



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1976b

#### Instrument Response Standard for X-Ray Powder Diffraction

This Standard Reference Material (SRM) consists of a sintered alumina disc intended for use in calibration of X-ray powder diffraction equipment with respect to line position and intensity as a function of  $2\theta$  angle. The solid form of the SRM eliminates any variability in intensity measurements introduced by the sample loading procedure. A unit of SRM 1976b consists of a sintered alumina disc approximately 25.6 mm in diameter by 2.2 mm in thickness.

**Material Description:** The manufacturing process used to produce this SRM was developed for the production of substrates for electronic components. The alumina powder used as the precursor for these substrates was of high phase purity (corundum structure) with a platelet particle morphology. The platelets were typically 5  $\mu\text{m}$  to 10  $\mu\text{m}$  in diameter by 2  $\mu\text{m}$  to 3  $\mu\text{m}$  in thickness. The compaction procedure for the discs resulted in an axisymmetric texture with the basal planes tending towards parallelism with the surface of the disc. This axisymmetric character of the texture permits sample mounting in any orientation about the surface normal. The compacts were liquid-phase sintered using a small percentage of a glass phase. No crystalline impurities have been detected. The glass phase involved in the liquid-phase sintering effectively prevents inter-particle contact and relaxes during the cooling of the pieces from the sintering temperature. This leads to a minimal development of micro-strain and its associated line broadening; though it is detectable as Gaussian broadening with a  $\tan\theta$  dependence. Given this, and the essential absence of crystallite size broadening, SRM 1976b can be used to obtain an approximation of the instrument profile function (IPF). Use of SRM 1976b is not recommended, however, for quantitative microstructure analyses. The discs comprising the feedstock of this SRM were manufactured in a single, dedicated production run to ensure consistency of microstructure with respect to grain size, shape, micro-strain, and texture.

**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 relative intensity (dimensionless ratios) shown in Table 1, and the lattice parameters are shown in Table 2. Metrological traceability is to the SI units for the relative intensity values are the dimension of one, and for lattice parameters the dimension is length (expressed as nanometers). The certified values and uncertainties were calculated according to the method described in the ISO/JCGM Guide [1].

**Information Values:** The analyses associated with certification of SRM 1976b included the computation of the diffraction line positions shown in Table 3. In order to use SRM 1976b on diffraction equipment of various optical configurations, the effects of polarization must be taken into account. The values shown in Table 4 reflect a bias applied to the certified values to account for this effect. The data of Tables 3 and 4 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 1976b** 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.

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

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Gaithersburg, MD 20899  
Certificate Issue Date: 04 November 2015  
*Certificate Revision History on Last Page*

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

The preparation, measurements and data analyses were performed by J.P. Cline, D. Black, and D. Windover of the NIST Materials Measurement Science Division and A. Henins of the NIST Quantum Measurement Division.

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

Statistical analysis was provided 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.

**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 AND USE

**Storage:** SRM 1976b consists of a sintered alumina substrate that is essentially identical to NIST SRMs that have been available since 1991. While no long-term stability studies have been performed, we have no evidence of any degradation of the diffraction properties of this SRM when used under laboratory conditions. Furthermore, alumina is known to be a highly stable oxide. Contamination of the surface with other crystalline materials may result in impurity lines in the data. Discoloration may occur from exposure to X-rays; this does not affect the certification.

**Use (Mounting of SRM 1976b):** The disc format of the SRM was chosen to be amenable to many sample holder geometries. Diffraction data should only be collected from the side opposite the label. The stamping operation used to make the disc during manufacture resulted in the edge of the disc surface being depressed by approximately 10  $\mu\text{m}$  relative to the center. While this is not regarded as a significant difficulty due to the low attenuation of X-rays by alumina, height justification during mounting should be with respect to the center of the disc (see also "Use of SRM 1976b for Testing of Instrument Sensitivity").

## SOURCE, PREPARATION, AND ANALYSIS<sup>(1)</sup>

**Materials:** The alumina discs used for this SRM were manufactured by International Business Machines Corporation, East Fishkill, NY.

**Certification Method:** Certification was performed using data from a NIST-built diffractometer [2] that includes several advanced design features, one of which is the convertibility between any of a number of optical configurations. Two configurations were used in the certification of SRM 1976b: 1) the lattice parameters were certified using data from conventional, slit-based incident optics and receiving optics that included a graphite monochromator located between the sample and detector, a post monochromator, and 2) the data for the certified relative intensities were collected with the machine configured with a Johansson incident beam monochromator (IBM). In both cases, the diffraction geometry was that of a divergent-beam X-ray diffractometer of Bragg-Brentano geometry.

For certification of the lattice parameters, the linkage to the fundamental unit for length as defined by the International System of Units (SI) [3] is established through the use of the emission spectrum of Cu K $\alpha$  radiation as the basis for constructing the diffraction profiles. Data were analyzed using the fundamental parameters approach (FPA) [4] wherein diffraction profiles are modeled as a convolution of functions that describe the X-ray emission 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. With respect to the certified relative intensity data, the Johansson IBM simplified the IPF such that accurate fits to the profiles could be obtained using analytical profile shape functions (PSF). Several functions were employed using several analysis packages; realization of commonality in results between methods lends credibility to the certification method. 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 and comprehensive uncertainties for the certified values.

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<sup>(1)</sup> 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.

**Certification Procedure:** When the diffractometer was set up with the post monochromator, a 2.2 kW sealed copper tube of long fine-focus geometry was operated at a power of 1.8 kW during certification measurements. 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. In the case of the Johansson IBM, a 1.6 kW copper tube of standard fine-focus geometry was used and operated at a power of 1.2 kW during certification measurements. The source size was approximately 8 mm × 0.04 mm. In this case a 2.2° Soller slit was used in the receiving optics to limit axial divergence. Common to both configurations was the use of a variable divergence slit, which was nominally set 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 source was allowed to equilibrate 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 NIST SRM 660b “Line Position and Line Shape Standard for Powder Diffraction” [5] and SRM 676a “Alumina Powder for Quantitative Analysis by X-Ray Diffraction” [6] using procedures discussed by Cline, *et al.* [2]. The response of the scintillation detector was verified to be linear over the count rate range of the certification measurements. A stepwise experiment was executed utilizing Al foils to generate a plot of Ln (count rate) vs. number of foils; the linearity of said plot verified the proper operation of the detector.

Twenty units of SRM 1976b were selected in a stratified random manner from the population of units being certified. Diffraction data used for the determination of certified relative intensities were collected in a single continuous scan using a 2θ range of 20° to 160°, with a step width of 0.008° and a count time of 4 seconds. This resulted in a total collection time of approximately 24 h per sample. The data collected for the determination of certified lattice parameters were collected from 11 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. Five of the regions contained one profile, and the remainder contained from three to six; this accessed all the reflections with a relative intensity greater than approximately 2.0 % 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 so as to realize constant counting statistics amongst the profiles. The total collection time for each sample was, again, approximately 24 h.

**Data Analysis:** Data for the certification of relative intensities were analyzed with three methods using three software packages; the results from one were used to develop certified values, while the other two were used for confirmation. Two of the methods involved profile fitting. The first used the split Pearson VII PSF as implemented within TOPAS [7] while the second used the split Voigt PSF as implemented within HighScore Plus [8]. The third used Rietveld [9,10] analyses via GSAS [11] (for a complete discussion of the Rietveld method see reference 12 and 13). The background in all analyses was represented by a tenth order shifted Chebyshev polynomial. With regards to both of the profile fitting analyses, the parameters refined included two shape and FWHM parameters for each split PSF. The low-intensity lines, typically less than 2 %, were fit with symmetric PSFs. The refined parameters of the Rietveld analyses included the scale factors, lattice parameters, sample shift and transparency terms, structural parameters and the “Type 1” Lorentz-polarization factor. Texture effects were modeled using a sixth order spherical harmonic. Profile shape was modeled with a Thompson-Cox-Hastings [14] “Type 3” function using the GU, GW and LY terms to represent instrumental, crystallite size and strain broadening. The Finger model [15] was used to fit profile asymmetry; one of the peak asymmetry parameters, S/L, was refined, while the second, H/L, was fixed such that the two terms were nearly equal.

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. No PSF is used. The Rietveld analyses served only to fit the background, determine the peak cutoff angles, and the ratio of the intensity distributed between overlapping lines. Data are reported from the Rietveld analyses as these are judged more accurate due to the fact that a profile shape model is not used. With use of the split Voigt PSF, deviations are typically negative and less than 1 % for all but the high angle (146) and (4.0.10) lines where it approaches a value 3 % larger than the certified values. In the case of the split Pearson VII PSF, the discrepancies are more systematically negative with the deviations increasing with 2θ, to a maximum value at high angle of about 3 % below the certified values. Nonetheless, the commonality of these data served to validate the results. The relative intensities of SRM 1976b and their expanded uncertainties, using the  $k = 2$  factor, are shown in Table 1.

Table 1. Certified Relative Intensity Data for SRM 1976b

Reflection, (hkl)	Relative Intensity	Expanded Uncertainty ( $k = 2$ )
(012)	23.68	$\pm 0.21$
(104)	100.0 <sup>(a)</sup>	---
(113)	37.33	$\pm 0.25$
(024)	21.05	$\pm 0.20$
(116)	87.79	$\pm 0.49$
(300)	12.63	$\pm 0.20$
(1.0.10) & (119)	71.20	$\pm 0.63$
(0.2.10)	13.27	$\pm 0.12$
(226)	8.17	$\pm 0.07$
(2.1.10)	16.32	$\pm 0.13$
(324) & (0.1.14)	25.46	$\pm 0.28$
(1.3.10)	14.86	$\pm 0.12$
(146)	12.68	$\pm 0.08$
(4.0.10)	11.04	$\pm 0.14$

<sup>(a)</sup> Value not certified.

The certification data for lattice parameters were analyzed using the FPA method with a Rietveld refinement as implemented in TOPAS. The analysis used the Cu  $K\alpha$  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 superlative-quality data from SRM 660b, the four Lorentzian breadths of the Cu emission spectrum were refined with constraints to preserve 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 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 1976b. 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 the 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] as the axial divergence of the incident and diffracted beams was constrained to be identical, specimen displacement, an attenuation term, structural parameters, a crystallite size broadening term of a Lorentzian profile, and a micro-strain broadening term of a Gaussian profile. Texture effects were modeled with a sixth 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, therefore, 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 analysis of the data indicated that the means of the measurements were  $a = 0.475\,913\,67$  nm and  $c = 1.299\,337\,2$  nm with a  $k = 2$  Type A expanded uncertainty of 0.000 000 66 nm and 0.000 001 1 nm for  $a$  and  $c$  respectively. However, a Type B uncertainty due to systematic error must be incorporated into the uncertainty bounds of the certified lattice parameter. Data were considered in the context of the uniformity in lattice parameter as a function of  $2\theta$  angle; this, in turn, would reflect the functionality of the FPA model. This consideration leads to an assignment of a Type B uncertainty that is roughly an order of magnitude larger than the Type A. The certified lattice parameters and their expanded uncertainties, Type A compounded with Type B, are shown in Table 2. Peak positions were computed from the certified lattice parameters for Cu  $K\alpha$  Radiation,  $\lambda = 0.154\,059\,29$  nm, and are shown in Table 3 as information values.

Table 2. Certified Lattice Parameters for SRM 1976b

	Lattice Parameter (nm)	Expanded Uncertainty ( $k = 2$ )
a	0.475 913 7	$\pm 0.000\ 008\ 0$
c	1.299 337	$\pm 0.000\ 015$

Table 3. Peak Position Information Values for SRM 1976b,  
Lines Listed with a Relative Intensity >5 %, Computed Using Cu K $\alpha$  Radiation,  $\lambda = 0.154\ 059\ 29$  nm

Reflection, (hkl)	Peak Position ( $2\theta$ , degrees)
(012)	25.575
(104)	35.147
(110)	37.775
(006)	41.673
(113)	43.351
(024)	52.548
(116)	57.495
(018)	61.297
(214)	66.515
(300)	68.207
(1.0.10)	76.866
(119)	77.229
(0.2.10)	88.989
(0.0.12)	90.699
(226)	95.242
(2.1.10)	101.066
(324)	116.091
(0.1.14)	116.588
(1.3.10)	127.669
(3.0.12)	129.862
(2.0.14)	131.083
(146)	136.063
(1.1.15)	142.292
(4.0.10)	145.152
(1.2.14)	150.081
(1.0.16)	150.380
(330)	152.402

**Use of SRM 1976b for Testing of Instrument Sensitivity:** The relative intensities reported for SRM 1976b are to be used for the basis of a calibration of an instrument for diffraction intensity as a function of  $2\theta$  angle, or instrument sensitivity. The  $k = 2$  expanded uncertainties, shown in Table 1, represent our degree of confidence in the reported relative intensity values; they do not play a role in any judgment concerning the qualification of a test instrument. It is the responsibility of the end user, in conjunction with the instrument vendor, to determine the acceptance criteria for their specific instrument. Such an acceptance criteria may be based on a number of factors concerning random and systematic influences on the measurement method. The “method” in this case refers to the entire suite of hardware, software, and procedures used to obtain the results under consideration. In determination of an acceptance criterion, it may be appropriate to perform an extensive study of the measurement method itself; such a study would have to include the sensitivity of the results on such environmental factors as power fluctuations, laboratory temperature variations, etc.

Accurate integrated intensity values, which can be determined via methods outlined in the certification, should be determined from the test equipment. Complete scans of the entire  $2\theta$  range are required for “whole pattern” methods; limited regions can be used if profile fitting is employed. The breadth of the region to be collected is, of course, dependent on the optics of the specific instrument; it should include at least  $0.3^\circ$  of apparent background on either side of the profile. Use of a sample spinner will improve particle counting statistics. Care should be taken to ensure proper modeling of the background in data analysis procedures. Graphical evaluation of the ratio of these test data to the

certified values vs.  $2\theta$  will allow for an appropriate judgment as to the condition of the test equipment. The desired result would consist of unity values across the two theta range. However, data should be considered as a whole in the context of observed trends; a few outliers do not constitute a failure. Furthermore, as discussed with the certification data, greater deviations can be expected from the high angle lines.

The use of SRM 1976b for the calibration of X-ray diffraction equipment of differing optical configurations may require that a bias be applied to the certified values to render them appropriate for the machine to be qualified. This bias is needed to account for differences in the polarization effects from the presence, absence, and character of crystal monochromators. The polarization factor for a diffractometer that is not equipped with a monochromator is, from Guinier [22]:

$$\frac{1 + \cos^2 2\theta}{2} \quad (1)$$

The polarization factor for a diffractometer equipped with only an incident beam monochromator is, from Azaroff [23]:

$$\frac{1 + \cos^2 2\theta_m \cos^2 2\theta}{1 + \cos^2 2\theta_m} \quad (2)$$

where  $2\theta_m$  is the  $2\theta$  angle of diffraction for the monochromator crystal. The polarization factor for a diffractometer equipped with only a diffracted beam, post monochromator is, from Yao & Jinno [24]:

$$\frac{1 + \cos^2 2\theta_m \cos^2 2\theta}{2} \quad (3)$$

where, again,  $2\theta_m$  is the  $2\theta$  angle of the monochromator crystal. Equations 2 and 3 are considered appropriate when the crystal is of an “ideal mosaic” structure, i.e., the diffracting domains are uniformly small and, therefore, the crystal is diffracting in a kinematic context. This is in contrast to a “perfect” crystal, which would diffract in accordance with dynamical scattering theory. The use of equations 2 and 3 was evaluated with the diffractometer used in the certification of SRM 1976b. With the Johansson IBM, as per Equation 2, a Ge crystal (111) reflection ( $2\theta_m = 27.3$  degrees) was used. With respect to Equation 3, a pyrolytic graphite crystal (0002) basal plane reflection ( $2\theta_m = 26.6$  degrees) as a diffracted beam, post monochromator was used. Rietveld analyses of data from SRMs 660b, 1976b, and 676a that included a refinement of the polarization factor, modeled as per Equations 2 and 3 in TOPAS, indicated that Equations 2 and 3 are appropriate for these crystals and configurations. Given that the certification data of SRM 1976b were collected using a Johansson IBM, the ratio of values computed from Equations 1, 2, and 3 were used to bias the certified values to correspond to those of alternative configurations. The results are shown in Table 4. The user may select the set of relative intensity values from Table 4 that are appropriate for the configuration of the instrument to be qualified and proceed accordingly. Use of SRM 1976b for additional configurations may require computation of biases alternative to those presented herein.

Table 4. Information Values for Biased Relative Intensity Data for SRM 1976b

Reflection, (hkl)	Relative Intensity	
	No Monochromator	Graphite Post Monochromator
(012)	23.95	23.70
(104)	100.0	100.00
(113)	36.87	37.30
(024)	20.43	21.00
(116)	84.37	87.57
(300)	11.86	12.58
(1.0.10) & (119)	65.72	70.68
(0.2.10)	12.15	13.19
(226)	7.49	8.13
(2.1.10)	15.04	16.24
(324) & (0.1.14)	24.14	25.38
(1.3.10)	14.44	14.84
(146)	12.51	12.66
(4.0.10)	11.04	11.04

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<b>Certificate Revision History:</b> 04 November 2015 (Editorial changes); 24 June 2015 (Updated graphite post monochromator values in Table 4; editorial changes); 08 February 2012 (Original certificate date).
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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](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*