METHOD 103 - BERYLLIUM SCREENING METHOD

1.0 Scope and Application.

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Beryllium (Be)	7440-41-7	Dependent upon analytical procedure used

1.2 Applicability. This procedure details guidelines and requirements for methods acceptable for use in determining Be emissions in ducts or stacks at stationary sources.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method.

2.1 Particulate Be emissions are withdrawn isokinetically from three points in a duct or stack and are collected on a filter. The collected sample is analyzed for Be using an appropriate technique.

3.0 Definitions. [Reserved]

4.0 Interferences. [Reserved]

5.0 Safety.

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its

use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Hydrochloric Acid (HCl). Highly corrosive and toxic. Vapors are highly irritating to eyes, skin, nose, and lungs, causing severe damage. May cause bronchitis, pneumonia, or edema of lungs. Exposure to concentrations of 0.13 to 0.2 percent can be lethal to humans in a few minutes. Provide ventilation to limit exposure. Reacts with metals, producing hydrogen gas. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burn as thermal burn.

6.0 Equipment and Supplies.

6.1 Sample Collection. A schematic of the required sampling train configuration is shown in Figure 103-1 in Section 17.0. The essential components of the train are as follows:

6.1.1 Nozzle. Stainless steel, or equivalent, with sharp, tapered leading edge.

6.1.2 Probe. Sheathed borosilicate or quartz glass tubing.

6.1.3 Filter. Millipore AA, or equivalent, with appropriate filter holder that provides a positive seal against leakage from outside or around the filter. It is suggested that a Whatman 41, or equivalent, be placed immediately against the back side of the Millipore filter as a guard against breakage of the Millipore. Include the backup filter in the analysis. To be equivalent, other filters shall exhibit at least 99.95 percent efficiency (0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles, and be amenable to the Be analysis procedure. The filter efficiency tests shall be conducted in accordance with ASTM D 2986-71, 78, 95a (incorporated by reference **)** see §61.18). Test data from the supplier's quality control program are sufficient for this purpose.

6.1.4 Meter-Pump System. Any system that will maintain isokinetic sampling rate, determine sample volume, and is capable of a sampling rate of greater than 14 lpm (0.5 cfm).

6.2 Measurement of Stack Conditions. The following equipment is used to measure stack conditions:

6.2.1 Pitot Tube. Type S, or equivalent, with a constant coefficient (±5 percent) over the working range.

6.2.2   Inclined Manometer, or Equivalent.   To measure velocity head to ±10 percent of the minimum value.

6.2.3   Temperature Measuring Device.   To measure stack temperature to ±1.5 percent of the minimum absolute stack temperature.

6.2.4   Pressure Measuring Device.   To measure stack pressure to ±2.5 mm Hg (0.1 in. Hg).

6.2.5   Barometer.   To measure atmospheric pressure to ±2.5 mm Hg (0.1 in. Hg).

6.2.6   Wet and Dry Bulb Thermometers, Drying Tubes, Condensers, or Equivalent.   To determine stack gas moisture content to ±1 percent.

6.3   Sample Recovery.

6.3.1   Probe Cleaning Equipment.   Probe brush or cleaning rod at least as long as probe, or equivalent. Clean cotton balls, or equivalent, should be used with the rod.

6.3.2   Leakless Glass Sample Bottles.   To contain sample.

6.4   Analysis.   All equipment necessary to perform an atomic absorption, spectrographic, fluorometric, chromatographic, or equivalent analysis.

7.0   Reagents and Standards.

7.1   Sample Recovery.

7.1.1 Water. Deionized distilled, to conform to ASTM D 1193-77, 91 (incorporated by reference - see §61.18), Type 3.

7.1.2 Acetone. Reagent grade.

7.1.3 Wash Acid, 50 Percent (V/V) Hydrochloric Acid (HCl). Mix equal volumes of concentrated HCl and water, being careful to add the acid slowly to the water.

7.2 Analysis. Reagents and standards as necessary for the selected analytical procedure.

8.0 Sample Collection, Preservation, Transport, and Storage.

Guidelines for source testing are detailed in the following sections. These guidelines are generally applicable; however, most sample sites differ to some degree and temporary alterations such as stack extensions or expansions often are required to insure the best possible sample site. Further, since Be is hazardous, care should be taken to minimize exposure. Finally, since the total quantity of Be to be collected is quite small, the test must be carefully conducted to prevent contamination or loss of sample.

8.1 Selection of a Sampling Site and Number of Sample Runs. Select a suitable sample site that is as close as practicable to the point of atmospheric emission. If

possible, stacks smaller than one foot in diameter should not be sampled.

8.1.1   Ideal Sampling Site.   The ideal sampling site is at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion or contraction.   For rectangular cross sections, use Equation 103-1 in Section 12.2 to determine an equivalent diameter, D_e.

8.1.2   Alternate Sampling Site.   Some sampling situations may render the above sampling site criteria impractical.   In such cases, select an alternate site no less than two diameters downstream and one-half diameter upstream from any point of flow disturbance.   Additional sample runs are recommended at any sample site not meeting the criteria of Section 8.1.1.

8.1.3   Number of Sample Runs Per Test.   Three sample runs constitute a test.   Conduct each run at one of three different points.   Select three points that proportionately divide the diameter, or are located at 25, 50, and 75 percent of the diameter from the inside wall.   For horizontal ducts, sample on a vertical line through the centroid.   For rectangular ducts, sample on a line through the centroid and parallel to a side.   If additional sample runs are performed per Section 8.1.2, proportionately divide the duct to accommodate the total number of runs.

8.2 Measurement of Stack Conditions. Using the
equipment described in Section 6.2, measure the stack gas
pressure, moisture, and temperature to determine the
molecular weight of the stack gas. Sound engineering
estimates may be made in lieu of direct measurements.
Describe the basis for such estimates in the test report.

8.3 Preparation of Sampling Train.

8.3.1 Assemble the sampling train as shown in Figure
103-1. It is recommended that all glassware be precleaned
by soaking in wash acid for two hours.

8.3.2 Leak check the sampling train at the sampling
site. The leakage rate should not be in excess of 1 percent
of the desired sample rate.

8.4 Sampling Train Operation.

8.4.1 For each run, measure the velocity at the
selected sampling point. Determine the isokinetic sampling
rate. Record the velocity head and the required sampling
rate. Place the nozzle at the sampling point with the tip
pointing directly into the gas stream. Immediately start
the pump and adjust the flow to isokinetic conditions. At
the conclusion of the test, record the sampling rate. Again
measure the velocity head at the sampling point. The
required isokinetic rate at the end of the period should not
have deviated more than 20 percent from that originally

calculated. Describe the reason for any deviation beyond 20 percent in the test report.

8.4.2 Sample at a minimum rate of 14 liters/min (0.5 cfm). Obtain samples over such a period or periods of time as are necessary to determine the maximum emissions which would occur in a 24-hour period. In the case of cyclic operations, perform sufficient sample runs so as to allow determination or calculation of the emissions that occur over the duration of the cycle. A minimum sampling time of two hours per run is recommended.

8.5 Sample Recovery.

8.5.1 It is recommended that all glassware be precleaned as in Section 8.3. Sample recovery should also be performed in an area free of possible Be contamination. When the sampling train is moved, exercise care to prevent breakage and contamination. Set aside a portion of the acetone used in the sample recovery as a blank for analysis. The total amount of acetone used should be measured for accurate blank correction. Blanks can be eliminated if prior analysis shows negligible amounts.

8.5.2 Remove the filter (and backup filter, if used) and any loose particulate matter from filter holder, and place in a container.

8.5.3 Clean the probe with acetone and a brush or long rod and cotton balls. Wash into the container with the

filter. Wash out the filter holder with acetone, and add to the same container.

9.0 Quality Control. [Reserved]

10.0 Calibration and Standardization.

10.1 Sampling Train. As a procedural check, compare the sampling rate regulation with a dry gas meter, spirometer, rotameter (calibrated for prevailing atmospheric conditions), or equivalent, attached to the nozzle inlet of the complete sampling train.

10.2 Analysis. Perform the analysis standardization as suggested by the manufacturer of the instrument, or the procedures for the analytical method in use.

11.0 Analytical Procedure.

Make the necessary preparation of samples and analyze for Be. Any currently acceptable method (*e.g.*, atomic absorption, spectrographic, fluorometric, chromatographic) may be used.

12.0 Data Analysis and Calculations.

12.1 Nomenclature.

A_s(avg) = Stack area, m^2 (ft^2).

L = Length.

R = Be emission rate, g/day.

V_s(avg) = Average stack gas velocity, m/sec (ft/sec).

V_{total} = Total volume of gas sampled, m^3 (ft^3).

W   = Width.

W_t = Total weight of Be collected, mg.

10^{-6} = Conversion factor, g/µg.

86,400   = Conversion factor, sec/day.

12.2 Calculate the equivalent diameter, De, for a rectangular cross section as follows:

$$D_e = \frac{2 \cdot L \cdot W}{L + W}$$ **Eq. 103-1**

12.3 Calculate the Be emission rate, R, in g/day for each stack using Equation 103-2. For cyclic operations, use only the time per day each stack is in operation. The total Be emission rate from a source is the summation of results from all stacks.

$$R = \frac{W_t V_{s(avg)} A_s (86,400) (10^{-6})}{V_{total}}$$ **Eq. 103-2**

12.4 Test Report. Prepare a test report that includes as a minimum: A detailed description of the sampling train used, results of the procedural check described in Section 10.1 with all data and calculations made, all pertinent data taken during the test, the basis for any estimates made, isokinetic sampling calculations, and emission results. Include a description of the test site, with a block diagram and brief description of the process, location of the sample

points in the stack cross section, and stack dimensions and distances from any point of disturbance.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References. [Reserved]

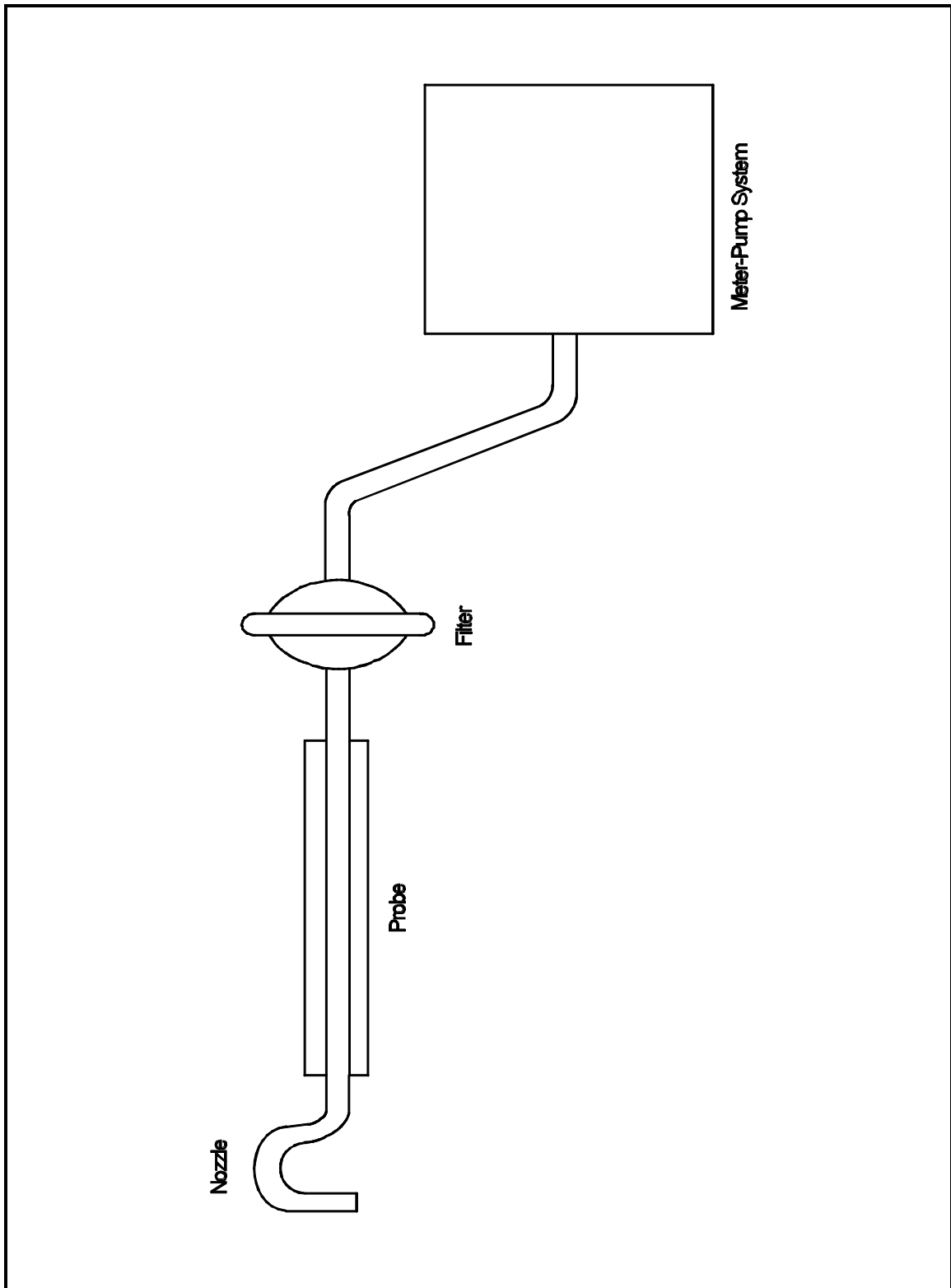*17.0 Tables, Diagrams, Flow Charts, and Validation Data.*

Figure 103-1. Beryllium Screening Method Sampling Train Schematic.

1776