National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 674b

X-Ray Powder Diffraction Intensity Set

(Quantitative Powder Diffraction Standard)

This Standard Reference Material (SRM) consists of four oxide powders (ZnO, TiO_2, Cr_2O_3, and CeO_2), intended primarily for use as internal standards for quantitative X-ray diffraction analysis. These four oxides offer a range of linear attenuations for Cu-Kα radiation: 279 cm^{-1} (ZnO, wurtzite structure), 536 cm^{-1} (TiO_2, rutile structure), 912 cm^{-1} (Cr_2O_3, corundum structure), and 2203 cm^{-1} (CeO_2, fluorite structure) that allow the user to nominally match the standard to the unknown in order to minimize the effects of microabsorption. A unit of SRM 674b consists of approximately 10 g of each powder, bottled in an argon atmosphere.

Material Description: The powders consist of fine-grained high-purity equi-axial grains that are not in an aggregated state. The isometric form of the grains effectively eliminates preferred orientation effects in these powders. The de-aggregated state of these materials ensures the homogeneity of test mixtures prepared by conventional methods.

An analysis of the lattice parameters and phase fractions determined from X-ray powder diffraction data collected from mixtures of SRM 674b and SRM 676 [1] 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 four materials and the lattice parameters that are provided in Tables 1 and 3, respectively. 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. Information values for the relative intensity values, I/I_c values [3] (for a complete discussion see reference 4) and microstructural parameters are provided in Tables 4-6. The particle size data, as determined by laser scattering, are given Table 7.

Expiration of Certification: The certification of **SRM 674b** is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Accordingly, periodic recalibration or 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 technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Technical direction and overall coordination on the certification of this SRM were provided by J.P. Cline of the NIST Materials Measurement Science Division.

John A. Small, Chief
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Gaithersburg, MD 20899
Certificate Issue Date: 15 November 2017
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Material preparation, measurements, and data analysis leading to the certification of this SRM were performed by R.S. Winburn of Minot State University (Minot, ND), J.P. Cline, D. Black, M.H. Mendenhall and A. Henins of the NIST Materials Measurement Science Division, and R.B. Von Dreele of Argonne National Laboratory (Argonne, IL).

Statistical analysis was provided by J.J. Filliben and I. Aviles of the NIST Statistical Engineering Division.

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

SOURCE, PREPARATION, AND ANALYSIS[(1)]

Materials: The ZnO and TiO_2 powders of SRM 674b were obtained from Alfa Aesar (Ward Hill, MA) and Cr_2O_3 and CeO_2 powders were obtained from Cerac Inc. (Milwaukee, WI).

Phase Purity: The consideration of a long-count-time X-ray powder diffraction pattern may indicate that the sample is a high-purity powder, i.e., no peaks from impurity phases and a background that is consistent with contributions of air scatter from the incident beam and thermal diffuse scatter from the sample. 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 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. The characterization of "phase purity" or "amorphous content" discussed herein is not in the context of a mechanically separable impurity phase, but it is a microstructural characteristic innate to the chemistry and the production history of the SRM feedstock.

Certification Method: The oxide powders of this SRM were certified with respect to the mass fraction of material that exhibits Bragg scattering in correspondence to their crystal structure, or phase purity, and lattice parameters. The certification procedure utilized Quantitative Rietveld Analyses (QRA) [5] (for a complete discussion of the Rietveld method, see references 6 and 7) of neutron time-of-flight (TOF) diffraction measurements in conjunction with the use of SRM 676 as the internal standard. This procedure referenced the phase purity of SRM 674b against that of SRM 676. The basis of the method rests on an analysis of the discrepancy between the results of powder diffraction experiments, which measure the mass of material exhibiting Bragg diffraction, relative to weighing operations, which include all components.

The QRA of laboratory, divergent beam X-ray powder diffraction (XRPD) data displayed a systematic bias of less than 2 %; however, these results were as precise as those determined from the TOF data. Therefore, the certified phase composition was determined from the TOF data while the homogeneity of the SRM material was verified with Rietveld analyses of XRPD data. It should be noted that the mechanism inducing this bias is not operative in Reference Intensity Ratio (RIR) based methods [8] (for a complete discussion of RIR methods, see reference 4). The emission spectrum of Cu Kα radiation of the XRPD experiment is used as the basis for constructing the diffraction profiles within the fundamental parameters approach (FPA) [9] method for diffraction line profile analysis. The emission spectrum provides the linkage of the certified lattice parameter values to the fundamental unit of length, as defined by the International System of Units (SI) [10]. 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 optics, 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 effect of 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: Ten bottles were selected from the population in accordance to a stratified random protocol. Samples taken from two bottles were combined and admixed with SRM 676 at the 50 % level to yield a total of five samples for TOF neutron diffraction analysis. TOF data were obtained on the High Intensity Powder Diffractometer (HIPD) at the Manuel Lujan, Jr., Neutron Scattering Center (LANSCE) (Los Alamos, NM). The samples were contained in 9.5 mm diameter by 50 mm long vanadium cans during the analysis. Each sample was exposed to the neutron beam for 1.3 h with the LANSCE source operating at 70 µA proton beam current. Data used for this certification were obtained from detector banks positioned at ± 153° 2θ covering a d-space range of 0.05 nm to 0.48 nm. The run order was randomized on an informal basis. Rietveld refinements using the General Structure Analysis System (GSAS) [11] of the phases in these samples included: scale factors, lattice parameters of the SRM 674b materials (those of SRM 676 were fixed at the certified values) a profile shape function term representing Lorentzian peak broadening [12], atomic positional and thermal parameters, a term for the diffractometer (DIFC), an

[(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.

absorption correction term, and six terms of a background function describing the effects of thermal diffuse and incoherent scattering. The amorphous phase content was determined from the mass fraction ratio determined from the diffraction experiment relative to that of the weighing operation, with the latter ratio being corrected for the known phase purity of SRM 676. The certified phase purity of these materials, expressed as a mass fraction is given in Table l.

Table l. Certified Phase Purity Mass Fractions

Crystalline Component	Phase Purity (%)
ZnO	95.28 ± 0.64
TiO_2	89.47 ± 0.62
Cr_2O_3	95.91 ± 0.60
CeO_2	91.36 ± 0.55

The interval defined by the certified value and its uncertainty represents an expanded uncertainty using $k = t$, where t is the appropriate 2-sided 95 % confidence interval coefficient, in the absence of systematic error. The uncertainty reported does not include that of the phase purity determination of the standard used for this certification, SRM 676.

Two X-ray diffractometers were used for collection of certification data. The first was a Siemens D5000 diffractometer equipped with a sample spinner, graphite post monochromator and scintillation detector. Data from this machine were used to verify the homogeneity of the powders, determine the relative intensities and I/I_c values. The second was a NIST-built machine used for the certification of lattice parameters and determination of microstructural data. A full discussion of this machine can be found in reference 13. It was configured in a manner analogous to the D5000. The instruments were aligned and calibrated using SRMs 660a [14], for the D5000, and 660b [15,16] for the NIST machine, in a manner described in reference 13. The data collected for certification of the lattice parameters were analyzed using the FPA method with Rietveld analyses as implemented in TOPAS [17]. Mendenhall *et al.* [18] verified that TOPAS operated in accordance with published models for the FPA. The analysis used the Cu $K\alpha_1/K\alpha_2$ emission spectrum, including a satellite component, as characterized by G. Hölzer *et al.* and Maskil & Deutsch [19,20]. The intensities and positions of the Cu $K\alpha_2$ line, the satellite line and the "tube tails" [21] were refined. Constraints were applied to positions and intensities of the $K\alpha_{21}$ and $K\alpha_{22}$ lines to preserve the overall shape as per Hölzer. A Soller slit value, constrained to be identical for both the incident and diffracted beam, using the "full" axial divergence model [22], was refined. This procedure allowed for determination of parameters specific to the instrument profile function (IPF) [13] and were fixed in subsequent analyses of SRM 674b materials.

Diffraction data for homogeneity testing were collected on two specimens removed from each of the ten aforementioned bottles. These specimens also had SRM 676 admixed with them in a 50:50 mass ratio. The scan range was from 20° to 155° 2θ with a step width of 0.02° and a count time of 3.5 s/step. The divergence slit width was 0.85°; a 2.3° incident Soller slit and a 0.05° receiving slit were used. These data were analyzed with Rietveld method via GSAS. The refined parameters included the scale factors, a background represented by a fifth order Chebyshev polynomial with a 1/x term, the lattice parameters of the SRM 674b materials, specimen displacement, attenuation, size and strain (when relevant) terms and structural parameters. Additional data were collected from three phase pure specimens of each of the SRM materials for determination of the relative intensity values. Both the I/I_c and relative intensity values were determined by profile fitting of all peaks within the angular range of 20° to 70° 2θ using the FPA method. The refined diffraction intensities used for determination of the I/I_c values were corrected for the known amorphous contents of the two phases, i.e., the material of SRM 674b and the alumina of SRM 676.

Data for certification of lattice parameters and determination of microstructural data were collected from 5 randomly selected bottles of the four SRM 674b materials. The NIST machine utilized essentially the same slit dimensions as were used on the D5000. The data were collected in three 2θ ranges with the step widths and count times optimized for each material and region with respect to profile breadth and diffraction intensity. Total scan time for each sample was approximately 10 hours. The analysis of the IPF included a refinement of the breadths of emission spectrum to account for the effects of the graphite monochromator [13]. The analysis was performed as per the homogeneity testing except that the crystallite size broadening was modeled with a log-normal size distribution of spherical crystallites using the Scardi and Leoni formalism [23]. The average of the five values for μ and σ obtained from use of this model were used to obtain the reported crystallite size data. The analyses of the TiO_2 and Cr_2O_3 materials included Lorentzian breadth term that varied as tan θ that is interpreted as microstrain, to determine the reported ε_0 values, where $(\varepsilon_0)^2$ is the mean squared strain. Again, the reported value is the average of the five obtained from the refinements. The refined lattice parameters were adjusted using the coefficient of thermal expansion values found in Touloukian *et al.* [24] to values in correspondence with 22.5 °C. Statistical analysis provided the Type A uncertainties reported in Table 2. 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 [25]; this, in turn, would reflect the functionality of the FPA model. This approach was applied to data from SRM 660b used to calibrate the machine. This consideration leads to an assignment of Type A + B uncertainty of 0.000 030 0 nm to the a and c lattice parameters; the certified values are shown in Table 3.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Storage: SRM 674b was bottled under an argon atmosphere to protect against humidity. Although there have been no long-term stability studies on this SRM, the oxides of which it is composed are known to be a stable in the ambient conditions of a typical laboratory. It is, therefore, believed that this SRM is stable after exposure to atmosphere. It is, nonetheless, recommended that the unused portion of the powder be stored in a tightly capped container such as the original bottle or in a manner to afford similar or greater protection against humidity.

Information Values and Uncertainties: NIST information values are considered to be of interest to the SRM user, but are not certified because the measurements are not traceable to the SI, or only a limited number of analyses were performed which disallowed imparting plausible uncertainties to the measured values. The informational values determined from the aforementioned analyses of XRPD data are presented in Tables 4 thorough 6. The particle size distributions, determined from a disc centrifuge analyzer, are given in Table 7. The interval defined by a value and its uncertainty is a 95 % confidence interval for the true value of the mean in the absence of systematic error.

Table 2. Lattice Parameters Values and Type A Uncertainties for SRM 674b Powders

Component	Lattice Parameter a (nm)	Uncertainty Type A (k = 2)	Lattice Parameter c (nm)	Uncertainty Type A (k = 2)
ZnO	0.324 986 50	± 0.000 000 48	0.520 673 64	± 0.000 002 92
TiO_2	0.459 394 52	± 0.000 002 30	0.295 890 90	± 0.000 001 23
Cr_2O_3	0.495 858 64	± 0.000 003 02	1.359 650 66	± 0.000 011 92
CeO_2	0.541 152 60	± 0.000 000 30		

Table 3. Certified Lattice Parameters of SRM 674b

Component	Lattice Parameter a (nm)	Uncertainty Type A + B (k = 2)	Lattice Parameter c (nm)	Uncertainty Type A + B (k = 2)
ZnO	0.324 987	± 0.000 030 0	0.520 674	± 0.000 030 0
TiO_2	0.459 395	± 0.000 030 0	0.295 891	± 0.000 030 0
Cr_2O_3	0.495 859	± 0.000 030 0	1.359 651	± 0.000 030 0
CeO_2	0.541 153	± 0.000 030 0		

Table 4. Information I/I_c Values for SRM 674b

Component	I/I_c
ZnO	4.95 ± 0.01
TiO_2	3.44 ± 0.01
Cr_2O_3	1.97 ± 0.02
CeO_2	12.36 ± 0.09

Table 5. Information Relative Intensity Values of SRM 674b

ZnO

h k l	Angle	Rel I (%)
1 0 0	31.76	61.30 ± 2.34
0 0 2	34.41	37.24 ± 2.10
1 0 1	36.25	100.0 ----
1 0 2	47.53	22.29 ± 0.66
1 1 0	56.59	37.72 ± 1.44
1 0 3	62.85	30.28 ± 1.14
2 0 0	66.37	5.25 ± 0.24
1 1 2	67.94	27.13 ± 0.12
2 0 1	69.08	14.12 ± 0.32

TiO_2

h k l	Angle	Rel I (%)
1 1 0	27.45	100.0 ----
1 0 1	36.09	37.70 ± 1.50
2 0 0	39.20	5.96 ± 0.12
1 1 1	41.25	18.70 ± 0.48
2 1 0	44.05	7.46 ± 0.25
2 1 1	54.32	55.14 ± 1.58
2 2 0	56.63	17.48 ± 0.31
0 0 2	62.76	6.94 ± 0.40
3 1 0	64.05	8.03 ± 0.21
3 0 1	69.00	19.41 ± 0.62
1 1 2	69.80	8.96 ± 0.58

Cr_2O_3

h k l	Angle	Rel I (%)
0 1 2	24.52	66.71 ± 1.71
1 0 4	33.62	100.0 ----
1 1 0	36.22	81.27 ± 4.78
0 0 6	39.77	7.36 ± 0.93
1 1 3	41.50	31.64 ± 0.60
2 0 2	44.22	5.24 ± 0.25
0 2 4	50.24	39.64 ± 1.10
1 1 6	54.86	96.42 ± 0.64
1 2 2	58.42	8.17 ± 0.74
2 1 4	63.48	31.06 ± 1.55
3 0 0	65.13	39.31 ± 1.43
1 0 10	72.95	20.88 ± 1.53

CeO_2

h k l	Angle	Rel I (%)
1 1 1	28.61	100.0 ----
2 0 0	33.14	27.21 ± 0.46
2 2 0	47.54	54.21 ± 0.56
3 1 1	56.39	43.58 ± 0.60
2 2 2	59.14	8.29 ± 0.38
4 0 0	69.46	8.03 ± 0.25

Table 6. Information Microstructural Data for SRM 674b

Component	Crystallite Size, Mass Percent Less Than (nm)					Microstrain, (ε_0) [(1)]
	10 %	25 %	50 %	75 %	90 %	
ZnO	5.3	8.8	15.5	27.3	45.4	*
TiO_2	0.8	1.6	3.6	8.0	16.7	0.00017
Cr_2O_3	73.4	93.5	122.4	160.3	204.2	0.00010
CeO_2	11.4	18.6	31.9	54.6	88.6	*

[(1)] *: None detected.

Table 7. Information Particle Size Data for SRM 674b Determined Using a Disk Centrifuge Analyzer

Mass Percent Less Than (μm)	Components			
	ZnO	TiO_2	Cr_2O_3	CeO_2
10 %	0.22	0.41	0.34	0.53
16 %	0.28	0.55	0.38	0.65
50 %	0.58	0.93	0.56	1.13
84 %	1.15	1.38	1.05	1.91
90 %	1.55	1.66	1.45	2.18

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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.