

**National Bureau of Standards**  
**Certificate of Analysis**  
**Standard Reference Material 1879**  
**Respirable Cristobalite**  
**(Quantitative X-Ray Powder Diffraction Standard)**

This Standard Reference Material (SRM) is intended primarily for use in the determination of cristobalite by x-ray powder diffraction methods (XRD) according to NIOSH Analytical Method P & CAM 259<sup>1</sup> or equivalent methods.

The certified crystalline purity, as determined by a quantitative x-ray diffraction modified method of additions technique for determination of amorphous silica content, is

$98.0 \pm 1.0$  wt. % crystalline cristobalite

The uncertainty of the certified value,  $\pm 1.0$  wt. %, is comprised of a standard deviation of 0.5 wt. % due to random and inhomogeneity errors plus an estimated likely systematic error of 0.5 wt. %.

No other crystalline phases were detected with an x-ray powder diffractometer or a Guinier de Wolff camera.

The homogeneity of this material was tested by four independent techniques: particle size distribution measurements, lattice parameter measurements, XRD relative intensity measurements and XRD intensity ratio measurements. No significant inhomogeneity relative to the certified value of weight percent crystallinity was detected.

The technical measurements leading to the certification of this SRM were performed by M. Kuchinski, C. Robbins and C. Hubbard under the direction of C. Hubbard, all of the Ceramics Division of the Center for Materials Science.

Consultation and evaluation of statistical analysis for the certification of this SRM were provided by K. Eberhardt of the Statistical Engineering Division of the National Engineering Laboratory.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.L. McKenzie.

Supplemental Information (NON-CERTIFIED) on Cristobalite Powder:

The equivalent spherical diameter as determined by an x-ray sedigraph technique was found to range from 2 - 5 microns (80 % by mass of the particles), with 8% by mass less than 2 microns. The mass median equivalent spherical diameter as determined from these measurements is 3.3 microns.

Using  $\text{CuK}\alpha_1$  radiation ( $\lambda = 1.5405981 \text{ \AA}$ ) the lattice parameters (estimated standard deviation in parentheses) at  $26 \text{ }^\circ\text{C} (\pm 1^\circ)$  were found to be  $\langle a \rangle = 4.9732(7) \text{ \AA}$ ,  $\langle c \rangle = 6.9226(10) \text{ \AA}$ , and the reference intensity ratio against  $\text{Cr}_2\text{O}_3$  (SRM 674) was  $\langle \text{RIR}_{\text{Cris}, \text{Cr}_2\text{O}_3} \rangle = 3.08$ ,  $\sigma = 0.20$ .

The average relative intensities obtained from integrated intensity measurements of all diffraction peaks less than 50 degrees two-theta are reported in Table 1.

Cautions

This SRM is not intended for use as a particle size reference material: it agglomerates readily during handling and storage resulting in an uncharacterized particle size distribution. Agglomeration has not been shown to affect the x-ray analysis.

Health hazards are described in the Safety Data Sheet.

This SRM is intended for "IN VITRO" use and analysis only. Respirable cristobalite has been linked with the disease silicosis.

Table 1. Average Relative Intensities of Cristobalite SRM 1879 for  $\text{CuK}\alpha$  Radiation

| <u>HKL</u> | <u>2 <math>\theta</math></u> | <u><math>\langle I^{\text{REL}} \rangle</math></u> | <u>SD</u> |
|------------|------------------------------|--|-----------|
| 101        | 22.0                         | 100  | -         |
| 110        | 25.3                         | 0.5  | 0.1       |
| 111        | 28.4                         | 8.7  | 0.1       |
| 102        | 31.5                         | 10.8   | 0.2       |
| 200,112    | 36.1, 36.3                   | 17.6   | 0.4       |
| 211        | 42.7                         | 2.8  | 0.1       |
| 202        | 44.8                         | 2.5  | 0.1       |
| 113        | 47.1                         | 5.0  | 0.1       |
| 212        | 48.6                         | 5.1  | 0.2       |

REFERENCES

1. NIOSH Manual of Analytical Methods, Volume 5, may be ordered from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402 or the National Information Service, Springfield, VA 22161.