen, F.W. "Temperature dependence of solubilities in water and heats of fusion of solid aromatic hydrocarbons." *J. Chem. Eng. Data*, **17**: 38-41, 1972.