

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 -Al₂O₃ (corundum structure), ZnO (wurtzite structure), TiO₂ (rutile structure), Cr₂O₃ (corundum structure), and CeO₂ (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 CuK α radiation. The intensity values are for a constant diffracting volume (fixed divergent slit and infinitely thick mounting or equivalent).

The certified relative intensity values (CuK α radiation), for the major diffraction lines below 70° 2(θ), for each phase are given in Table 1. The certified reference intensity ratios (I/I_c) for ZnO, TiO₂, Cr₂O₃, and CeO₂, relative to α -Al₂O₃ 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 μ m. Crystallite size broadening of the diffraction peaks is small but present. This is particularly apparent for α -Al₂O₃, which has a nominal crystallite size less than 1.0 μ m. The lattice parameters, uncorrected for refraction, were determined with a relative uncertainty of about three parts in 10⁵ 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 2 θ '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.

Washington, D.C. 20234
June 1, 1983

(over)

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Table 1. Relative Intensities of Major Lines

CuK α Radiation ($\mu = 124.1 \text{ cm}^{-1}$)

<u>hkl</u>	<u>2θ</u>	<u>I^{rel}</u>	<u>hkl</u>	<u>2θ</u>	<u>I^{rel}</u>
$\alpha\text{-Al}_2\text{O}_3^{\text{a}}$ (Corundum Structure)			TiO ₂ (cont.)		
012	25.58°	55.4 $\pm$ 2.4	111	41.20°	21.4 $\pm$ 1.4
104	35.13	87.4 $\pm$ 1.9	211	54.27	56.9 $\pm$ 2.8
110	37.78	36.5 $\pm$ 1.4	220	56.51	16.9 $\pm$ 1.4
113	43.36	100.0	002	62.76	8.2 $\pm$ 0.7
024	52.55	45.5 $\pm$ 1.3	301	68.93	(21.1) approximate due to
116	57.52	92.5 $\pm$ 2.6	112	69.79	(11.1) partial overlap
214	66.54	34.7 $\pm$ 1.0			
300	68.19	55.5 $\pm$ 2.2			
ZnO (Wurtzite Structure)			Cr ₂ O ₃ ^c (Corundum Structure)		
100	31.75°	57.6 $\pm$ 1.1	012	24.52°	69.2 $\pm$ 1.6
002	34.44	40.2 $\pm$ 1.4	104	33.59	100.0
101	36.25	100.0	110	36.20	87.1 $\pm$ 2.3
102	47.54	22.8 $\pm$ 0.5	113	41.47	32.3 $\pm$ 1.0
110	56.55	34.4 $\pm$ 0.8	024	50.21	39.4 $\pm$ 0.8
103	62.87	31.0 $\pm$ 0.9	116	54.81	94.5 $\pm$ 2.2
200	66.40	4.7 $\pm$ 0.2	214	63.43	30.8 $\pm$ 1.3
112	67.91	25.9 $\pm$ 0.6	300	65.09	40.6 $\pm$ 1.0
TiO ₂ ^b (Rutile Structure)			CeO ₂ ^d (Fluorite Structure)		
110	27.42°	100.0	111	28.55°	100.0
101	36.04	44.0 $\pm$ 1.7	200	33.08	28.0 $\pm$ 0.8
			220	47.49	53.5 $\pm$ 2.0
			311	57.32	43.4 $\pm$ 2.3

^aTwo broad, very weak peaks (32.80 and 45.5° 2 θ) can be detected. These lines are possibly due to $\gamma\text{-Al}_2\text{O}_3$ (PDF 29-63).^bAnatase was observed at ~ 1.0 wt. % level. Unexplained very weak peaks at 13.9 and 18.0° 2 θ were also detected.^cSmall unexplained intensity deviations from smooth background.^dThree unexplained, broad, very weak diffraction peaks were observed at 14.40, 16.64, and 23.75° 2 θ .

Table 2. Reference Intensity Ratios (relative to the 113 line of Al_2O_3)
 $\text{CuK}\alpha$ Radiation ($\mu = 124.1 \text{ cm}^{-1}$)

Phase	hkl	I/Ic
ZnO	101	5.17 ± 0.13
TiO_2	110	3.39 ± 0.12
Cr_2O_3	104	2.10 ± 0.05
CeO_2	111	7.5 ± 0.2

Table 3. Lattice Parameters ($25 \pm 2 \text{ }^\circ\text{C}$)
 $\text{CuK}\alpha$ Radiation ($\mu = 124.1 \text{ cm}^{-1}$)

Phase	Crystal Structure	a ($\text{\AA}$)	c ($\text{\AA}$)
Al_2O_3	Trigonal	4.75893 ± 0.00010	12.9917 ± 0.0007
ZnO	Hexagonal	3.24981 ± 0.00012	5.20653 ± 0.00013
TiO_2	Tetragonal	4.59365 ± 0.00010	2.95874 ± 0.00008
Cr_2O_3	Trigonal	4.95916 ± 0.00012	13.5972 ± 0.0006
CeO_2	Cubic	5.41129 ± 0.00008	