U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
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National Bureau of Standards

Certificate of Analysis

Standard Reference Material 2674

Lead on Filter Media

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

Sample No.	Lead Content, μg/filter	
	Average Value	Tolerance Limits
I	100	97 - 103
II	303	294 - 312
III	1505	1477 - 1533
Blank	1.4	0.7 - 2.1*

*Range of measured values

The average value is the mean based on the analysis of 26 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 this certificate.

This Standard Reference Material was prepared by B. I. Diamondstone. The analytical measurements were made by E. J. Maienthal and M. S. Epstein of the Inorganic Analytical Research Division. Statistical analysis of the data was provided by J. Orban of the Statistical Engineering Division.

The overall direction and coordination of the preparation and analytical measurements leading to certification were performed in the Center for Analytical Chemistry under the charimanship 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 T. E. Gills.

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 of the type normally used in high volume samplers for the measurement of atmospheric particulates. Solutions containing known amounts of lead nitrate were prepared gravimetrically, and aliquots of 25 μL were placed on the filter strips using micropipets. Four aliquots were 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 into 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-eight filters were randomly selected from the production lot (26 in the case of Sample I) for chemical measurement of extractable lead. Selected filters were extracted with dilute nitric acid, using ultrasonic vibration to assist in the extraction. After the nitric acid extraction, the filters were thoroughly rinsed with distilled water to ensure complete extraction. The extracts were quantitatively diluted and analyzed by linear sweep voltammetry which had been calibrated with solution standards prepared from high purity lead.

The selected filters were analyzed in random order. No significant systematic errors were observed related to order of preparation. Average values were calculated together with the overall standard deviation, s_o, of individual measurements. This standard deviation includes measurement error and filter content error. The resulting confidence interval for the mean is of the form

$$\overline{X} \pm t\, s_o/\sqrt{n},$$

where s_o is based on $n - 1 = 27$ degrees of freedom.

A selected number of filters were also analyzed by atomic absorption. The results obtained were well within the tolerance limits given and were consistent with the certified values.

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 lead content is of the form

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

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

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

Set	Average (μg)	s_o	s_f
I	99.7	1.59	0.89
II	302.9	5.37	3.00
III	1505.2	12.73	9.24

The average values calculated from the composition of the solutions and the quantities delivered by the pipets are in general agreement with the analytical values. However, the values certified are those obtained by the 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 45-50 °C in an ultrasonic bath, with dilute nitric acid (2. mL HNO_3 + 15 mL H_2O). After extraction the filters should be thoroughly rinsed with distilled water. The filter base should not be dissolved.