J. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 674

X-Ray Powder Diffraction Intensity Set

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This Standard Reference Material (SRM) consists of five different phases. The phases, separately bottled, are α-Al_2O_3 (corundum structure), ZnO (wurtzite structure), TiO_2 (rutile structure), Cr_2O_3 (corundum structure), and CeO_2 (fluorite structure). These phases can be used as internal standards for quantitative analysis and as external standards for checking the intensity response of x-ray diffraction instruments. The five phases cover the range of linear absorption coefficient from 100 to 1000 cm^{-1} for CuKα radiation. The intensity values are for a constant diffracting volume (fixed divergent slit and infinitely thick mounting or equivalent).

The certified relative intensity values (CuKα radiation), for the major diffraction lines below 70° 2(θ), for each phase are given in Table 1. The certified reference intensity ratios (I/Ic) for ZnO, TiO_2, Cr_2O_3, and CeO_2, relative to α-Al_2O_3 and the lattice parameters are given in Tables 2 and 3. The uncertainties of the relative intensity and lattice parameter values are the standard deviations derived from multiple measurements. The uncertainties of the reference intensity ratio values were determined from the value of the internal inconsistency derived from all possible binary mixtures.

The average crystallite size for each of the five phases is near 2.0 μm. Crystallite size broadening of the diffraction peaks is small but present. This is particularly apparent for α-Al_2O_3, which has a nominal crystallite size less than 1.0 μm. The lattice parameters, uncorrected for refraction, were determined with a relative uncertainty of about three parts in 10^5 using an internal standard. This uncertainty in the lattice parameter must be increased by a factor of two or three if one wishes to determine absolute d-spacings as the uncertainties due to the internal standard 2θ's and to thermal expansion would need to be added.

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.K. Kirby.

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(over)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

Table 1. Relative Intensitites of Major Lines

CuKα Radiation (μ = 124.1 cm^{-1})

hkl	2θ	I^{rel}	
α-Al_2O_3[a]	(Corundum Structure)		
012	25.58°	55.4 ± 2.4	
104	35.13	87.4 ± 1.9	
110	37.78	36.5 ± 1.4	
113	43.36	100.0	
024	52.55	45.5 ± 1.3	
116	57.52	92.5 ± 2.6	
214	66.54	34.7 ± 1.0	
300	68.19	55.5 ± 2.2	
ZnO	(Wurtzite Structure)		
100	31.75°	57.6 ± 1.1	
002	34.44	40.2 ± 1.4	
101	36.25	100.0	
102	47.54	22.8 ± 0.5	
110	56.55	34.4 ± 0.8	
103	62.87	31.0 ± 0.9	
200	66.40	4.7 ± 0.2	
112	67.91	25.9 ± 0.6	
TiO_2[b]	(Rutile Structure)		
110	27.42°	100.0	
101	36.04	44.0 ± 1.7	
TiO_2	(cont.)		
111	41.20°	21.4 ± 1.4	
211	54.27	56.9 ± 2.8	
220	56.51	16.9 ± 1.4	
002	62.76	8.2 ± 0.7	
301	68.93	(21.1)	approximate due to
112	69.79	(11.1)	partial overlap
Cr_2O_3[c]	(Corundum Structure)		
012	24.52°	69.2 ± 1.6	
104	33.59	100.0	
110	36.20	87.1 ± 2.3	
113	41.47	32.3 ± 1.0	
024	50.21	39.4 ± 0.8	
116	54.81	94.5 ± 2.2	
214	63.43	30.8 ± 1.3	
300	65.09	40.6 ± 1.0	
CeO_2[d]	(Fluorite Structure)		
111	28.55°	100.0	
200	33.08	28.0 ± 0.8	
220	47.49	53.5 ± 2.0	
311	57.32	43.4 ± 2.3	

[a] Two broad, very weak peaks (32.80 and 45.5° 2θ) can be detected. These lines are possibly due to γ-Al_2O_3 (PDF 29-63).

[b] Anatase was observed at ~1.0 wt. % level. Unexplained very weak peaks at 13.9 and 18.0° 2θ were also detected.

[c] Small unexplained intensity deviations from smooth background.

[d] Three unexplained, broad, very weak diffraction peaks were observed at 14.40, 16.64, and 23.75° 2θ.