National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1878b

Respirable Alpha Quartz

(Quantitative X-Ray Powder Diffraction Standard)

This Standard Reference Material (SRM) is intended for use in preparation of calibration standards for quantitative analyses of α-quartz by X-ray powder diffraction in accordance to National Institute for Occupational Safety and Health (NIOSH) Analytical Method 7500 [1] or equivalent. A unit of SRM 1878b consists of approximately 5 g of powder bottled in an argon atmosphere.

Material Description: The SRM material was prepared from single crystal nodules of Brazilian quartz that, with comminution, were reduced to a respirable powder with a median particle size of 3.3 µm. The powder was treated with hydrofluoric and hydrochloric acids to reduce phase and elemental contamination. An analysis of the quantitative results from Rietveld analyses of X-ray powder diffraction data indicated that the SRM material was homogeneous with respect to diffraction properties.

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. The measurands are the certified values for the crystalline phase purity of the material (α-quartz) and the lattice parameters are provided in Table 1. Lattice parameters were recertified in 2017. Metrological traceability is to the SI units for the derived unit of mass fraction (expressed as milligrams per kilogram), and for length (expressed as nanometers); for crystalline phase purity and lattice parameters, respectively. The certified values and uncertainties were calculated according to the method described in the ISO/JCGM Guide [2].

Information Values: An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to adequately assess the uncertainty associated with the value, or it is a value derived from a limited number of analyses. Information values cannot be used to establish metrological traceability. The information values for the particle size distribution, as determined by laser scattering, are given in Figure 1. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1878b** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see “Instructions for Storage and Use”). Periodic 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 changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall coordination and technical direction of the certification were performed by J.P. Cline of the NIST Materials Measurement Science Division.

Material preparation, measurements, and data analysis leading to the certification of this SRM were provided by J.P. Cline, D. Black, M.H. Mendenhall, and A. Henins of the NIST Materials Measurement Science Division.

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Steven J. Choquette, Director
Office of Reference Materials

P.S. Whitfield of Oak Ridge National Laboratory contributed to the analysis of the neutron data.

A portion of this research used resources at the High Flux Isotope Reactor and Spallation Neutron Source, a Department of Energy (DOE) Office of Science User Facility operated by the Oak Ridge National Laboratory.

Statistical analysis was performed by J.J. Filliben of the NIST Statistical Engineering Division.

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

INSTRUCTIONS FOR STORAGE AND USE

Storage: SRM 1878b was bottled in an argon atmosphere to protect against humidity. NIST has not performed any studies concerning the possible degradation of the diffraction properties of quartz, SRM 1878b, as a function of a long-term exposure to humidity. Nor are we aware of any published work concerning this issue. However, SRM 1878b is a high surface area powder; some reactivity to moisture, if only physisorption, is possible. It is recommended that the unused portion of this powder be stored in the original bottle, tightly capped in a dry atmosphere.

Use: The powder of SRM 1878b contains soft agglomerates that can be readily broken up with a kneading operation using a mortar and pestle.

SOURCE, PREPARATION, AND ANALYSIS(1)

Materials: The feedstock for SRM 1878b was prepared through a collaborative effort between NIST; NIOSH, Cincinnati, OH; and the US Geological Survey (USGS, Denver, CO). The starting material consisted of single crystal nodules of Brazilian quartz obtained from Top Gem Minerals (Tucson, AZ). Preliminary processing and comminution were performed by the USGS. After cleaning to remove surface contamination, the nodules were then processed with a jaw crusher to realize a particle size of approximately 1 cm. Further comminution was accomplished with a ceramic lined ball mill using 2.5 cm corundum grinding balls which reduced the median size to approximately 1 mm. The material was then transferred to NIST whereupon it was jet milled to a median particle size of 3.3 μm by Hosokawa Micron Powder Systems (Summit, NJ). The disordered, amorphous surface region of the powder was preferentially dissolved with a wash in hydrofluoric acid. Additional contaminants were removed with a second wash in hydrochloric acid. The powder was then rinsed several times and ignited at 500 °C. These treatments were performed by MV Laboratories, Inc. (Frenchtown, NJ).

Phase Purity: A long-count-time X-ray powder diffraction pattern of SRM 1878b will offer data consistent with a high-purity α-quartz powder. However, the surface region of any crystalline material will not diffract as the bulk due to relaxation of the crystal structure and inclusion of surface reaction products. While this disordered, amorphous surface layer may only be on the order of a few crystallographic units in thickness, in a finely divided solid it can easily account for several percent of the total mass. Phase purity as discussed herein is a microstructural characteristic innate to a finely divided crystalline solid and influenced by the production history of the quartz powder used as the feedstock.

Certification Method: The certified measurement values of SRM 1878b include the crystalline phase purity and the lattice parameters. Ancillary data include the particle size distribution determined via laser scattering and microstructural information determined from the X-ray experiments. The data that led to the certification of phase purity consisted of neutron time-of-flight (TOF) and constant wavelength (CW) powder diffraction data obtained at ORNL. TOF data were collected on the POWGEN beamline at the Spallation Neutron Source [3]. CW neutron data were collected on the HB2a High Resolution Neutron Powder Diffractometer housed at the High Flux Isotope Reactor [4]. SRM 676a Alumina Powder for Quantitative Analysis by X-Ray Diffraction, which was certified with respect to amorphous content [5,6], was used as the internal standard in all diffraction experiments. The phase purity of SRM 1878b was certified through an analysis of the discrepancy between the mass fractions of quartz and alumina determined from Quantitative Rietveld Analysis (QRA) [7], relative to those of the weighing operation. QRA yields only the mass fractions of the crystalline materials; whereas the weighing operation includes both the crystalline and amorphous components. Neutron data are considered to be essentially free of a systematic bias in phase quantification that is often observed in the analysis laboratory X-ray powder diffraction data.

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(1) Certain commercial instruments, materials, or processes are identified in this certificate to adequately specify 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 instruments, materials, or processes identified are necessarily the best available for the purpose.

Laboratory XRD data were collected on a NIST-built diffractometer configured with a Johansson Ge [111] incident beam monochromator, sample spinner and scintillation detector, and, in 2017, a linear Si-strip position sensitive detector (PSD) [8]. Data analyses were performed with the fundamental parameters approach (FPA) [9] for line profile modeling in conjunction with the Pawley [10] and Rietveld methods for analysis of lattice and structural parameters. The homogeneity of SRM 1878b was verified with an analysis of the mass fractions of quartz versus alumina and the lattice parameters of in specimens that consisted of 50-50 mixtures of SRMs 1878b and 676a. The linkage of the certified lattice parameter values to the fundamental unit for length, as defined by the International System of Units (SI) [11], was established with use of the emission spectrum of Cu Kα radiation as the basis for constructing the diffraction profiles. With the use of the FPA, diffraction profiles are modeled as a convolution of functions that describe the wavelength spectrum, the contributions from the diffraction geometry, and the sample contributions resulting from microstructural features. Analysis of data from a divergent-beam instrument requires knowledge of both the diffraction angle and the effective source sample detector distance. Two additional models are therefore included in the FPA analyses to account for the factors that affect the sample height and attenuation. Certification data were analyzed in the context of both Type A uncertainties, assigned by statistical analysis, and Type B uncertainties, based on knowledge of the nature of errors in the measurements, to result in the establishment of robust uncertainties for the certified values.

Certification Procedure: The certification procedure for SRM 1878b is examined in Black *et al.* [12]. Ten bottles of SRMs 1878b and 676a (internal standard) were removed from their respective populations in accordance to a stratified random protocol. All samples consisted of 50:50 mixes of SRMs 1878b and 676a. Five samples were prepared for neutron diffraction analysis with each sample consisting of four grams of material, 1 g from each of two randomly selected bottles of SRMs 1878b and 676a. For X-ray powder diffraction analyses, two specimens were prepared from each bottle of SRM 1878b; the mass of these specimens was 1 g. Both the order in which the specimens were prepared and the bottle of SRM 676a used were randomized. All specimens were homogenized with a kneading operation with a mortar and pestle. Data for certification of lattice parameters were collected from 5 randomly selected phase pure specimens.

With the collection of TOF neutron diffraction data at POWGEN, approximately 3 g of sample were loaded in 8 mm diameter vanadium cans for data collection with center wavelengths of 0.1066 nm and 0.2655 nm at 300 K. This resulted in diffraction patterns with d-spacing spans from 0.03 nm to 0.62 nm. The data were collected for 3 beam hours at an accelerator power of 850 kW. With the CW neutron data, samples were contained in 6.0 mm diameter by 50 mm long vanadium cans during the measurement. Data were collected for 2 h at a wavelength of 0.15366 nm by the [115] reflection from a vertically focused Ge monochromator with a collimation of 0.2°, 0.35°, and 0.2°, before the monochromator, sample and detectors, respectively. The d-spacing range of these data was from 0.05 nm to 0.48 nm. The run order was randomized on an informal basis.

The neutron diffraction data were analyzed with a QRA using the software General Structure Analysis System (GSAS) [13]. This was done with two global refinements of the 5 data sets; one refinement for each diffraction method. The crystal structure for low quartz as reported by Le Page and Donney [14] was used in these analyses. The refined parameters common to both analyses included: scale factors, lattice parameters of the SRM 1878b, atomic positional and thermal parameters. With respect the analysis of TOF data, calibration runs on POWGEN using SRM 660b Lanthanum Hexaboride Powder Line Position and Line Shape Standard for Powder Diffraction [14,15] were used to determine values for the parameters of the GSAS TOF profile function –3 [16], and values for the diffractometer constants DIFC, DIFA and zero. With the analysis of the SRM 1878b / SRM 676a mixtures, only terms pertaining to Lorentzian size broadening were refined. They were constrained with respect to histogram and phase. Given that the lattice parameters of the alumina of SRM 676a were fixed at certified values; the diffractometer constants DIFA and zero were refined. The TOF refinement included four terms of a shifted Chebyshev background function. The CW data were analyzed using the GSAS profile function type 3 [17]. Refined terms included GU, GV, GW, LZ, LY and SL; all but LX and LY were constrained globally; the LX and LY terms were constrained by phase. One of the two parameters of the Finger model [18] for axial divergence, SL, was refined, the other, HL, was fixed at a value nominally identical to SL. Also, given that the lattice parameters of the SRM 676a phase were again fixed, the wavelength and zero values were refined. The CW refinement included five terms of a shifted Chebyshev background function.

The crystalline phase content was determined from the mass fractions determined from the diffraction experiment relative to those of the weighing operation, with the latter ratio being corrected for the known crystalline phase purity of SRM 676a. Considering the results from the two data collections methods independently, the mean for the results from the TOF experiments was 96.53 % crystalline quartz, while the mean for the CW experiments is 96.93 %. The difference between these two means being statistically significant; they were combined with a “Mean of Means” method to yield the certified value and $k = 2$ expanded uncertainty for the certified crystalline phase purity.

With the collection of X-ray powder diffraction data, the 1.5 kW copper tube of fine focus geometry was operated at a power of 1.2 kW. The variable divergence incident slit was also set to 0.9°. With the use of the scintillation detector, data were collected with a 0.2 mm (0.05°) receiving slit, a step width of 0.01° 2θ and a count time of 5 s. With the use of the PSD, a 1.5° Soller slit was located in front of the its window to limit axial divergence, no Soller slits were used in the incident beam either configuration. The PSD was scanned using a variable window length and a combination of coarse and fine steps in $\theta/2\theta$ was used that allowed for data to be collected at high resolution in a timely manner [19]. Samples were spun at 0.5 Hz during data collection. The machine was equipped with an automated anti-scatter slit that blocked air scatter from the incident beam from entering the PSD, where it would otherwise contribute to the low angle background level. The machine was located within a temperature-controlled laboratory space where the nominal short-range control of temperature was ± 0.1 °K. The temperature was monitored using two 10 kΩ thermistors with a Hart/Fluke BlackStack system that was calibrated at the NIST temperature calibration facility [20] to ± 0.002 °C. The source equilibrated at operating conditions for at least an hour prior to recording any certification data. The performance of the machine was qualified with the use of SRMs 660b and 676a using procedures discussed by Cline *et al.* [8]

The certification data were analyzed using the FPA method with a QRA as implemented in TOPAS [21]. Mendenhall *et al.* [22] verified that TOPAS operated in accordance with published models for the FPA. The analysis used energies of the Cu Kα_1 emission spectrum as characterized by G. Hölzer *et al.* [23]. The refined parameters included the scale factors, Chebyshev polynomial terms for modeling of the background, the lattice parameters, specimen displacement and attenuation terms, structural parameters (with the Rietveld analyses), terms for Lorentzian size and strain broadening, a Gaussian strain profile was also refined for the quartz of SRM 1878b. A discussion of the incident spectrum from the IBM and the approach used in its modeling are discussed in Cline *et al.* [8]. High-count-time data were collected from SRM 660b using a relatively small divergence slit angle of 0.5°. The incident spectrum was then modeled with a refinement of the breadths and intensities of three Gaussian profiles at the Kα_{11} location, as defined by Hölzer, and a fourth one located at the Kα_{12} location. Additional refined parameters included the Soller slit angles with the "full" axial divergence model [24]. This analysis provided parameters describing the incident beam spectrum and Soller slit angles characterizing the IPF of the instrument utilizing the IBM and PSD. A second FPA analyses of SRM 660b was performed, as part of the calibration of the instrument [8] provided values for with the incident slit size and were used for evaluation of other parameters pertinent only to the IPF such as the "receiving slit size"; with the use of the PSD this is actually the equatorial width of the silicon strips. With analyses of SRM 1878b, these IPF specific values were fixed at those obtained from the refinements of SRM 660b. The refined lattice parameters were adjusted using the coefficient of thermal expansion values found in Kosinski *et al.* [25] to values in correspondence with 22.5 °C.

The measurand is the certified value for lattice parameters obtained with TOPAS via the Pawley method is provided in Table 1. Metrological traceability is to the SI unit for lattice parameters. The statistical, Type A, evaluation of the lattice parameters resulted in estimates of the lattice parameters of a = 0.491 377 56 nm and c = 0.540 535 84 nm with $k = 2$ expanded uncertainties of 0.000 001 67 nm and 0.000 000 67 nm for a and c, respectively. However, the components of uncertainty that were evaluated by Type B methods must also be taken into account, and these are roughly one order of magnitude larger than those that were evaluated using statistical methods. Data were considered primarily in the context of the uniformity in lattice parameter as a function of 2θ angle; this, in turn, would reflect the functionality of the FPA model [26]. This approach was applied to data from SRM 660b used to calibrate the machine, and both SRMs 676a and 1878b that were contained in the samples. These consideration lead to an assignment of a Type A + B uncertainty of 0.000 030 0 nm to the a and c lattice parameters.

Table 1. Certified Values for SRM 1878b

	Mass Fraction
Crystalline phase purity (α-quartz)	96.73 % ± 0.40 %

	Lattice Parameter (nm)
a	0.491 378 ± 0.000 030
c	0.540 536 ± 0.000 030

Information Values: The FPA experiments included an analysis of crystallite size using the Scardi and Leoni formalism [27] for a log-normal size distribution of spherical crystallites. Owing to the lack of said broadening observed with SRM 1878b on laboratory equipment, these refinements yielded non-physical results for crystallite size. While SRM 1878b does display a slight amount of broadening varying as $1/\cos\theta$, it is insufficient for a valid determination of crystallite size. The FWHM terms varying as tan θ, interpreted as microstrain, refined to exceedingly small values. The refined structural parameters obtained from the Rietveld analyses of SRM 1879b, from both the X-ray and neutron data, did not differ substantially from those reported by Le Page and Donnay [14]. The information values for the particle size distribution, as determined by laser scattering, are given in Figure 1.

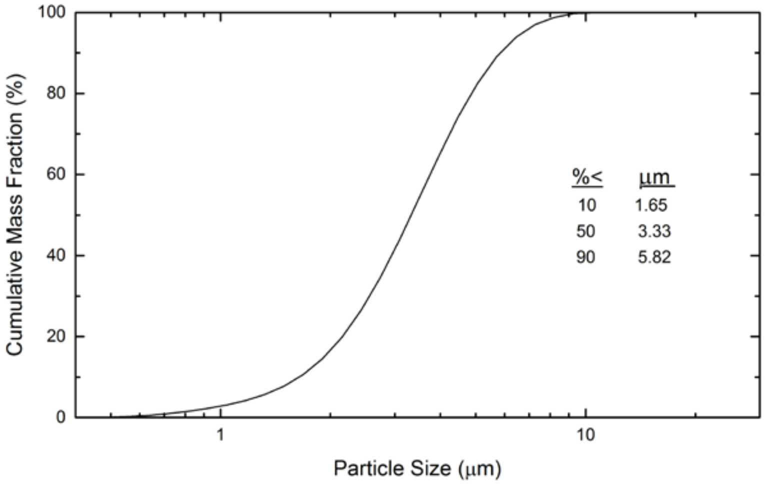

Figure 1. Typical Particle Size Distribution by Laser Scattering

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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) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.