



Certificate of Analysis

Standard Reference Material[®] 2965

Respirable Cristobalite on Filter Media

(Nominal Mass of Cristobalite – 100 µg)

This Standard Reference Material (SRM) is intended primarily for the calibration of X-ray diffraction (XRD) spectrometers for the determination of respirable cristobalite using National Institute for Occupational Safety and Health (NIOSH) Method 7500 [1], or the equivalent (see INSTRUCTIONS FOR USE). The SRM was produced by depositing a known amount of SRM 1879a *Respirable Cristobalite* as a slurry on a 25 mm diameter polyvinyl chloride (PVC) filter. A unit of SRM 2965 consists of five filters, each containing a nominal mass of 100 µg of respirable cristobalite and is provided with five blank PVC filters containing no cristobalite. The blank filters and the loaded filters are stored in two clear plastic petri dishes, respectively, with blue spacer sheets separating the filters.

Table 1. Mass Value of Cristobalite in SRM 2965

Certified Cristobalite Mass: 92.8 µg ± 6.2 µg

Certified Mass of Cristobalite on Filter: The certified mass of cristobalite on each filter, provided in Table 1, is based on results obtained from the gravimetric preparation of the filters and from the determination of silicon by inductively coupled plasma optical emission spectrometry (ICP-OES) [2,3]. The results are expressed as the certified value ± the expanded uncertainty [4]. The gravimetric preparation value of the cristobalite mass per filter was calculated as the average mass of SRM 1879a on each filter multiplied by the certified mass fraction of cristobalite in SRM 1879a. The ICP-OES value for cristobalite mass per filter was calculated from the determined mass of silicon per filter, the determined mass fraction of silicon in SRM 1879a, and the certified mass fraction of cristobalite in SRM 1879a [3].

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [4]. The certified value is the unweighted average of the masses determined by gravimetric and ICP-OES measurements. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined uncertainty due to material variability and measurement uncertainty calculated according to the method described in the ISO and NIST Guides [5]. The coverage factor, $k = 2.31$, is determined from the Student's t -distribution corresponding to the calculated effective degrees of freedom at 95 % level of confidence.

Expiration of Certification: The certification of this SRM is valid until **31 December 2016**, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. The certification is nullified if the SRM is damaged, contaminated, or 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) will facilitate notification.

The coordination of the technical measurements leading to the certification was under the direction of L.L. Yu of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Robert L. Watters, Jr., Chief
Measurement Services Division

Gaithersburg, MD 20899
Certificate Issue Date: 07 February 2007

Analytical measurements were performed at NIST by T.A. Butler and L.L. Yu of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by W.F. Guthrie of the NIST Statistical Engineering Division.

Support for the preparation and certification of this Standard Reference Material was provided by the National Institute for Occupational Safety and Health.

The support aspects involved in the issuance of this SRM were coordinated through the Measurement Services Division.

INSTRUCTIONS FOR USE

This SRM was developed for use with NIOSH Method 7500 [1]. Sample preparation for this method calls for filters to either be ashed or dissolved in tetrahydrofuran prior to XRD measurements. The ashing option must be used with this SRM rather than dissolution. This SRM may also be used with NIOSH Method 7602 [1] using infrared absorption spectrometry.

Sealed filters, as received, should be stored in the dark at temperatures between 15 °C and 25 °C.

NOTICE AND WARNING TO USERS

This material contains respirable cristobalite that is known to cause silicosis. It must be handled with care and disposed of according to federal, state and local regulations.

Cooperating Laboratory¹: The materials for this SRM were prepared at High Purity Standards, Inc. (Charleston, SC).

REFERENCES

- [1] *NIOSH Manual of Analytical Methods (NMAM[®])*, 4th ed.; Cassinelli, M.E.; O'Connor, P.F., Eds.; Department of Health and Human Services (NIOSH) Publication No. 94-113 (1994).
- [2] Yu, L.L.; Fassett, J.D.; Lindstrom, A.P.; *Determination of Si in Standard Reference Material SRM 295x Silica-on-Filter*; J. Anal. At. Spectrom., Vol. 18, p. 738 (2003).
- [3] Yu, L.L.; Fassett, J.D.; MacDonald, B.S.; Butler, T.A.; Ramsey, D.M.; Key-Schwartz, R.J.; Rains, T.C.; *Development of SM295x and 296x, Respirable Crystalline Silica on Filter*; J. ASTM International, Vol. 2, Issue 5 (2005).
- [4] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assessment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000).
- [5] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); see 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).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.

¹Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.