# National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material® 676a

### Alumina Powder
### (Quantitative Analysis Powder Diffraction Standard)

This Standard Reference Material (SRM) consists of an alumina powder (corundum structure) intended primarily for use as an internal standard for quantitative phase analysis using powder diffraction methods. It is also suitable for determination of $I/I_c$ values [1] (for a complete discussion of the $I/I_c$ see [2]). A unit of SRM 676a consists of approximately 20 g of powder, bottled in an argon atmosphere.

**Material Description:** The alumina powder was produced via the alum [$NH_4Al(SO_4)_2 \bullet 12H_2O$] precursor route, calcined to 1400 °C, and jet milled to a fully disaggregated state. The alumina grains are sub-micrometer in size and equi-axial in shape. The high calcination temperature ensures high phase purity while the isometric form of the grains effectively eliminates preferred orientation effects in this powder. The disaggregated state of this material ensures the homogeneity of mixtures prepared by conventional methods.

An analysis of the phase fractions determined from X-ray powder diffraction data from mixtures of SRM 676a and silicon powder, SRM 640c [3], 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 certified phase purity of the material expressed as a mass fraction is:

Crystalline Alumina: 99.02 % ± 1.11 %

The interval defined by the certified value and its uncertainty represents an expanded uncertainty using $k = 2$, in the absence of systematic error [4,5]. The certified value of the phase purity may not exceed 100 %, even though the uncertainty error bounds define a range in excess of 100 %. The measurand is the certified value for the phase purity of the material (alumina). Metrological traceability is to the SI unit for the derived unit of mass fraction (expressed as a percent). The measurands are the certified lattice parameters listed in Table 1. Metrological traceability is to the SI unit of length (expressed as nanometers).

**Information Values:** The analyses associated with certification of SRM 676a included the computation of the diffraction line positions shown in Table 2. Measured relative intensity values are shown in Table 3. These data are presented as information values. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. Information values cannot be used to establish metrological traceability.

**Expiration of Certification:** The certification of **SRM 676a** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is stored and handled in accordance with instructions given in this certificate (See "Instructions for Storage"). Accordingly, periodic recalibration or recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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

John A. Small, Chief
Materials Measurement Science Division

Gaithersburg, MD 20899
Certificate Issue Date: 04 November 2015

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

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The material preparation, measurements, and data analysis were provided by J.P. Cline, D. Black, D. Windover, and A. Henins of the NIST Materials Measurement Science Division; R.B. Von Dreele of Argonne National Laboratory, Argonne, IL; R. Winburn of Minot State University, Minot, ND; P.W. Stephens of the State University of New York, Stony Brook, NY, and the National Synchrotron Light Source, Brookhaven, NY.

Statistical analysis was provided by J.J. Filliben and A.M. Possolo of the NIST Statistical Engineering Division.

J. Evans of Durham University, Durham, UK developed a template for the input files used in data analysis procedures for certification of lattice parameters.

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

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

## INSTRUCTIONS FOR STORAGE

**Storage:** SRM 676a was bottled in an argon atmosphere. While no long-term stability studies have been performed, no degradation of the diffraction properties of this SRM have been observed when stored and used under laboratory conditions. Furthermore, alumina is known to be a highly stable oxide and is expected to remain stable after exposure to atmospheric conditions. However, the unused portion of this SRM powder should be stored, tightly capped, in the original bottle or in one of analogous integrity.

## SOURCE, PREPARATION, AND ANALYSIS(1)

**Materials:** The material used for this SRM was obtained from Baikowski International Corporation (Charlotte, NC).

**Phase Purity:** A long-count-time X-ray powder diffraction pattern of SRM 676a will offer data consistent with a high-purity alumina 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 alumina powder used as the feedstock.

**Certification Method:** The certified measurement values of SRM 676a include the crystalline phase purity and the lattice parameters. The data that led to the certification of phase purity consisted of powder diffraction measurements performed on neutron time-of-flight (TOF) and synchrotron radiation equipment. The lattice parameters were certified with data from a NIST-built X-ray diffractometer [6] located in the temperature-controlled environment of the NIST Advanced Measurement Laboratory (AML).

The phase purity was certified through an analysis of the discrepancy between the results of powder diffraction experiments, which measure the mass ratio of material exhibiting Bragg diffraction, relative to weighing operations, which include all components. The procedure involved a comparison of the phase abundance measured from a series of mixtures of SRM 676a and hyper-pure silicon powder. The experimental design included the assumptions that (1) the silicon consists of single crystal particles, (2) the non-Bragg-diffracting material associated with the silicon was confined to the crystallite surface, and (3) the amorphous layer thickness was invariant with respect to crystallite size. Systematic variation in the amorphous content of the silicon was then effected within the aforementioned series of mixtures by the selection/variation of the particle size (hence specific surface area) of the silicon powder. The effects of extinction in the silicon, which lead to distortions in observed diffraction intensity, were addressed with use of the neutron TOF and synchrotron X-ray powder diffraction at energies of 25 keV and 67 keV, in conjunction with the Rietveld data analysis method [7]. For a complete discussion of the Rietveld method see references 8 and 9. The mass fractions of crystalline silicon, determined from the Rietveld refinements, were plotted relative to the surface areas of the silicon of each sample. An extrapolation of these data to a hypothetical (and physically impossible) silicon sample that would have no specific surface area and, therefore, no amorphous content yielded the mass fraction of SRM 676a that exhibited Bragg diffraction.

(1) Certain commercial equipment, instruments, or materials 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 materials or equipment identified are necessarily the best available for the purpose.

The linkage of the certified lattice parameter values to the fundamental unit of length, as defined by the International System of Units (SI) [10], was established with use of the emission spectrum of Cu Kα radiation as the basis for constructing the diffraction profiles. Data were analyzed using a fundamental parameters approach (FPA) [11] wherein 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. A rigorous 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 must therefore be included in the FPA analyses to account for the factors that affect the distances critical in the use of this geometry. 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 procedure by which SRM 676a was certified with respect to phase purity is examined in Cline, *et al.* [12]. Additional information concerning the characteristics and use of SRM 676a, quantitative phase analysis, and the issue of amorphous content can also be found in this reference.

Data for the certification of lattice parameters were collected using a 2.2 kW copper X-ray tube of long fine-focus geometry operated at a power of 1.8 kW. The source size was approximately 12 mm × 0.04 mm. Axial divergence of the incident beam was limited by a 2.2° Soller slit. Scattered X-rays were filtered with a graphite post-sample monochromator, and detected with a scintillation detector. The variable divergence slit was set nominally to 0.8°. Also, a 2 mm anti-scatter slit was placed approximately 113 mm in front of the 0.2 mm (0.05°) receiving slit. The goniometer radius was 217.5 mm. Samples were spun at 0.5 Hz during data collection. The machine was located within a temperature-controlled laboratory space where the nominal short-range control of temperature was ± 0.1 K. The instrument was controlled via LabVIEW software. Data were recorded in true x-y format. The performance of the machine was qualified with the use of NIST SRM 660b Line Position and Line Shape Standard for Powder Diffraction [13,14] and SRM 676a Alumina Powder for Quantitative Analysis by X-Ray Diffraction using procedures discussed by Cline, *et al.* [6]. (Note: Use of SRM 676a, as referenced in Cline, *et al.* [6], takes advantage of the non-orientating nature of this alumina powder; it does not employ its certified values.)

Ten samples of SRM 676a were selected for certification measurements in a stratified random manner. The data were collected from 10 selected regions; run-time parameters for each region were adjusted with regards to observed full-width at half-maximum (FWHM) and diffraction intensity to optimize data quality per unit time. The scanned regions accessed all but two of the reflections with a relative intensity greater than 5 % and within the 2θ range of 20° to 154°. The angular widths of the scan ranges were 20 to 30 times the observed FWHM values of the profiles and were chosen to provide at least 0.3° 2θ of apparent background straddling each peak. The step width was chosen to include at least eight data points above the FWHM. The count time spent on each profile was inversely proportional to the observed diffraction intensity to realize constant counting statistics amongst the profiles. The total collection time for each sample was about 24 hours.

The certification data for lattice parameters were analyzed using the FPA method with a Rietveld refinement as implemented in TOPAS [15]. The analysis used the Cu Kα emission spectrum, including a satellite component, as characterized by G. Hölzer *et al.* and M. Maskil *et al.* [16,17]. Hölzer models the Cu $K\alpha_1/K\alpha_2$ doublet using four Lorentzian profiles, two primary ones, $K\alpha_{11}$ and $K\alpha_{21}$, and two secondary ones, $K\alpha_{12}$ and $K\alpha_{22}$; the latter two are of reduced intensity and only serve to account for the asymmetry, towards high energy, observed in the spectrum. During calibration of the instrument using high-quality data from SRM 660b, the four Lorentzian breadths of the Cu emission spectrum were refined with constraints to preserve the asymmetric profile shape as modeled by Hölzer. This analysis accounted for the reduction in the FWHM values of the emission spectrum due to the non-uniform band-pass of the of graphite monochromator. The wavelengths and intensities of the $K\alpha_2$ lines were also refined, again with the values of the $K\alpha_{21}$ and $K\alpha_{22}$ lines constrained to one another to preserve the asymmetric shape as modeled by Hölzer. Once this analysis had quantified the impact of the monochromator and yielded an appropriate set of breadths, they were fixed for the subsequent analyses of SRM 676a. The wavelengths and intensities of the $K\alpha_2$ and satellite lines were refined, with constraints applied to the $K\alpha_2$ lines as aforementioned. The other refined parameters included scale factors, second-order Chebyshev polynomial terms for modeling of the background, the lattice parameters, terms indicating the position and intensity of the "tube tails" [18], a Soller slit value in the "full" axial divergence model [19,20] (the axial divergence of the incident and diffracted beams was constrained to be identical), specimen displacement, an attenuation term, structural parameters, size-strain and micro-strain broadening terms of a Lorentzian profile, and a micro-strain broadening term of a Gaussian profile. The very slight level of texture was modeled with a 6th order spherical harmonic.

Examination of the fit to the individual profiles revealed a discrepancy between the model and the observations in the low-angle region. It is well known that low-angle profiles are more prone to error than high-angle lines as the optical aberrations affecting their position are more complex. Also, the reported lattice parameter is more strongly affected

by angular errors in the low-angle region. The (012) line was not used in obtaining the certified parameters. The thermal expansion of alumina as reported by Shvyd'ko *et al.* [21] was used to adjust the lattice parameter to 22.5 °C. A statistical model (bivariate Gaussian random effects model [22]) was used to evaluate the components of uncertainty that reflect differences between samples of the material, and the dispersion of the replicated values measured for each sample. The resulting estimates of the lattice parameters are a = 0.475 935 53 nm and c = 1.299 231 1 nm. The corresponding Type A evaluation of these components of measurement uncertainty was done in conformity with the GUM Supplement 2 [23], and yielded $k = 2$ expanded uncertainties of 0.000 000 61 nm and 0.000 002 7 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 (Type A). Data were considered 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. This consideration leads to an assignment of Type B uncertainties of 0.000 008 0 nm and 0.000 015 nm for a and c, respectively. The certified lattice parameters and their expanded uncertainties, Type A compounded in quadrature with Type B, are shown in Table 1. Peak positions were computed from the certified lattice parameters for Cu Kα Radiation, λ = 0.154 059 29 nm, and are shown in Table 2.

Table 1. Certified Lattice Parameters of SRM 676a

| | Lattice Parameter (nm) | Expanded Uncertainty ($k = 2$) |
|---|---|---|
| a | 0.475 935 5 | ± 0.000 008 0 |
| c | 1.299 231 | ± 0.000 015 |

Data for relative intensity determinations were collected from 10 randomly selected specimens on a Siemens D500 diffractometer. This machine was equipped with a focusing Ge incident beam monochromator, sample spinner/changer, and a quartz-wire position-sensitive proportional detector (PSD). The divergence slit was of 0.67° while the receiving angle of the PSD was nominally 4.5°. The PSD was also fitted with a Soller slit of 2°. Calibration of the equipment was performed using SRM 660a [24] and SRM 676 [25] using methods outlined in Cline, *et al.* [6]. Data were collected from 20° to 154° 2θ with a step width of 0.01° and a scan rate of 1° per minute. Data were analyzed with two methods using two software packages, though the results from only one are reported. The first procedure was to fit the profiles using the split Pearson VII profile shape function (PSF) as implemented within TOPAS. The second involved Rietveld analyses via GSAS [26]. The background in both analyses was represented by a tenth-order Chebyshev polynomial with a $1/x$ term. The refined parameters of the Rietveld analyses included the scale factors, Gaussian and Lorentzian crystallite size, and strain broadening as represented by the GP, LX, and LY terms of the Thompson-Cox-Hastings "Type 3" profile shape model [27], the "Type 1" polarization factor, sample shift and transparency terms, and structural parameters. Profile terms GU, GV, GW, and the peak asymmetry parameters of the Finger model [28] were fixed at values obtained from an analysis of SRM 660a. Relative intensity data were extracted with the GSAS utility REFLIST, which uses the observed structure factors, corrected for multiplicity and Lorentz-polarization factor, to compute relative intensity values. The observed structure factors are determined from a background-subtracted summation of the counts in the peak region of the raw data. The Rietveld analyses served to fit the background, determine the peak cutoff angles, and the ratio of the intensity distributed between overlapping lines. Relative intensity data correlated to better than 1 % between the two methods, which served to validate the results. Data are reported from the Rietveld analyses as these are judged more accurate because no PSF is used. The relative intensities of SRM 676a and their expanded uncertainties, using the $k = 2$ factor, are shown in Table 3.

Table 2. Peak Position Information Values for SRM 676a,
Lines Listed with a Relative Intensity >5 %, Computed Using Cu Kα Radiation, λ = 0.154 059 29 nm

| Reflection Indices, h k l | Peak Position (2θ, degrees) | Reflection Indices, h k l | Peak Position (2θ, degrees) |
|---|---|---|---|
| 0 1 2 | 25.574 | 3 2 4 | 116.085 |
| 1 0 4 | 35.149 | 0 1 14 | 116.602 |
| 1 1 0 | 37.773 | 4 1 0 | 117.835 |
| 1 1 3 | 43.351 | 4 1 3 | 122.019 |
| 0 2 4 | 52.548 | 1 3 10 | 127.671 |
| 1 1 6 | 57.497 | 3 0 12 | 129.870 |
| 2 1 4 | 66.513 | 2 0 14 | 131.098 |
| 3 0 0 | 68.203 | 1 4 6 | 136.056 |
| 1 0 10 | 76.873 | 1 1 15 | 142.314 |
| 1 1 9 | 77.233 | 4 0 10 | 145.153 |
| 2 2 3 | 84.348 | 0 5 4 | 149.185 |
| 0 2 10 | 88.994 | 1 2 14 | 150.102 |
| 1 3 4 | 91.179 | 1 0 16 | 150.413 |
| 2 2 6 | 95.240 | 3 3 0 | 152.380 |
| 2 1 10 | 101.070 | | |

Table 3. Information Values for Relative Intensity Data from SRM 676a

| Reflection Indices, h k l | Relative Intensity | Expanded Uncertainty ($k$ = 2) |
|---|---|---|
| 0 1 2 | 57.06 | ± 0.122 |
| 1 0 4 | 88.41 | ± 0.254 |
| 1 1 0 | 37.75 | ± 0.112 |
| 1 1 3 | 100.0 | ------ |
| 0 2 4 | 47.33 | ± 0.075 |
| 1 1 6 | 95.78 | ± 0.250 |
| 2 1 4 | 37.74 | ± 0.101 |
| 3 0 0 | 57.49 | ± 0.157 |

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**Certificate Revision History:** 04 November 2015 (Editorial changes); 23 April 2012 (Lattice parameters reported as certified values; Discussion of lattice value certification procedure included; Reference to phase purity determination included; Editorial changes); 28 January 2008 (Original certificate date).

*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.*

APPENDIX

**Acknowledgements**

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