U.S. Department of Commerce
Elliot L. Richardson,
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards Certificate

Standard Reference Material 2679

Quartz on Filter Media

This Standard Reference Material is intended primarily for use as an analytical standard for the determination of quartz in the workplace atmosphere. The SRM consists of a mixture of quartz and clay deposited on a set of membrane filters. Only the quartz content is certified.

	Quartz, μg/filter	clay, μg/filter
Filter A	3.8 ± 0.5	(400)
Filter B	29.9 ± 3.6	(370)
Filter C	76.1 ± 9.1	(320)
Filter D	193.2 ± 23.2	(200)

The filters were prepared by a two-stage process from aqueous suspensions of the components, as described on the reverse side of this certificate.

The certified values are based upon determination of the quartz content by a spectrophotometric chemical procedure. In these analyses, the entire filters were dissolved and digested in acid before measurement. The certified values are the means of analytical values. The uncertainties represent the 95 percent tolerance limits based on measurement error and variability among samples*.

The filters are contained in individual plastic boxes, labelled A, B, C, and D, respectively. An entire filter must be used for each measurement as the materials are not uniformly distributed.

The filters were prepared by R. W. Burke and B. I. Diamondstone. Spectrophotometric analyses were made by E. R. Deardorff. Homogeneity measurements were made by x-ray fluorescence, by R. L. Myklebust.

The overall direction and coordination of the technical measurements leading to certification were under the chairmanship of J. K. Taylor.

The technical and support aspects involved in certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

*In brief, if measurements were made on all the units, almost all (at least 95 percent) of these measured values would be expected to fall within the indicated tolerance limits with a confidence coefficient of 95 percent (or probability = .95)

See page 14, The role of Standard Reference Materials in Measurement Systems, NBS Monograph 148, 1975. The concept of tolerance limit is also discussed in Chapter 2, Experimental Statistics, NBS Handbook 91, 1966.

Washington, D.C. 20234
September 24, 1976

J. Paul Cali, Chief
Office of Standard Reference Materials

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Supplemental Information

The SRM's were prepared by a two-step process in which suspensions of quartz and clay were successively deposited on the face of a membrane filter. The procedure used is essentially that described in NBSIR 73-400 (see below), except that deposition was done in two steps. This enabled evaluation of the homogeneity by x-ray fluorescence (XRF) measurement of the quartz present, before deposition of the clay. Because the XRF technique measures any silicon, it was essential that quartz be the only silicon species present.

The filter on which the materials are deposited is a mixed cellulose ester membrane filter. The quartz used was a commercial material known as "Min-u-sil 15" with an average particle size of 4.5 μm. The clay consisted of NBS SRM 97a, Flint Clay. The materials were suspended in a 0.5 percent aqueous solution of gum tragacanth to retard settling during the pipetting procedure. The suspensions were transferred to the filters, using calibrated pipettes. The filters were sequentially numbered during preparation.

Twenty filters were randomly selected from the production lot for x-ray fluorescence measurement. The samples were measured with a vacuum x-ray, wavelength-dispersive spectrometer. The intensity of the Si Kα x-ray was measured for 100 seconds. At least three replicate measurements were made on each sample and on a blank membrane filter.

Chemical analyses were made on samples selected to represent the entire range of values found by XRF measurements. These consisted of 5 for Filter A, 10 for Filter B, 10 for Filter C, and 21 for Filter D.

The amount of quartz deposited on the filters was determined by the method described in NBSIR 73-400, except that the phosphoric acid separation was omitted. This was possible because the measurements were made prior to deposition of the clay.

The XRF and spectrophotometric analyses were made on the same samples, thus enabling a correlation analysis to be made to identify any systematic errors in preparation of the samples. No correlation was found, i.e., the high and low measurements by each technique were not correlated. Accordingly, it was concluded that the variation in the determinations was largely due to measurement error, rather than differences in composition of the SRM's.

Several filters, containing both quartz and clay, were analyzed spectrophotometrically after phosphoric acid separation with the following results:

Sample	μg SiO_2, certified	μg SiO_2, found	No. of determinations	S.D.
A	3.8	1.7	4	1.0
B	29.9	27.3	4	1.2
C	76.1	72.8	5	3.1
D	193.2	176.3	5	5.0

Although reported for information only, these data indicate an SiO_2 loss of approximately 7 percent as a result of the phosphoric acid separation step.

Reference

NBSIR 73-400 "Preparation of Simulated Environmental Filters Containing Quartz and Clay," J. K. Taylor, E. R. Deardorff, C. D. Olson, and R. W. Burke, National Bureau of Standards, Washington, D.C. 20234.