National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1990

Single Crystal Diffractometer Alignment Standard - Ruby Sphere

Standard Reference Material (SRM) 1990 is intended primarily for use as an alignment standard for single crystal diffractometry. One unit consists of three chromium-doped single crystal aluminum oxide (ruby) spheres. The spheres are nominally 152 μm in diameter with 1.3 μm sphericity. The spherical geometry was chosen to facilitate alignment and to avoid corrections for absorption. These spheres produce reflections at high angles for copper and molybdenum radiation. The space group is $R\bar{3}c$.

Certified Values and Uncertainties: The certified lattice parameters of SRM 1990 at 25 °C are:

a: 0.476080 nm ± 0.000029 nm
c: 1.299568 nm ± 0.000087 nm

The certified values were obtained using λ ($CuK\alpha_1$) = 0.154059292 (45) nm, and λ ($MoK\alpha_1$) = 0.070931631 (84) nm [1,2]. The estimated total uncertainty is an expanded uncertainty with a coverage factor, k = 3.18 (the resulting interval is an approximate 95 % confidence level), and accounts for (1) uncertainty due to a small variation in chromium content, (2) random errors of measurement, and (3) the small uncertainty in λ ($CuK\alpha_1$) and λ ($MoK\alpha_1$) [3]. Other systematic effects such as absorption, extinction, and divergence were not found to be significant and are not included in the uncertainty estimate.

Expiration of Certification: The certification of this SRM is valid indefinitely within the measurement uncertainties specified, provided the SRM is used in accordance with the instructions in this certificate. However, the certification will be nullified if the SRM is physically damaged, contaminated, or otherwise altered. If alteration is suspected, discontinue use and purchase a new unit. This SRM is a physical artifact, the physical properties of which are inherently stable under normal laboratory and usage conditions.

Reference Value: SRM 1990 may also be used as a reference for chromium content. The spheres contain 0.42 % ± 0.01 % chromium oxide (Cr_2O_3) expressed as a mole fraction, where the uncertainty represents an expanded uncertainty. The chromium content was measured using a microprobe, while specimen-to-specimen variability was measured by scanning electron microscope energy dispersive spectroscopy.

Technical coordination leading to certification of this SRM was provided by W. Wong-Ng of the NIST Ceramics Division with significant contributions from T. Siegrist of Lucent Technologies, Murray Hill, NJ.

Statistical consultation was provided by M.S. Levenson of the NIST Statistical Engineering Division.

Measurements of the chromium content were provided by J.T. Armstrong of the NIST Microanalysis Science Division and L.P. Cook of the NIST Ceramics Division.

The support aspects concerning the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by R.J. Gettings and C.R. Beauchamp.

Stephen W. Freiman, Chief
Ceramics Division

Gaithersburg, MD 20899
Certificate Issue Date: 28 February 2001

Nancy M. Trahey, Chief
Standard Reference Materials Program

Organization of the round robin project and demonstration of the viability of the ruby spheres were provided by G.T. DeTitta of Hauptman-Woodward Medical Research Institute, Buffalo, NY and L. Finger of the Geophysical Laboratory, Washington, DC.

Preparation and Certification Procedures: A boule of single crystal ruby, supplied by Arcanum Corporation, Ann Arbor, MI, was prepared by using the Verneuil technique (flame fusion) then ground into small spheres [4]. Four sets of spheres were measured, both at NIST and by cooperating laboratories, on three Enraf-Nonius CAD-4 diffractometers and on a Picker diffractometer using both molybdenum and copper radiation[1]. A total of 39 spheres were measured to obtain 45 data sets. The diffractometer control program, DIFRAC, developed at the National Research Council of Canada (NRC), was used for data collection and reduction for all data sets [5]. The $CuK\alpha_1$ and $MoK\alpha_1$ peak positions were determined by fitting the profiles of high angle peaks. Lattice parameters were obtained using least squares methods. Corrections for thermal expansion [6] and refraction [7] were applied to convert the data to 25 °C for each sample. The thermal expansion coefficients used were $\alpha_a = 5.0 \times 10^{-6}\ deg^{-1}$ and $\alpha_c = 6.66 \times 10^{-6}\ deg^{-1}$ [6]. These lattice parameters were verified by a powder diffraction study conducted at the U.S. Geological Survey on 60 crushed spheres using the Guinier-Hägg transmission technique [8].

Instructions for Use: Select one of the spheres, clean off adhesive using acetone or alcohol, and mount securely on the tip of a fiber (or capillary tube) about 0.1 mm in diameter using a minimum amount of adhesive material in the area of contact between the sphere and the fiber. Additional spheres are provided in the event of loss. This SRM is intended for use at ambient conditions. With appropriate thermal expansion corrections, the SRM can be used under non-ambient temperature conditions.

Table 1 lists information values for high-angle 2θ reflections to obtain both the initial sphere orientation and to accurately align the diffractometer. The h, k, and l values are the Miller indices, M is the multiplicity, and Fc is the calculated structure amplitude. The 2θ values are derived from the certified lattice parameters of the ruby spheres by using the PC version (MICRO-POWD) of computer software POWD10 [9]. The structure amplitudes are listed to serve as an approximate guide of the relative intensity of these reflections. These reflections, and symmetrically equivalent ones, should be selected such that the Miller indices h, k, and l are evenly distributed in reciprocal space. The same reflection should be measured at some or all of the eight possible diffractometer settings. It is recommended that the alignment routine incorporates the algorithm by King and Finger for calculating various alignment corrections [10].

Table 1. Information Values for High Angle 2θ Reflections

(A) For initial orientation

h	k	l	M	Fc	$2\theta_{CuK_{\alpha 1}}$ (°)	$2\theta_{MoK_{\alpha 1}}$ (°)
0	1	2	6	47	25.567	11.694
1	0	4	6	83	35.139	15.978
1	1	0	6	61	37.762	17.137
0	0	6	2	13	41.665	18.848
3	0	0	6	141	68.180	29.910
0	0	12	2	57	90.678	38.232

[1] Certain commercial equipment, instruments, or materials are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

(B) For accurate alignment

h	k	l	M	Fc	$2\theta_{CuK_{\alpha 1}}$ (°)	$2\theta_{MoK_{\alpha 1}}$ (°)
1	3	4	12	51	91.144	
2	2	6	12	74	95.203	
0	4	2	6	38	98.342	
2	1	10	12	64	101.031	
4	0	4	6	38	103.262	
3	1	8	12	36	110.931	
2	2	9	12	30	114.010	
3	2	4	12	64	116.030	
0	1	14	6	52	116.553	
4	1	0	6	47	117.777	
4	1	3	12	26	121.956	
1	3	10	12	66	127.605	
3	0	12	6	41	129.802	
2	0	14	6	58	131.031	
4	1	6	12	56	135.971	
1	1	15	12	29	142.225	
4	0	10	6	69	145.049	
0	5	4	6	56	149.060	
1	2	14	12	52	149.986	
1	0	16	6	32	150.300	
3	3	0	6	79	152.239	
0	4	20	6	42		80.363
5	1	16	12	28		80.798
7	1	0	6	26		80.998
7	0	10	6	50		82.767
3	3	18	12	36		83.234
1	7	6	12	29		84.097
1	1	24	12	41		84.397
5	4	4	12	32		85.789
6	3	0	6	42		86.120
0	8	4	6	32		88.336
4	2	20	12	26		90.584
2	2	24	12	37		92.038
2	6	14	12	34		93.525
8	1	4	12	32		95.978
3	4	20	12	38		98.243
6	1	20	12	30		103.425
4	6	10	12	36		105.883
7	3	10	12	41		108.546
0	7	20	6	39		108.721
0	0	30	2	44		109.913
5	2	24	12	29		115.794
3	0	30	6	40		118.280
1	9	10	12	26		119.713

Cooperating Round Robin Laboratories:

Geophysical Laboratory, Washington, DC; L. Finger
Hauptman-Woodward Medical Research Institute, Buffalo, NY; G.T. DeTitta
Lucent Technology, Murray Hill, NJ; T. Siegrist
National Research Council of Canada, Ottawa, Ont; G.D. Enright
National Research Council of Canada, Ottawa, Ont; E.J. Gabe
Oak Ridge National Laboratory, Oak Ridge, TN; C.R. Hubbard
U.S. Geological Survey, Reston, VA; H. Evans

REFERENCES

[1] Härtwig, J., Hölzer, G., Förster, E., Goetz, K., Wokulska, K., and Wolf, J., "Remeasurement of Characteristic X-ray Emission Lines and Their Application to Line Profile Analysis and Lattice Parameter Determination," Phys. Stat. Sol. A **143**, p. 23, (1994).

[2] Cohen, E.R. and Taylor, B.N., "The 1986 Adjustment of the Fundamental Physical Constants," Rev. Mod. Phys. **59**, p. 1121, (1987).

[3] Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurements," NIST Tech. Note 1297, U.S. Government Printing Office, Washington DC, (1994); available at http://physics.nist.gov/Pubs/.

[4] Hurle, D.T.J., "Melt Growth," in Crystal Growth: An Introduction, edited by P. Hartman, North-Holland Publishing Company, p. 215, (1973).

[5] DIFRAC, software suite for data collection and reduction of x-ray single crystal diffractometers, developed by E. Gabe, et al. at the National Research Council (NRC) in Ottawa, Canada.

[6] Belyaev, L.M., "Ruby and Sapphire," Nauka Publishers, Moscow, translated from Russian, National Bureau of Standards, National Science Foundation, and Amerind Publishing Co. Pvt. Ltd., New Delhi, India, p. 7, (1980).

[7] Hubbard, C.R. and Mauer, F.A., "Precision and Accuracy of the Bond Method as Applied to Small Spherical Crystals," J. Appl. Cryst. **9**, p. 1, (1976).

[8] Hägg, G. and Ersson, N.O., "An Easily Adjustable Guinier Camera of Highest Precision," Acta Cryst. A25, Sppl. p. S64, (1960).

[9] Smith, D.K., Nichols, M.C., and Zolensky, M.E., "A FORTRAN IV Program for Calculating X-ray Powder Diffraction Patterns-Version 10," The Pennsylvania State University, University Park, PA, p. 72, (1983).

[10] King, H.E. and Finger, L.W., "Diffracted Beam Crystal Centering and Its Application to High Pressure Crystallography," J. Appl. Crystallography, **12**, p. 374, (1979).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776, fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet www.nist.gov/srm.