# 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 wave length 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 $\mu$gm 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 $\mu$gm Be/ml. At least 3 individual standards normally in the range of  0 - 5 $\mu$gm 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 substracted from each sample.

$$*\mu gm\ Be/m^3\ =\ \frac{\mu gm\ Be\ from\ std\ curve\ x\ 10}{Air\ Sample\ Volume\ (m^3)}$$

*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 $\mu gm$ Be  =  $\mu gm$ Be from standard curve x 10.**

# 7)    REFERENCE

**7.1**    40  CFR 61 Appendix B, Method 104.  **"Reference Method for Determination of Beryllium Emissions from Stationary Sources".**