National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 674a

X-Ray Powder Diffraction Intensity Set

This Standard Reference Material (SRM) is of a set of five oxide powders which are intended for use as internal standards for quantitative x-ray diffraction analysis and as external standards for checking the intensity response of x-ray diffraction instruments. The five powders are α-Al_2O_3 (corundum structure), ZnO (wurtzite structure), TiO_2 (rutile structure), Cr_2O_3 (corundum structure), and CeO_2 (fluorite structure). This set of stable oxides covers a range of absorptivities, μ, from 126 to 2203 cm^{-1} for $CuK\alpha$ radiation. This range of absorptivities allows the user to nominally match the absorptivity of an unknown with a standard in order to minimize the effects of microabsorption. The particle size distributions of all the powder samples are centered about the 1 μm range, with upper limits of about 5 μm. The small particle size serves to minimize the effects of both microabsorption and extinction.

Measurements leading to the certification of SRM 674a were performed by J.P. Cline, A.L. Dragoo, M. Kuchinski, L. Lum, and C.R. Robbins of the Ceramics Division of the Institute for Materials Science and Engineering.

The technical leadership and overall coordination and direction of the certification measurements were provided by J. P. Cline and A.L. Dragoo, respectively, of the Ceramics Division of the Institute for Materials Science and Engineering..

Statistical design of the measurements and interpretation and analysis of the certification data were provided by K. Eberhardt of the Statistical Engineering Division of the National Engineering Laboratory.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.L. McKenzie.

Gaithersburg Md. 20899
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Stanley D. Rasberry, Chief
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The powders comprising SRM 674a were characterized by a number of different analytical methods. Particle size distributions of the powders were measured with an x-ray sedigraph. X-ray diffraction intensity measurements were made with a Bragg-Brentano type (vertical goniometer) diffractometer having a fixed incident beam slit (constant irradiated volume), a 0.01 degree receiving slit, a graphite diffracted beam monochrometer, and a scintillation detector. In addition to the relative intensity of the diffraction lines for a given sample, data are also presented in terms of the Reference Intensity Ratio (RIR). The RIR is defined as the intensity ratio of the strongest lines measured from each of the two phases in a 50-50 mixture. The I/I_c of a given phase is its RIR with the intensity value in the denominator being that of the strongest line of α-Al_2O_3, corundum structure, in a 50-50 mixture. RIR (I/I_c) data were collected on an instrument identical to that used for relative intensity measurements, except equipped with a sample spinner. Lattice parameter data reduction was made using the software NBSLSQ and integrated intensity data were collected and reduced to relative intensity (I^{rel}) and RIR (I/I_c) values with NBS*QUANT.

Certified relative intensity (I^{rel}) and RIR (I/I_c), values are given in Tables 1 and 2, respectively. The sigma values reported for the relative intensity data are the standard errors of the means of 6 measurements; those reported for the RIR (I/I_c) data are the standard errors of the means of 3 measurements. Lattice parameter and particle size data are shown in Tables 3 and 4, respectively. The sigma values reported for the lattice parameters are the standard errors of the means of 20 measurements. Integrated intensity scans ranged from a width of 2 to 2.5 degrees 2θ. Measurement of the 301 and 112 peaks of TiO_2 suffered from some degree of overlap, relative intensities reported for them are biased below the actual value.

All diffraction intensity measurements are subject to errors arising from photon counting statistics, particle counting statistics, and preferred orientation effects. Photon counting statistics are tracked throughout data collection and processing by NBS*QUANT. Values outside error windows indicated by this random nature of photon counting can be considered systematic in origin. Particle counting statistics arise from a potential of too few particles being illuminated by the incident beam--this is not considered to be a problem with SRM 674a due to the small particle size of the samples. Preferred orientation effects result from alignment of the crystallites due to the anisotropic fracture habit. The samples chosen here are not prone to large degrees of orientation, though some slight orientation effects can be expected in any system.

The relative intensity values indicated some degree of preferred orientation, a small number of the values measured for certain reflections fell outside the range allowable by photon counting statistics. Preferred orientation manifested itself as an appearance of random deviations about a mean as a function of the particular sample mount. Relative intensity measurements were made on samples which had been loaded by the side drifting method. The RIR data, collected with the aid of the sample spinner, did not suffer from effects of preferred orientation.

High quality scans of the five powders indicated a small amount, less than 1% (by peak height), of unidentified phase(s) in each. The alumina contained an impurity which is interpreted, due to the breadth of the peaks, to be a surface phase. The TiO_2 contained approximately 1.5% anatase, in addition to an impurity phase of unknown composition. The CeO_2 contained a phase which corresponded exactly with JCPDS #6-665; CoMnSn, though this is not considered to be the actual phase present.

Table 1. SRM 674a
Relative Intensity Data

α-Al_2O_3, $\mu_{CuK\alpha}$ = 126 cm^{-1}

hkl	2θ	Relative Intensity	σ
012	25.54	58.7	0.19
104	35.10	87.2	0.22
110	37.72	38.3	0.09
113	43.30	100.0	—
024	52.48	46.1	0.07
116	57.44	94.1	0.09
214	66.44	35.7	0.09
300	68.14	52.4	0.34

TiO_2, $\mu_{CuK\alpha}$ = 536 cm^{-1}

hkl	2θ	Relative Intensity	σ
110	27.38	100.0	—
101	36.04	42.7	0.09
111	41.18	20.4	0.03
211	54.28	56.6	0.07
220	56.58	16.6	0.04
002	62.72	8.0	0.03
301	68.96	(17.2)	0.07
112	69.76	(5.6)	0.12

ZnO, $\mu_{CuK\alpha}$ = 279 cm^{-1}

hkl	2θ	Relative Intensity	σ
100	31.70	57.9	0.12
002	34.36	42.1	0.06
101	36.18	100.0	—
102	47.48	23.5	0.02
110	56.52	35.4	0.07
103	62.80	31.6	0.05
200	66.30	4.8	0.01
112	67.88	25.6	0.06

Cr_2O_3, $\mu_{CuK\alpha}$ = 912 cm^{-1}

hkl	2θ	Relative Intensity	σ
012	24.42	67.6	0.35
104	33.53	100.0	—
110	36.12	80.8	0.68
113	41.40	30.9	0.13
024	50.14	36.9	0.19
116	54.76	92.1	0.22
214	63.38	28.6	0.13
300	65.04	37.5	0.20

CeO_2, $\mu_{CuK\alpha}$ = 2203 cm^{-1}

hkl	2θ	Relative Intensity	σ
111	28.60	100.0	—
200	33.12	27.8	0.05
220	47.52	55.2	0.19
311	56.38	43.8	0.14

Table 2. Reference Intensity Ratio, I/I_c

	I/I_c	σ
ZnO	5.33	0.06
TiO_2	3.32	0.04
Cr_2O_3	2.16	0.02
CeO_2	13.15	0.10

Additional Information: The following tables present data describing the lattice parameters and particle size distribution of the samples in SRM 674a. These data are not certified, but are presented as Information Only values which may be of interest to the user of this SRM.

Table 3. Lattice Parameters

	a (Å)	σ	c (Å)	σ
α-Al$_2$O$_3$	4.759397	0.000080	12.99237	0.00022
ZnO	3.249074	0.000055	5.206535	0.000101
TiO$_2$	4.593939	0.000062	2.958862	0.000063
Cr$_2$O$_3$	4.959610	0.000079	13.59747	0.00025
CeO$_2$	5.411102	0.000097		

Table 4. Particle Size Data

	90% finer than (μm)	50% finer than (μm)
α-Al$_2$O$_3$	1.5	0.6
ZnO	2.5	0.79
TiO$_2$	0.7	0.45
Cr$_2$O$_3$	2.0	0.87
CeO$_2$	2.8	0.75