U.S. Department of Commerce
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# National Bureau of Standards

# Certificate of Analysis

# Standard Reference Materials 2678, 2680, and 2681

## Trace Constituent Elements in Blank Filters

These Standard Reference Materials (SRM's) are intended primarily for use in evaluating the performance of air sampling filter methods wherein dust, particulates, aerosols, mists, etc. are collected by passing a known volume of air through a filter to remove the chemical hazard. SRM's 2678, 2680, and 2681 are blank filters that have been analyzed at NBS using technique and methods generally employed in the NBS certification procedures. Thirty-one constituent elements, including some listed as Environmental Protection Agency (EPA) priority pollutants, were determined and or analyzed in the three different filter types or sizes that are commonly used in air sampling of industrial atmospheres. For information only, the filter types and descriptions are listed below.

Blank Filters

| SRM | Description | Pore Size | Diameter | Nominal Filter, wt., g |
|---|---|---|---|---|
| 2678 | Cellulose Acetate Membrane | 0.45 micron | 47 mm | 0.09 |
| 2680 | Cellulose Acetate Membrane | 0.80 micron | 37 mm | 0.05 |
| 2681 | Ashless | ---- | 42.5 mm | 0.14 |

The filters were analyzed for constituent elements using instrumental neutron activation analysis (INAA) and for leachable cations and anions using ion chromatography (IC). The leachable constituent elements were brought into solution using a standardized procedure (see Ion Chromatography under Analysis Section).

The certified values presented as tolerance intervals for these SRM's are given in Tables 1 and 2. Two-sided tolerance intervals for the constituent elements are given whenever possible and one-sided tolerance intervals are given for the constituent elements for which the data would not support two-sided intervals. In cases where a constituent element was not detected, a limit of detection ($X_D$) is given based on conditions and sensitivity of the technique employed. (See Definitions.)

The statistical analysis of the data for these SRM's was performed by S.B. Schiller and K.R. Eberhardt of the Statistical Engineering Division.

Analyses for certification were performed in the Inorganic Analytical Research Division by L.A. Holland, W.F. Koch, K.W. Pratt, S.F. Stone, and R. Zeisler.

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 T.E. Gills.

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Gaithersburg, MD 20899

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Notice and Recommended Use: These SRM's are commercial filters that were not treated or washed before analysis. The variation in concentration levels of the constituent elements may or may not be typical of the variability found in similar commercial filters, as production and treatment processes do differ. However, NBS has characterized each of the three SRM lots to give the user a stated confidence level for the range of concentration levels of constituent elements. Thus the user can use the filters to accurately assess the magnitude of blank levels of the air in the workplace or industrial environment.

Analysis

Instrumental Neutron Activation Analysis (INAA): The filters were pelletized using a cleaned pellet press, and packaged in acid-cleaned linear polyethylene film. The analysis procedure used was the general INAA scheme, using a comparator technique with appropriate standards and control samples [1].

Ion Chromatography: The filters were analyzed for soluble chloride, fluoride, nitrate, sulfate, sodium, and potassium using ion chromatography. Individual filters were covered in polyethylene vessels using distilled, deionized water. The samples were then placed in an ultrasonic bath for periods of 15 minutes (SRM's 2678, 2680) and for 30 minutes (SRM 2681) to assure complete dissolution of the soluble species. Shorter times less than those listed above yielded low, variable results.

The cations and anions were determined simultaneously using a two channel ion chromatograph and ion chromatography software for peak integration.

[1] Greenberg, R.R., Fleming, R.F., and Zeisler, R., "High Sensitivity Neutron Activation Analysis of Environmental and Biological Standard Reference Materials," Environ. Intern., 10, 129-136, (1984).

Table 1
Certified Values for Blank Filters

| Element | SRM 2678 | SRM 2680 | SRM 2681 |
|---|---|---|---|
| Ag (μg) | $X_D$: < 0.015 | $X_D$: < 0.004 | $X_D$: < 0.012 |
| Al (μg) | 0.03-0.42 | 0-0.18 | 0.10-0.86 |
| As (μg) | $X_D$: < 0.0006 | $X_D$: < 0.0005 | 0-0.001 |
| Au (ng) | 0-0.32 | 0.005-0.075 | 0.008-0.95 |
| Ba (μg) | $X_D$: < 0.2 | $X_D$: < 0.07 | $X_D$: < 0.16 |
| Ca (μg) | 1.2-2.4 | 1.9-4.1 | 1.1-3.5 |
| Cd (μg) | $X_D$: < 0.006 | 0-0.002 | $X_D$: < 0.005 |
| Ce (μg) | $X_D$: < 0.004 | $X_D$: < 0.001 | $X_D$: < 0.003 |
| Cl (μg) | 10-14 | 7-13 | 7-12 |
| Co (μg) | 0-0.003 | 0-0.009 | $X_D$: < 0.002 |
| Cr (μg) | 0.06-0.22 | 0.03-0.23 | 0.03-0.66 |
| Cs (μg) | $X_D$: < 0.002 | $X_D$: < 0.0007 | $X_D$: < 0.002 |
| Cu (μg) | 0-0.09 | 0.15-0.36 | 0-0.08 |
| Fe (μg) | 0-1.3 | 0-0.53 | 0-1.0 |
| I (μg) | 0.004-0.008 | 0.006-0.013 | 0-0.004 |
| K (μg) | 0-2.2 | 0-1.3 | $X_D$: < 0.8 |
| La (μg) | 0-0.0006 | 0-0.0007 | 0-0.0008 |
| Mg (μg) | 0.8-2.1 | 1.6-3.0 | 0-1.2 |
| Mn (μg) | 0.005-0.02 | 0.002-0.011 | 0.003-0.016 |
| Mo (μg) | $X_D$: < 0.005 | 0-0.003 | $X_D$: < 0.004 |
| Na (μg) | 8.2-13.2 | 3.2-4.8 | 5.2-7.1 |
| Rb (μg) | $X_D$: < 0.05 | $X_D$: < 0.016 | $X_D$: < 0.04 |
| Sb (μg) | 0.0004-0.001 | 0.0003-0.0008 | 0.0001-0.008 |
| Sc (μg) | $X_D$: < 0.0002 | 0-0.0004 | 0-0.06 |
| Se (μg) | $X_D$: < 0.010 | $X_D$: < 0.003 | $X_D$: < 0.008 |
| Sm (ng) | 0-0.002 | 0-0.009 | 0-0.08 |
| Sn (μg) | $X_D$: < 0.8 | $X_D$: < 0.3 | $X_D$: < 0.6 |
| Ti (μg) | 0-0.10 | 0-0.065 | 0-0.21 |
| V (μg) | 0-0.0007 | 0.0002-0.001 | 0-0.001 |
| Zn (μg) | 0-1.1 | 0-0.04 | 0-0.08 |

Table 2

Certified Values for Leachable Anions and Cations

| Constituent | SRM 2678 | SRM 2680 | SRM 2681 |
|---|---|---|---|
| $Cl^-$ (μg) | 4.5 - 7.9 | 3.9 -10.4 | 4.3 - 7.7 |
| $F^-$ (μg) | $X_D$: <1 | $X_D$: <1 | $X_D$: <1 |
| $K^+$ (μg) | 0.33-0.80 | 0.37-0.69 | $X_D$: <1 |
| $Na^+$ (μg) | 7.3 - 12.1 | 3.0 - 4.7 | 2.2 - 3.6 |
| $NO_3^-$ (μg) | 4.7 - 8.5 | $X_D$: <10 | 0.82 - 4.7 |
| $SO_4^=$ (μg) | $X_D$: <3 | $X_D$: <1.2 | $X_D$: <1.5 |

Definitions:

Limit of detection ($X_D$): The $X_D$ refers to the underlying true analyte concentration that the employed chemical measurement process is capable of detecting. [Ref: "Detection in Analytical Chemistry - Importance, Theory, and Practice," ACS Symposium Series 361, pp 10, Lloyd A. Currie, Editor, 1988.]

Two-sided Tolerance Interval: A two-sided tolerance interval was computed for those constituent elements in which no "less than" results were reported. The computed two-sided tolerance intervals are constructed, at the 90% confidence level, to cover the analyte concentrations of 90% of the population of filters. These intervals were computed as $X \pm (2.077)s$ for $n=25$ and $X \pm (2.065)s$ for $n=26$, after the data were first converted to logarithmic scale.

One-sided Tolerance Interval (interval from 0 to some limit): For elements that had one or more "less than" values in the measurement data, a one-sided upper tolerance interval was computed using a nonparametric method. [Ref.: Natrella, Mary G., Experimental Statistics, National Bureau of Standards Handbook 91, U.S. Government Printing Office, 1966, page 2-15.] These intervals, which extend from 0 to the maximum observed concentration, are also constructed to cover 90% of the population of filters at a confidence level of 90%.