U. S. Department of Commerce
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National Bureau of Standards
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National Bureau of Standards

Certificate

Standard Reference Material 675

Low 2θ (Large d-Spacing) Standard

for X-Ray Powder Diffraction

C. R. Hubbard

This Standard Reference Material (SRM) was prepared for use as an external or internal low 2θ (large d-spacing) calibration standard for powder diffractometry. The material is synthetic fluorophlogopite mica and is best suited for reflection diffractometry as pressed samples have a high degree of preferred orientation in which only the 00ℓ reflections have significant intensity. The mica was ground to pass a 75-μm (200 mesh) sieve.

A total of 28 samples, mixed with silicon [1] and tungsten [2] internal standards, were measured using a high angle goniometer controlled by a minicomputer. For each sample, the peak positions of eighteen internal standard and eleven 00ℓ fluorophlogopite reflections were determined by profile fitting procedures and corrected for the effects of thermal expansion. The fluorophlogopite reflections were then corrected for instrumental and physical aberrations (except refraction) through use of the internal standard lines [3].

Each corrected set was refined by a least-square routine that minimized $\Sigma (\theta_{obs} - \theta_{calc})^2$ to obtain estimates of d(001) and a transparency-displacement parameter. The certified value, based on the average of the 28 refined d values at 25.0 °C and uncorrected for refraction, is

$$d(001) = 9.98104 \pm 0.00007 \text{ Å}$$

where $\lambda(CuK\alpha_1) = 1.5405981$ Å [4]. The estimated uncertainty given above is dominated by the random errors in measurement. Without correction for the transparency-displacement effect [3], the 2θ angles for the 001 and 003 reflections for CuKα x rays should be accurate to better than 0.01° 2θ for a highly absorbing sample. For a sample with absorption similar to that of fluorophlogopite, these lines will be accurate to nearly 0.001° 2θ.

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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Washington, D.C. 20234

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

The 2θ values given in Table 1 were calculated from the certified value of d(001). The approximate peak height relative intensities, I(rel), measured using θ-compensating slits, are also given in Table 1. They can be used as an aid in identifying the fluorophlogopite lines when SRM 675 is mixed with other phases. The uncertainty in I(rel) is approximately ± 1 in the least significant digit except for the 001 line. For this low angle line, the uncertainty is ± 5. These intensities, converted to an approximately fixed sample volume basis by applying a $1/\sin\theta$ factor, are given in the last column. These intensities were reproducible to ± 1 in the least significant digit except for the 001 reflection for which the uncertainty is approximately ± 15.

Table 1
Diffraction Angles and Relative Intensities
[$\lambda(CuK\alpha_1)$ = 1.5405981 Å, T = 25.0 °C]

ℓ^+	$2\theta_{peak}$	I(rel)	
		θ--Comp Slits	Fixed Volume
1	8.853°	27	81
2	17.759	3.2	4.8
3	26.774	100	100
4	35.962	9	6.8
5	45.397	46	28
6	55.169	3.2	1.6
7	65.399	4.6	2.0
8	76.255	5.4	2.0
10	101.025	1.7	0.5
11	116.193	1.8	0.5
12	135.674	0.5	0.1

+The 009 reflection is omitted as its intensity is very low.

References

[1] C. R. Hubbard (1982) "Certification of Si Powder Diffraction Standard Reference Material 640a", J. Appl Cryst., to be published.

[2] H. E. Swanson, H. F. McMurdie, M. C. Morris, and E. H. Evans (1966) NBS Monograph 25, Sec. 4, pp. 3-4.

[3] C. R. Hubbard (1982) "Fluorophlogopite--Low 2θ Large d-Spacing Powder Diffraction Standard Reference Material 675", submitted to J. Appl. Cryst.

[4] R. Deslattes and A. Hening (1973) Phy. Rev. Lett., 31, 972.

Table 2. Reference Intensity Ratios (relative to the 113 line of Al_2O_3)

CuKα Radiation (μ = 124.1 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 ± 2 °C)

CuKα Radiation (μ = 124.1 cm^{-1})

Phase	Crystal Structure	a (Å)	c (Å)
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	