



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2950a

Respirable Alpha Quartz on Filter Media

(Nominal Mass of Alpha Quartz: 5 µg – 500 µg)

This Standard Reference Material (SRM) is intended primarily for the calibration of X-ray diffraction (XRD) spectrometers for the determination of respirable alpha quartz using National Institute for Occupational Safety and Health (NIOSH) Method 7500 [1], or the equivalent (see “Instructions for Storage and Use”). The SRM was produced by depositing a known amount of SRM 1878a *Respirable Alpha Quartz*, as a slurry on a 25 mm diameter polyvinyl chloride (PVC) filter. A unit of SRM 2950a consists of 35 blank PVC filters containing no alpha quartz and 5 loaded PVC filters at each of the following nominal levels: 5 µg, 10 µg, 20 µg, 50 µg, 100 µg, 250 µg, and 500 µg. The blank filters are stored in seven petri dishes each containing five filters. The loaded filters are stored in seven petri dishes each containing five filters of the nominal level.

Certified Mass of Alpha-Quartz on Filter: The certified mass of alpha quartz 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 \pm the expanded uncertainty [4]. The gravimetric preparation value of the alpha quartz mass per filter was calculated as the average mass of SRM 1878a on each filter multiplied by the certified mass fraction of alpha quartz in SRM 1878a. The ICP–OES value for alpha quartz mass per filter was calculated from the determined mass of silicon per filter, the determined mass fraction of silicon in SRM 1878a, and the certified mass fraction of alpha quartz in SRM 1878a [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 taken into account [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/JCGM and NIST Guides [5]. The coverage factor, k , 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 **SRM 2950a** is valid, within the measurement uncertainty specified, until **30 June 2022**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Storage and Use”). 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 or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification was under the direction of L.L. Yu of the NIST Chemical Sciences Division.

Analytical measurements were performed at NIST by L.L. Yu.

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

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Certificate Issue Date: 03 June 2015

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

Support for the preparation and certification of this SRM was provided by NIOSH.

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

Traceability: The measurand is the total mass per filter of alpha quartz and the certified value is metrologically traceable to the SI unit of mass, expressed as micrograms.

Table 1. Mass of Alpha Quartz in SRM 2950a

| Nominal Alpha Quartz Mass (μg) | Certified Alpha Quartz Mass (μg) | | | Coverage Factor (k) |
|--|--|-------|------|----------------------------|
| 5 | 4.85 | \pm | 0.44 | 2.31 |
| 10 | 9.6 | \pm | 1.1 | 2.36 |
| 20 | 19.4 | \pm | 1.2 | 2.20 |
| 50 | 48.6 | \pm | 4.5 | 2.36 |
| 100 | 96.1 | \pm | 4.8 | 2.05 |
| 250 | 245 | \pm | 19 | 2.36 |
| 500 | 483 | \pm | 30 | 2.31 |

INSTRUCTIONS FOR STORAGE AND USE

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

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 for NIOSH Methods 7602 [1] and 7603 [1], and Quartz Analytical Method (P-7) [6] using infrared absorption spectrometry.

NOTICE AND WARNING TO USERS

This material contains respirable alpha quartz that is known to cause silicosis. It must be handled with care and disposed of according to federal, state and local regulations. See Safety Data Sheet.

Cooperating Laboratory⁽¹⁾: The materials for this SRM were prepared at High Purity Standards, Inc. (Charleston, SC).

⁽¹⁾ Certain 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.

REFERENCES

- [1] NIOSH, *Manual of Analytical Methods (NMAM®)*, 4th ed.; Cassinelli, M.E.; O'Connor, P.F., Eds.; United States Department of Health and Human Services; NIOSH Method 7500 (2003); available at <http://www.cdc.gov/niosh/docs/2003-154/pdfs/7500.pdf> (accessed Jun 2015).
- [2] Yu, L.L.; Fassett, J.D.; Lindstrom, A.P.; *Determination of Si in Standard Reference Material SRM 295x Silicon-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, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260–136, U.S. Government Printing Office: Washington, DC (2000); available at <http://www.nist.gov/srm/upload/SP260-136.PDF> (accessed June 2015).
- [5] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jun 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/index.cfm> (accessed Jun 2015).
- [6] OSHA, *Infrared Determination of Quartz in Respirable Coal Mine Dust*, United States Department of Labor, Occupational Safety and Health Administration (OSHA), Quartz Analytical Method (P-7) (1994), available at <https://www.osha.gov/dsg/topics/silicacrystalline/p7/p7.html> (accessed Jun 2015).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2000; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm_