

METHOD 4B

REF: Reg. 10-1-301
11-1-301

DETERMINATION OF TOTAL LEAD IN EFFLUENTS

1) PRINCIPLE

1.1 Lead emissions are isokinetically sampled at the source using a glass wool filter, followed by acidic iodine monochloride impingers. The particulate lead content of the filter is removed by acid digestion and analyzed by atomic absorption spectroscopy. The gaseous lead compounds collected by iodine monochloride are determined by atomic absorption spectroscopy after the absorbing solution has been reduced to iodide.

2) APPARATUS

2.1 **Glassware.** Borosilicate glassware should be used throughout the analysis. The glassware must be cleaned with 10% HNO₃ and rinsed with distilled water prior to use.

2.2 Atomic Absorption Spectrophotometer

2.2.1 Acetylene

2.2.2 Air Supply

2.2.3 Lead Hollow Cathode Lamp

2.3 Hot Plate

2.4 500 ml Phillips Beakers

3) REAGENTS

3.1 **Concentrated Nitric Acid** (69 to 71%)

3.2 **Dilute Nitric Acid.** Dilute 10 ml of concentrated nitric acid to 1 liter with distilled water.

3.3 **Lead Nitrate** (Pb (NO₃)₂)

- 3.4 Iodine Monochloride Stock Solution (1 M).** Dissolve 100 g of potassium iodide in sufficient distilled water to make 400 ml of solution. Add slowly, 400 ml of concentrated hydrochloric acid and cool to room temperature. Add slowly with vigorous stirring, 68 g of potassium iodate. Continue stirring until all the free iodine initially formed has redissolved to yield a clear orange-red solution. Dilute to 1 liter with distilled water and store the solution in a dark bottle.
- 3.5 Iodine Monochloride Scrubber Solution (0.1 M).** Dilute 100 ml of stock iodine monochloride solution to 1 liter with distilled water. This solution should be kept in dark glass bottle to prevent degradation. This reagent should be stable for at least 2 months; however, periodic checks should be performed to insure quality.
- 3.6 Potassium Iodide Solution (25%).** Dissolve 25 g potassium iodide in sufficient distilled water to make 100 ml of solution.
- 3.7 Sodium Sulfite Solution (10%).** Dissolve 10 g of anhydrous sodium sulfite in sufficient distilled water to make 100 ml of solution.

4) ANALYTICAL PROCEDURE

4.1 Particulate Lead

- 4.1.1** Transfer the glass wool from the sampling probe to a 500 ml Phillips beaker.
- 4.1.2** Add 50 ml of concentrated nitric acid or an amount sufficient to cover the glass wool, and cover the beaker with a ribbed watch glass. Place the beaker on a hot plate and heat gently to boiling. Reflux for one hour and cool. Add 50 ml of distilled water to the beaker and mix.
- 4.1.3** Filter the acid extract through a Whatman No. 1 filter paper, collecting the filtrate in a 400 ml beaker. Wash the glass wool three times with 50 ml portions of hot distilled water. Filter each wash, adding each to the original filtered acid extract.
- 4.1.4** Evaporate the filtrate on a hot plate to approximately 5 ml. Cool to room temperature and quantitatively transfer the filtrate to a 50 ml volumetric flask using distilled water. Bring to volume with distilled water.
- 4.2** From each batch of purchased glass wool, analyze a representative portion in the same manner as the sample.

- 4.3** Set spectrophotometer parameters to obtain maximum sensitivity at a wavelength of 283.3 nm by use of lead standards, and manipulation of the acetylene and air flows.
- 4.4** Aspirate each sample and blank into the flame and record the percent absorption for each. Convert the readings to absorbance units.
- 4.4.1** Samples that have percent absorption values higher than the range of the standard calibration curve of the instruments must be diluted with dilute nitric acid to be within the working range.
- 4.4.2** Determine the lead concentration of the sample from the lead standard curve.
- 4.4.3** Standards should be aspirated after every fourth or fifth sample to insure instrument response has not changed.
- 4.5 Gaseous Lead**
- 4.5.1** Measure and record the total volume of the iodine monochloride solution remaining in the impingers.
- 4.5.2** Transfer an aliquot of 20.0 ml to a 25 ml graduated test tube. Add 2.0 ml of 25% potassium iodide to the sample. Mix well to insure that all the iodine is in solution.
- 4.5.3** Add dropwise, 10% sodium sulfite solution until the solution turns pale yellow. Mix well, and add distilled water to the 25 ml mark.
- 4.6** Prepare a reagent blank in the same manner as the samples to ascertain the presence of any lead impurities.
- 4.7** Proceed as in Sections **4.3** and **4.4**.

5) STANDARD CURVE

- 5.1 Standard Stock Lead Solution.** Weigh and transfer 1.5984 g of lead nitrate to a 1 liter volumetric flask. Dissolve and bring to volume with dilute nitric acid. This solution contains 1000 μgm Pb/ml.
- 5.2 Working Standard Lead Solution.** At least three individual standards, normally in the range of 0 to 20 μgm Pb/ml, are freshly prepared by appropriate dilution of the standard stock lead solution with dilute nitric acid.
- 5.3** Prepare a standard curve by plotting on linear graph paper the absorbance of the individual standards vs. concentration of lead.

6) CALCULATION

6.1 Particulate Lead

6.1.1 The blank glass wool value, if any, is subtracted from each sample.

$$*Total \text{ mg Pb} = \frac{\mu\text{gm Pb/ml from Std Curve} \times 50}{1000}$$

* If sample was diluted to be within the standard curve range (4.4.1), the calculation must be multiplied by the appropriate dilution factor.

6.2 Gaseous Lead

$$*Total \text{ mg Pb per impinger} = \frac{\mu\text{gm Pb/ml} \times 25 \times \text{Vol of Impinger (ml)}}{1000 \times 20}$$

* If sample was diluted to be within the standard curve range (4.4.1), the calculation must be multiplied by the appropriate dilution factor.

Total mg Pb = Total mg Pb in 1st Impinger + 2nd Impinger.

7) REFERENCE

7.1 Air and Industrial Hygiene Laboratory, State Department of Health, Method No. 54. "Analysis of Lead Content of Atmospheric Particulate Matter Collected on High Volume Glass Fiber Filters".

7.2 Purdue, L., Enrione, E. and Thompson, R., "Determination of Organic and Total Lead in the Atmosphere by Atomic Absorption Spectrometry", Anal. Chem. 45, 527-530, 1973.