National Institute of Standards and Technology

Certificate

Standard Reference Material® 1967a

High-Purity Platinum Thermoelement

This Standard Reference Material (SRM) is a high-purity (approximately 99.999 % mass fraction) platinum wire, suitable for use as a standard reference thermoelement for calibrations of base and noble metal thermocouple materials in the temperature range –196 °C to approximately 1700 °C. NIST Monograph 175 [2] gives standardized thermoelectric voltage versus temperature relations for thermocouples, based on the International Temperature Scale of 1990 (ITS-90) [1]. SRM 1967a replaces the former thermoelectric standard SRM 1967. Historically, SRM 1967 has been commonly designated Pt-67; similarly, SRM 1967a may be referred to as Pt-67a. A unit of SRM 1967a consists of one 0.51 mm in diameter and 1 m long platinum wire.

Certified Electromotive Force Relation: The certified electromotive force (emf) values obtained for SRM 1967a are stated relative to the average thermoelectric response of the former SRM 1967. For a reference junction at 0 °C, the certified difference between the emfs of SRM 1967, E_{67}, and SRM 1967a, E_{67a}, and its expanded uncertainties are given as:

$$E_{67a}(t) - E_{67}(t) = 0\ \mu\text{V}\ \text{, and}\ U_{95}(t) = \begin{cases} 0.2\ \mu\text{V}\ \text{ for } -196\ °\text{C} < t < 0\ °\text{C}, \\ 0.2\ \mu\text{V} + 0.75\ \text{nV}(t/°\text{C})\ \text{for}\ 0\ °\text{C} \le t \le 1700\ °\text{C} \end{cases}$$

where t is the temperature on the International Temperature Scale of 1990. The certified equivalence is for the emf produced by the samples of SRM 1967a relative to that of SRM 1967 and is uncertain within the bounds of the expanded uncertainty, $\pm U_{95}(t)$. The uncertainties account for: the variability of the SRM 1967a batch from its average value; the variability of the SRM 1967 batch from its average value; and the reproducibility of the difference between the emfs of SRM 1967 and SRM 1967a [3,4]. The $U_{95}(t)$ expanded uncertainties represent a confidence interval of approximately 95 %, or a coverage factor of 2. The uncertainty bounds $\pm U_{95}(t)$ have values of approximately ± 1.48 µV at 1700 °C. The measurands are the electromotive force relation values for the platinum wire. The values are metrologically traceable to the SI unit for voltage.

Expiration of Certification: The certification of **SRM 1967a** is valid indefinitely, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Handling, Storage, and Use”). Accordingly, periodic recalibration or recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was under the direction of K.M. Garrity of the NIST Sensor Science Division and D.C. Ripple of the NIST Biomolecular Measurement Division.

Preparation of and analytical measurements on the SRM were performed by K.M. Garrity, and W.L. Tew of the NIST Sensor Science Division and D.C. Ripple.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Gerald T. Fraser, Chief
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Gaithersburg, MD 20899
Certificate Issue Date: 22 September 2015

Robert L. Watters, Jr., Director
Office of Reference Materials

A NIST Special Publication describes the preparation, characterization, and use of SRM 1967a [3].

WARNING TO USERS

Electrical annealing of the platinum wire requires application of lethal voltages to the wire. Appropriate safeguards should be in place to prevent contact of personnel with lethal voltages.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: The SRM 1967a samples consist of high-purity platinum wire. The sample should be handled wearing powder-free gloves. Avoid chemical contamination and kinks in the wire.

Storage: The SRM 1967a wire, as received, should be kept in the original box and bag, and stored at temperatures less than 30 °C.

Use: Proper usage of this SRM requires careful annealing and handling. The recommended procedure is:

1. Clean the wire with a wipe saturated with ethanol and allow it to air dry.
2. Suspend the wire in a clean, dust-free enclosure and connect to a current source.
3. Continuously increase the current until the wire is at a temperature of 1200 °C, as measured by a ratio or disappearing-filament pyrometer with emissivity set to 0.3 (see "Warning to Users" above).
4. Maintain this temperature for 10 minutes.
5. Slowly decrease the current until the wire appears red in color and only slightly incandescent.
6. Maintain this temperature for 60 minutes and then slowly cool to room temperature.
7. Avoid mechanical stress of the wire.
8. High-purity (nominally 99.7 % purity or higher) sintered alumina tubing is recommended for insulating and protecting the platinum wire during use at high temperatures. After assembling the wire in an insulating tube, re-anneal the assembly in a furnace for 60 minutes at 1100 °C followed by cooling to 450 °C. The assembly should be maintained at 450 °C for 12 hours or overnight and then cooled to room temperature.

Users are cautioned that at temperatures above 1300 °C, impurities may diffuse over time into the platinum from even the best alumina. Once the wire has been contaminated at high temperature, there is rarely any mechanism to remove the contamination.

PREPARATION AND ANALYSIS

The wire used for SRM 1967a consisted of 32 m of 0.51 mm diameter platinum wire, obtained from a commercial vendor on a single spool. The SRM was prepared by cuts of the stock into 1 m lengths of wire. Each cut was packaged in a polyethylene bag. Four cuts of SRM 1967a, evenly spaced through the lot, were used as test samples. From a spool of the original stock for SRM 1967, four 1 m long cuts were used as reference samples. Each wire was electrically annealed by the procedure described in "Instructions for Handling, Storage, and Use".

Single-bore alumina insulators of nominal 99.7 % purity (mass fraction) were used to hold and insulate the platinum wires. Lengths of copper wire, all from the same spool, were soldered on to form reference junctions. Finally, all eight of the test and reference samples were welded together with a hydrogen torch to form a common measuring junction.

The emf generated by various combinations of the eight platinum wires were measured at −196 °C, −90 °C, ambient temperature, 200 °C, 450 °C, 700 °C, 900 °C, and 1100 °C. A total of 166 emf comparison measurements were made over this range of temperature. Through statistical analysis of the data, we obtained measures of the thermoelectric homogeneity of the two lots of platinum wire and of the average emf of SRM 1967a relative to SRM 1967.

HOMOGENEITY

During the certification process, material homogeneity was assessed by statistical analysis of the thermoelectric comparison data. The stated uncertainties include components for the homogeneity of both SRM 1967 and SRM 1967a. Analysis and details are reported in ref. [3]. NIST performed thermoelectric homogeneity testing and measurements of thermoelectric voltage relative to SRM 1967 over the temperature range −196 °C to +1100 °C.

REFERENCES

[1] Preston-Thomas, H.; *The International Temperature Scale of 1990 (ITS-90)*; Metrologia, Vol. 27, pp. 3–10, (1990); see also Preston-Thomas, H.; *The International Temperature Scale of 1990 (ITS-90)*; Metrologia, Vol. 27, p. 107, (1990).

[2] Burns, G.W.; Scroger, M.G.; Strouse, G.F.; Croarkin, M.C.; Guthrie, W.F.; *Temperature-Electromotive Force Reference Functions and Tables for the Letter-Designated Thermocouple Types Based on the ITS-90*; NIST Monograph 175 (1993).http://srdata.nist.gov/its90/authors/authors.html

[3] Garrity, K.M.; Ripple, D.C.; Tew, W.L.; *SRM 1967a: High-Purity Platinum Thermoelement*; NIST Special Publication 260-183 (Expected release date 2015).

[4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2015). See also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/tn1297/index.cfm (accessed Sep 2015).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200 fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.