METHOD 2

REF: Regs: 11-3-301 to 11-3-303

DETERMINATION OF BERYLLIUM IN EFFLUENT AND ATMOSPHERIC PARTICULATE MATTER

1) PRINCIPLE

Samples are collected by passing a known volume of air through an appropriate filter. The filter is decomposed by wet acid oxidation. The aqueous beryllium solution is then analyzed by atomic absorption spectrophotometry.

2) APPARATUS

2.1 Atomic Absorption Spectrophotometer

2.1.1 Acetylene

2.1.2 Nitrous Oxide

2.1.3 Beryllium Hollow Cathode Lamp

2.2 Hot Plate

3) REAGENTS

3.1 Concentrated Nitric Acid

3.2 Concentrated Sulfuric Acid

3.3 Concentrated Perchloric Acid

3.4 Hydrochloric Acid 25% (v/v). Dilute 250 ml of concentrated hydrochloric acid to 1 liter with distilled water.

3.5 Sulfuric Acid (12 N). Slowly add 33.6 ml concentrated sulfuric acid to 50 ml of distilled water, cool and dilute to 100 ml.

3.6 Beryllium Powder (98% minimum purity).
4) **ANALYTICAL PROCEDURE**

4.1 Place the sample filter in a 150 ml beaker. Add 35 ml concentrated nitric acid. Heat on a hot plate until light brown fumes are evident, thus destroying all the organic matter. Cool to room temperature; add 5 ml of concentrated sulfuric acid and 5 ml of concentrated perchloric acid.

4.2 Replace the beaker on the hot plate and evaporate to dryness in a perchloric acid hood. Cool and dissolve the residue by pipetting 10 ml of 25% v/v hydrochloric acid into the beaker. The sample is now ready for analysis.

4.3 Prepare and analyze a blank filter in the same manner as the samples.

4.4 Set the spectrophotometer parameters to obtain maximum sensitivity at a wavelength of 234.8 nm by use of beryllium standards, and manipulation of the acetylene and nitrous oxide gas flows. Use 25% v/v hydrochloric acid to set the instrument at 0% absorption.

4.4.1 Aspirate each sample into the flame and record the percent absorption. Convert the readings to absorbance units.

4.4.2 Samples that have percent absorption values greater than the range of the standard calibration curve of the instrument must be diluted with 25% v/v hydrochloric acid to be within the working range.

4.4.3 Determine the beryllium concentration of the sample from the beryllium standard curve.

4.4.4 Standards should be aspirated after every fourth or fifth sample to insure instrument response has not changed.

5) **STANDARD AND CALIBRATION CURVE**

5.1 **Standard Stock Beryllium Solution.** Dissolve 0.1000 g of beryllium powder in 80 ml of 12 N sulfuric acid and dilute to 1 liter with 25% v/v hydrochloric acid. This solution contains 100 µgm Be/ml.

5.2 **Working Standard Beryllium Solution.** Dilute 5.0 ml of the standard stock beryllium solution to 100.0 ml with 25% v/v hydrochloric acid to give a concentration of 5 µgm Be/ml. At least 3 individual standards normally in the range of 0 - 5 µgm Be/ml are freshly prepared by appropriate dilution of the working standard solution. The working standard solution should be freshly prepared prior to use.
5.3 Prepare a standard curve by plotting absorbance vs. concentration of beryllium on linear graph paper.

6) **CALCULATION**

6.1 A blank filter background beryllium value, if any, is subtracted from each sample.

\[ *\mu\text{g m Be/m}^3 = \frac{\mu\text{g m Be from std curve x 10}}{\text{Air Sample Volume (m}^3) \} \]

*If sample was diluted to be within the standard curve range (4.4.2), the calculation must be multiplied by the appropriate dilution factor.*

6.2 Total \( \mu\text{g m Be} = \mu\text{g m Be from standard curve x 10.} \)

7) **REFERENCE**