Department of Commerce
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Secretary

National Bureau of Standards
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National Bureau of Standards

Certificate of Analysis

Standard Reference Material 2673

Sulfate and Nitrate on Filter Media

This Standard Reference Material is intended for use in the evaluation of apparatus and methods used in the determination of atmospheric particulate sulfate and nitrate which have been collected on filters. It consists of a series of filter strips upon which sulfate and nitrate have been deposited in an essentially central location. The values certified correspond to the quantities of the substances leached from the filter strip without destruction of the filter matrix.

Sample Number	Sulfate content μg/filter		Nitrate content μg/filter	
	Average value	Tolerance limits	Average value	Tolerance limits
I	503	493 - 513	100	98 - 102
II	2002	1955 - 2049	1002	978 - 1026
III	6939	6635 - 7243	2513	2404 - 2622
Blank	2	0 - 6*	2	0 - 3*

* Range of measured values

The average value is the mean based on the analysis of 24 or 28 filters randomly selected from the lot. The tolerance limits are determined so that at the 95% confidence level they will contain the central 95% of the population of filter values. Details of the preparation, analysis, and statistical treatment of the data are given on the reverse side of the certificate.

This Standard Reference Material was prepared by B. I. Diamondstone. The analytical measurements were made by W. F. Koch. Statistical analysis of the data was provided by J. Orban. The overall direction and coordination of the preparation and analytical measurements leading to certification were performed in the Center for Analytical Chemistry under the chairmanship of J. K. Taylor.

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

Washington, D.C. 20234
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George A. Uriano, Chief
Office of Standard Reference Materials

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Preparation

This SRM consists of strips cut from glass fiber filters, such as are commonly used for measurements of atmospheric particulates, using high volume samplers. Solutions containing known amounts of potassium sulfate and potassium nitrate were prepared, gravimetrically, and multiple aliquots of 25 μL were placed on the filter strips. Four aliquots were so transferred to each filter in the case of samples I and II, and five in the case of Sample III. The pipets were calibrated by weighing similar aliquots transferred into weighing bottles.

The filters were prepared in a clean room and allowed to air-dry before packaging in glassine envelopes. The filters were prepared in groups of 100 and the proper number of aliquots were dispensed into weighing bottles at the beginning and end of each sequence, to monitor the performance of the pipet.

Analytical Measurements

Twenty four samples were randomly selected from the production lot (28 in the case of sample III) for chemical measurement of their extractable sulfate and nitrate. Each filter strip was extracted with a standard eluent (see below) for 15 minutes in a 55 °C ultrasonic bath. The extracts were analyzed for sulfate and nitrate by ion chromatography. The eluent was a solution of 0.003 M $NaHCO_3$ and 0.0018 M Na_2CO_3. A 100 μL sample loop was used. Peak heights were compared with those obtained from accurately prepared standard solutions. Three standards were prepared for each concentration level so as to bracket the extracts.

The randomly selected filters were also analyzed in random order. No significant systematic errors were observed related to the order of preparation. Average values and the overall standard deviation, s_0, of individual measurements were calculated. This standard deviation includes measurement error and filter content error.

The standard deviation, s_f, due to filter content variability was computed from a sample of 12 or 14 weighed quantities delivered by the pipets during the preparation of the filters. The resulting tolerance interval for the sulfate/nitrate contents is of the form

$$\overline{X} \pm k\, s_f\ .$$

It is this tolerance interval that should be of greater interest to the participating laboratory since it gives practical bounds for the likely values of the sulfate or nitrate contents that might be found in a given filter.

The following table lists the values of s_0 and s_f for each set of filters. The measurement standard deviation, s_m, for NBS can be computed using the relation $s_0^2 = s_m^2 + s_f^2$

Set	Average (μg)	s_o	s_f
I SO_4	502.6	8.89	3.14
NO_3	100.3	2.56	.63
II SO_4	2001.7	29.30	14.88
NO_3	1001.7	15.92	7.44
III SO_4	6939.0	109.35	100.75
NO_3	2513.0	43.70	36.30

The average values calculated from the composition of the solutions and the quantitites delivered by the pipets are in general agreement with the analytical values. However, the values certified are those obtained by analysis.

Recommended Usage

The material is not homogeneously distributed on the filter; hence the sample must be used in its entirety for analysis. It is recommended that the filter be extracted at 55 °C in an ultrasonic bath, with water or other nonreactive solvents. The filter base should not be digested to put it into solution.