

**Aluminum Oxide in Workplace Atmospheres**

Method no.:	ID-198SG
Matrix:	Air
OSHA Standard:	10.0 mg/m <sup>3</sup>
Collection Procedure:	A known volume of air is drawn through a 0.80 µm AA filter.
Recommended Air Volume:	960 liters maximum
Recommended Sampling Rate:	2.0 liters per minute
Analytical Procedure:	The filter is digested with acids using a microwave and is analyzed by Atomic Absorption Spectroscopy (AAS).
Quantitative Detection Limit:	0.025 ppm
Method Classification:	PV

Withdrawn  
Provided for Historical Reference only

1. Introduction

This method describes the collection on a 0.80 m AA filter and analysis using Atomic Absorption Spectroscopy of Aluminum Oxide (Al<sub>2</sub>O<sub>3</sub>). The method measures the total concentration of Al<sub>2</sub>O<sub>3</sub> as aluminum.

1.1. History

Originally, aluminum oxide samples were collected on an AA filter, digested by acid digestion, and analyzed by AAS. This procedure was abandoned when it was found to give low recoveries. Prior to the use of this method, Al<sub>2</sub>O<sub>3</sub> was collected on PVC filters, weighted, and sent to the lab only if the sample weight allowed the sample to be over the standard. The filters were digested using a borate fusion and analyzed by AAS. There is no significant difference between the means of the found/theoretical values of the microwave and the borate fusion digestions at a 95% confidence level (See [Appendix I](#)).

1.2. Uses (9.1.)

About 90% of aluminum oxide is used in the production of aluminum metal. The remainder is used in applications such as flame retardant filters, preparation of aluminum compounds, pigments, absorbents, catalysts, ceramics, refractories, and abrasives.

1.3. Physical Properties (9.2.)

Physical Characteristics:	White powder, insoluble in water
Specific Gravity:	3.4 - 4.0
Melting Point:	2030°C

2. Working Range and Detection Limit

- 2.1. The upper limit of sample analysis is based on the upper limit of linearity of the atomic absorption analysis of aluminum, which is 50 µg/mL (50 ppm). (9.3.)
- 2.2. The lower limit of sample analysis is based on the detection limit of 0.05 µg/mL. For a 50 mL sample volume and a 100-liter air volume, the lower limit is 0.025 mg/m<sup>3</sup>.

3. Interferences

Withdrawn  
Provided for Historical Reference only

- 3.1. Aluminum and salts of aluminum can cause an interference. If aluminum or its compounds are present, they must be reported to the laboratory.
4. Advantages and Disadvantages
  - 4.1. The method is less tedious than the borate fusion method.
  - 4.2. The sampling procedure uses AA filters as opposed to PVC filters and the weighing step is not necessary.
5. Sampling Procedure
  - 5.1. Apparatus - 0.80  $\mu\text{m}$  AA filters, personal sampling pumps which are calibrated at the recommended flow rate with an AA filter in line to an accuracy of  $\pm 10\%$  at the 95% confidence limit.
  - 5.2. The AA filter is attached to a calibrated personal sampling pump and the sampling tube is placed in the worker's breathing zone or in a area of, the workplace. At least 100 liters and no more than 960 liters of air should be drawn through the sampling tube at a rate of 2.0 liters/minute. (9.5.)
  - 5.3. After the desired sampling period is completed, the filter is removed from the pump. The filter is properly identified and sealed with an OSHA Form 21, and then shipped to the laboratory for analysis.
  - 5.4. With each batch of up to 20 samples, a blank filter which has had no air drawn through it, is submitted for analysis. The blank filter should be from the same lot of filters as those used for sampling.
  - 5.5. It is very important that when aluminum or salts of aluminum are known to be present in the workplace atmosphere, they should be listed.
6. Analytical Procedure
  - 6.1. Apparatus - Atomic Absorption Spectrophotometer and appropriate volumetric glassware for dilutions and standard preparation.
  - 6.2. Reagents - All reagents used should be ACS analyzed reagent grade or better.
    - 6.2.1. Deionized water with a specific conductance of 10  $\mu\text{mho/cm}$  or less for preparation of standards, samples, dilutions, and other solutions which will be used in the analysis.
    - 6.2.2. Potassium Chloride (KCl).
    - 6.2.3. Potassium Chloride, 5000 ppm solution. Add 9.54 grams of potassium chloride to a 1000 mL volumetric flask and dilute to volume with deionized water.
    - 6.2.4. Acid Digestion Solution. Make the acid digestion solution by adding Sulfuric, Phosphoric, and Nitric Acids in a 2:2:1 ratio.
    - 6.2.5. Aluminum Stock Standard (1000  $\mu\text{g/mL CH}_3\text{COO}^-$ ). Use 1000 ppm Aluminum Stock Standard. Aluminum working standards are made by diluting the stock solution with deionized water and by adding enough KCl solution to prepare the standards in 1000 ppm KCl.
  - 6.3. Safety Precautions

Withdrawn  
Provided for Historical Reference only

- 6.3.1. Care should be exercised when using laboratory glassware. Chipped pipettes, volumetric flasks, beakers, or any glassware with sharp edges exposed should not be used to avoid the possibility of cuts, abrasions, and lost samples.
- 6.3.2. Pipetting should never be done by mouth - a bulb should *a/ways* be used.

7. Standard Preparation

- 7.1. Working standards are prepared in the analytical range of 0.5 µg/mL to 50 µg/mL from dilutions of the 1000 µg/mL stock solutions. These standard solutions should be prepared in 1000 ppm KCl.

8. Sample Preparation

- 8.1. If the air volume is greater than or equal to 100 liters, moisten the filter with deionized water and place the filter in the bottom of the Teflon microwave vessel. Pipette 5 mL of acid solution into each vessel, and cap using the capping station. If the air volume is less than 100 liters use 2.5 mL acid solution.
- 8.2. Place the reaction vessels, with tubes, into the microwave rack and close the door. Set the time to 2 minutes per vessel. For example, if you use all 12 vessels, set the time to 24 minutes. Next set the power to 50%. Start the microwave. After the digestion is over let the samples cool for about an hour.
- 8.3. Uncap the vessels using the capping station and pour the digested solutions into 50 mL volumetric flasks. Rinse the vessels several times with deionized water and add the rinse solutions to the volumetric flasks. Add 10 mL of the 5000 ppm KCl solution. If the air volume is less than 100 liters, pour the digested solutions into 25 mL volumetric flasks and add 5 mL of the 5000 ppm KCl solution.
- 8.4. For general microwave instructions, refer to the Microwave Bulk and Wipe Digestion Procedure.

9. Analysis (9.4.)

- 9.1. For general instrument start up and operation, refer to the Atomic Absorption Standard Operating Procedure.

- 9.1.1. The normal instrument parameters are:

Flame:	N <sub>2</sub> O <sub>2</sub> /C <sub>2</sub> H <sub>2</sub>
Wavelength:	309 nm
Slit:	4
Range:	UV

9.2. Calculations

- 9.2.1. Absorbance standards are used to construct a standard curve using the OSHA Auto AA Program. The sample results are obtained from a plot of absorbance vs. concentration. The blank corrected sample

Withdrawn  
Provided for Historical Reference only

values are then calculated using the Auto AA Program. Air Concentration values are calculated by the computer, using a gravimetric factor of 1.8989.

- 9.3. Reporting Results for Compounds Determined by Atomic Absorption
- 9.3.1. Results are reported on the OSHA Form 91 in mg/m<sup>3</sup> for Al<sub>2</sub>O<sub>3</sub>, using two significant figures.
  - 9.3.2. The estimated detection limit calculated by the Auto AA Program is reported on the OSHA Form 91 when no analyte is detected.
  - 9.3.3. All data processor printouts and chart-recorded chromatograms are filed in a central file according to laboratory sample identification.
  - 9.3.4. Calculations are checked by a fellow chemist before the completed OSHA Form 91's are given to the supervisor.

10. References

- 10.1. *Encyclopedia of Chemical Technology*, Third Edition, 1978, Vol. 2, pg. 218.
- 10.2. *The Condensed Chemical Dictionary*, Tenth Edition, 1981, pg. 42.
- 10.3. *Analytical Methods for Atomic Absorption Spectrophotometry*, Perkin-Elmer Corporation, 1975.
- 10.4. OSHA Sampling and Analytical Techniques Table, Industrial Hygienists Field Operations Manual, OSHA Instruction CPL 2-2.20, April 2, 1979.

This Method Was Last Revised On August 11, 1988.

Withdrawn  
 Provided for Historical Reference only

APPENDIX I

COMPARISON OF MICROWAVE DIGESTION WITH BORATE FUSION

Microwave Digestion found/theoretical		Borate Fusion found/theoretical
0.93		1.10
0.97		1.08
0.85		0.88
0.93		0.97
0.89		0.78
0.87		1.04
0.86		0.84
0.86		0.70
0.70		0.74
0.89		0.79
0.58		0.52
0.59		0.58
0.83	Mean	0.835
0.125	Standard Deviation	0.180
12	n	12

Degrees of Freedom = 22, alpha = 0.05, Critical Region = 2.074, t = 0.079

Using the t-test, it was found that there is no significant difference between the sample means at the 95% confidence level.