National Institute of Standards & Technology

Certificate of Analysis

STANDARD REFERENCE MATERIAL 660

Instrument Line Position and Profile Shape Standard

For X-Ray Powder Diffraction

Standard Reference Material (SRM) 660 is a lanthanum hexaboride fine powder intended for use as an X-ray powder diffraction instrument line position and profile shape standard. As a line position standard the powder is useful to check instrument alignment and to provide a 2θ calibration curve. As a profile shape standard, the material is useful to accurately model the instrumental diffraction line shape as a function of 2θ. The model parameters can be used to generate an instrumental line shape at any 2θ, useful, for example, in crystallite size and residual microstrain analysis or in Rietveld refinement of atomic structure.

The material is a high purity lanthanum hexaboride (LaB_6) that was ball milled and size classified to nominally <10 μm. The powder was then annealed in argon atmosphere to reduce residual microstrain broadening. The anneal step introduced additional agglomerates which were broken down by a short ball milling step. Spectrochemical analysis prior to sizing, annealing and final ball milling indicates the material to be >99% pure. A trace crystalline impurity was detected in the final powder.

The certified lattice parameter is the average of lattice parameters determined from 12 samples mixed with a tungsten internal standard [1]. All major diffraction line profiles above 100 degrees 2θ were collected and analyzed using a split Pearson VII function profile model [2]. The temperature was maintained at 299 ± 1 K. The four tungsten lines were used to calibrate for sample, instrumental and physical aberrations (except refraction) [3]. The lattice parameter of each sample was obtained by least-squares refinement of the corrected 2θ values. The certified lattice parameter, uncorrected for refraction, at 299 K is

$$\langle a \rangle = 4.15695 \text{ Å}$$

$$\text{sigma}(\langle a \rangle) = 0.00006 \text{ Å}$$

where λ ($CuK\alpha_1$) = 1.5405981 Å [4]. The estimated uncertainty given above includes contributions from three sources (listed in decreasing importance): (1) uncertainty of the lattice parameter of the tungsten standard; (2) random errors of the measurement including thermal expansion effects; and (3) the uncertainty in λ ($CuK\alpha_1$).

The technical coordination leading to certification was provided by C.R. Hubbard, with technical measurements provided by Y. Zhang and C.R. Hubbard of the Ceramics Division of the NIST Institute of Materials Science and Engineering.

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
Office of Standard Reference Materials

(Over)

Sample homogeneity was confirmed by particle size analysis, by X-ray diffraction line profile measurements, and by lattice parameter determinations. A total of 12 to 16 randomly selected bottles were examined in each procedure. The particle size analysis, performed with a sedigraph, yields a size distribution of 96% less than 20 μm, 55% less than 10 μm, and >99% above 1 μm. Table 1 shows a typical size distribution curve as determined by sedigraph. The X-ray diffraction line profile measurements were performed using a high angle $\theta/2\theta$ goniometer utilizing a fine focus Cu target X-ray tube, 6 degree take off angle, approximately 1 degree incident divergent slit, 0.1 degree receiving slit, diffracted beam graphite monochromater, scintillation detector, and pulse-height analysis. Three 2θ regions (36.2 - 38.6, 74.7 - 76.9, and 139.5 - 144.3) were digitally recorded and analyzed using a split Pearson VII function [1]. Homogeneity was also confirmed by measurement of the lattice parameters from 12 randomly selected bottles.

Based on an international round robin to compare potential line profile reference materials, the certified LaB_6 has the narrowest line profiles observed [2]. Careful comparison with other specially prepared LaB_6 samples with a much larger crystallite size shows that there is a small residual microstrain line broadening (typically 0.01 degrees 2θ) observable at 2θ values above 110 degrees.

The 2θ values given in Table 2 were calculated from the certified value of the lattice parameter. The relative intensities, from reference [6] and given in Table 2, can be used as an aid in identifying the LaB_6 lines when the SRM is mixed with test materials.

Table 1

Typical Particle Size Distribution by Sedigraph

(These Values Are Not Certified and Are Supplied For Informational Use Only)

Size (μm)	% Smaller
40	99
30	99
20	97
10	57
8	41
6	23
4	9
2	2
1	<1

Table 2

Calculated Diffraction Angles and Relative Intensities

(T = 299 K)

(These Values Are Not Certified and Are Supplied For Informational Use Only)

hkl	I(rel)	2θ	hkl	I(rel)	2θ
1 0 0	54	21.358	3 2 1	13	87.790
1 1 0	100	30.384	4 0 0	2	95.669
1 1 1	41	37.441	4 1 0	8	99.640
2 0 0	22	43.506	3 3 0	7	103.658
2 1 0	46	48.957	3 3 1	3	107.746
2 1 1	24	53.988	4 2 0	4	111.930
2 2 0	8	63.217	4 2 1	9	116.241
3 0 0	23	67.546	3 3 2	3	120.719
3 1 0	16	71.744	4 2 2	3	130.404
3 1 1	10	75.843	5 0 0	3	135.795
2 2 2	2	79.868	5 1 0	10	141.769
3 2 0	6	83.844	5 1 1	6	148.657

REFERENCES:

1. Swanson, H. E., McMurdie, H. F., Morris, M. C., and Evans, E. H, Standard X-Ray diffraction powder patterns. National Bureau of Standards (U.S.) Monograph 25, Section 4; 1966, 83p.

2. Stewart, J. M., Zhang, Y., Hubbard, C. R., Morosin, B., and Venturini, E. L., "XRAYL: A powder diffraction profile refinement program. National Institute of Standards and Technology (U.S.) NISTIR 88-3850; 1988, 29 p.

3. Hubbard, C. R., Certification of Si powder diffraction Standard Reference Material. J. Appl. Cryst. 16: 285-88; 1983.

4. Deslattes, R. D. and Henins, A., X-ray to visible wavelength ratios. Phys. Rev. Letters 31: 972-75; 1973.

5. Fawcett, T. et. al., Establishing an instrumental peak profile calibration standard for powder diffraction analyses: International round robin conducted by the JCPDS-ICDD and the U.S. National Bureau of Standards. Powder Diffraction 3(4):209-218; 1988.

6. Morris, M. C. et. al., Standard X-ray diffraction powder patterns. National Bureau of Standards (U.S.) Monograph 25, Section 20; 1984, 149p.