State of California
Air Resources Board

Method 433

Determination of Total Nickel Emissions
from Stationary Sources

Adopted: September 12, 1989

Method 433 - Determination of Total Nickel Emissions from Stationary Sources

1   APPLICABILITY AND PRINCIPLE

     1.1   Applicability

           This method applies to the measurement of total nickel emissions from
           stationary sources.

     1.2   Principle

           Particulate and gaseous emissions are extracted isokinetically from the
           stack and passed through a filter-impinger train where the nickel is
           collected on a glass-fiber filter and in a solution of nitric acid. The
           combined filter extract and impinger solution are analyzed for nickel by
           atomic absorption spectrometry using the direct aspiration flame method
           (flame AA). (Ref. 10.1.) Unless otherwise specified, all discussion in this
           test method is based on these principles.

2   DETECTION LIMIT AND WORKING RANGE

     The typical detection limit for the recommended analytical technique, flame AA,
     is 40 ug/L of solution analyzed. The typical working range is 300 to 5,000 ug/L of
     solution analyzed.

     2.1   Coordination of Sampling and Analysis

           At a minimum, the following calculations shall be performed before
           conducting a test. The best available measured values or estimates and
           their ranges should be used for each item. These calculations are not
           intended to predict test results. Rather, they shall be used to predict
           incompatibilities among the expected ranges for the items based upon
           information supplied by all of the parties to the testing and analysis.

           Eq. 1   Sample Concentration x Flow x Time = Mass Sampled

           Eq. 2   Mass Analyzed / Liquid Volume = Reported Concentration

           Sample Concentration is the concentration of the analyte in the sampled
           gas. **Flow** is the sample flow rate. **Time** is the time per sampling run.
           Mass Sampled is the mass collected per sampling run. **Mass Analyzed**
           may be the same as the **Mass Sampled** or, if appropriate, the aggregate
           mass of more than one run. **Liquid Volume** is the volume of liquid which
           dissolves the analyte at the time of analytical detection. **Reported
           Concentration** is the concentration reported from analysis; the range
           minimum is the detection limit.

2.2   Other Methods

If the test method herein recommended does not give the desired sensitivity in the presence of interfering substances in the sample, the tester may select an equivalent procedure, subject to approval by the Executive Officer. The tester must then produce data to demonstrate that the method is equivalent, and substantiate this data through an adequate quality assurance program approved by the Executive Officer. Other techniques, including furnace atomic absorption spectrometry (furnace AA) (Ref. 10.2), argon plasma atomic emission spectrometry (Ref. 10.3), neutron activation analysis (NAA) (Ref. 10.4), and x-ray fluorescence (XRF) (Ref. 10.5) are discussed in Section 9.

3   INTERFERENCES

3.1   Background

Background correction is required as nonspecific absorption and light scattering can be significant at the analytical wavelength.

3.2   Other Metals

High concentrations of iron, cobalt, and chromium can suppress nickel absorbance. If this interference becomes measurable, either the method of matrix matching or a nitrous oxide/acetylene flame should be employed.

3.3   Viscosity

Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.

3.4   Matrix Effects

Matrix effects can be kept to a minimum with the Method of Standard Additions. (Ref. 10.6) (Ref. 10.7)

4   APPARATUS

Mention of trade names or specific products does not constitute endorsement by the California Air Resources Board.

The following sampling apparatus is recommended. The tester may use alternative sampling apparatus only if, after review by the Executive Officer, it is deemed equivalent for the purposes of this test method.

4.1   Sampling Train

A schematic diagram of the sampling train is shown in Figure 1. This is similar to the ARB Method 5 with some minor changes which are described below.

4.1.1   Probe Nozzle, Probe Liner, Pitot Tube, Differential Pressure Gauge, Filter Holder, Filter Heating System, Metering System, Barometer and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1 to 2.1.6, and 2.1.8 to 2.1.10, respectively, except that all components which contact the sampled gas shall be of borosilicate or quartz glass.

4.1.2   Impingers. Four impingers are connected in series with glass ball joint fittings. The first, third and fourth impingers are of the Greenburg-Smith modified by replacing the tip with a 1-cm (0.5 in.) I.D. glass tube extending to 1 cm from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip.

The first and second impingers shall contain known quantities of 0.1 N HNO_3 (Section 6.1.3). The third shall be empty, and the fourth shall contain a known weight of silica gel or equivalent desiccant.

A thermometer which measures temperatures to within 1^oC (2^oF), should be placed at the outlet of the fourth impinger.

4.2   Sample Recovery

The following items are needed:

4.2.1   Probe Liner and Probe Nozzle Brushes, Petri Dishes, Plastic Storage Containers, Rubber Policeman and Funnel. Same as Method 5, Sections 2.2.1, 2.2.4, 2.2.6 and 2.2.7, respectively, except that brushes shall be entirely nonmetallic.

4.2.2   Wash Bottles. Glass (2).

4.2.3   Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for 0.1 nitric acid (HNO_3) impinger and probe solutions and washes, 1000 mL. Use screw-cap liners that are either rubber-backed Teflon or leak-free and resistant to chemical attack by 0.1 N HNO_3. (Narrow mouth glass bottles have been found to be less prone to leakage.

4.2.4 Graduated Cylinder and/or Balance. To measure the volume of condensed water to within 2 mL, or the weight to within 1 g. Use a graduated cylinder that has a minimum capacity of 500 mL, and subdivisions no greater than 2 mL. Most laboratory balances are capable of weighing to the nearest 0.5 g or less.

4.2.5. Glass Funnel

4.3 Analysis

The following equipment is needed:

4.3.1 Atomic Absorption Spectrometer. This device must be compatible with the performance requirements of this method.

4.3.2 Strip Chart Recorder (optional)

4.3.3 Hot Plate

4.3.4 Erlenmeyer Flasks. 125 mL. 24/40 t/s.

4.3.5 Whatman No. 42 Filter Paper (or equivalent)

4.3.6 Volumetric Flasks. 100 mL, 200 mL, 250 mL, and 1000 mL.

5 REAGENTS

Unless otherwise specified, use American Chemical Society (ACS) reagent grade (or equivalent) chemicals throughout.

5.1 Sampling

The following reagents are needed:

5.1.1 Filters, Silica Gel, Crushed Ice and Stopcock Grease. Same as Method 5, Sections 3.1.1, 3.1.2, 3.1.4 and 3.1.5, respectively.

5.1.2 Water. Deionized, distilled, to conform to ASTM Specification D1193. Type II. (Ref. 10.8.) If high concentrations of organic matter are not expected to be present, the analyst may omit the potassium permanganate test for oxidizable organic matter.

5.1.3 Nitric Acid, 0.1 N. Dilute 6.5 mL of concentrated HNO_3 to 1 L with deionized distilled water. (It may be desirable to run blanks before field use so that all analyses can be corrected.)

5.2   Pretest Preparation

6 N HNO_3 is needed. Dilute 390 mL of concentrated HNO_3 to 1 liter with deionized distilled water.

5.3   Sample Recovery

0.1 N HNO_3 (same as 5.1.3 above).

5.4   Analysis

The following reagents are needed:

5.4.1   Water. Same as 5.1.2 above.

5.4.2   Nitric Acid, Concentrated. Acid should be analyzed to determine whether the sample analysis should be corrected.

5.4.3   Nitric Acid, 50 Percent (v/v). Dilute 500 mL of concentrated HNO_3 to 1 liter with deionized distilled water.

5.4.4   Nickel Standard Stock Solution (1000 mg Ni/L). This may be a commercially available standard that has been certified, and is traceable to NBS Standards, or it may be prepared as follows:

Dissolve 4.953 g of nickel nitrate, $Ni(NO_3)_2 \cdot 6H_2O$ (analytical reagent grade), in Type II water. Add 10 mL of concentrated nitric acid and dilute to 1 liter with Type II water in a volumetric flask.

5.4.5   Intermediate Stock Standards. Pipet 2.00 mL of the stock nickel standard solution (5.4.4) into a 200 mL volumetric flask, and dilute to volume with deionized distilled water. This corresponds to 10.00 mg Ni/L.

5.4.6   Working Nickel Standards. Pipet 0.00, 4.00, 10.00, 20.00, 30.00 and 40.00 mL of the intermediate stock standard (5.4.5) into 200 mL volumetric flasks. Add 6 mL of concentrated HNO_3 to each flask and dilute to volume with Type II water. The concentrations of these working standards are 0.0, 0.20, 0.50, 1.00, 1.50 and 2.00 mg Ni/L, respectively. Prepare additional standards at lower concentrations as needed. The final concentration of acid in the working standards should be the same as that of the sample to be analyzed.

5.4.7   Air. Suitable quality for atomic absorption analysis. The air should be cleaned, dried and filtered to remove oil, water and other foreign

substances. The air may be supplied from a compressed air line, a laboratory compressor, a cylinder of compressed air, or as recommended by the instrument manufacturer.

5.4.8   Acetylene. Suitable quality for atomic absorption analysis. Commercial grade acetylene or as recommended by the instrument manufacturer. Acetone, which is usually present in acetylene cylinders, can be prevented from entering and affecting flame conditions by replacing the cylinder before the pressure has fallen to 50 psig.

5.4.9   Hydrogen Peroxide, 3 Percent (v/v). Dilute 10 mL of 30 percent H_2O_2 to 100 mL with Type II water.

6   PROCEDURE

6.1   Sampling

Because of the complexity of this method, testers should be trained and experienced with the test procedures in order to ensure reliable results.

6.1.1   Pretest Preparation. Follow the same general procedure given in Method 5, Section 4.1.1, except the filter need not be weighed.

6.1.2   Preliminary Determinations. Follow the same general procedure given in Method 5, Section 4.1.2.

6.1.3   Preparation of Collection Train. Follow the same general procedure given in Method 5, Section 4.1.3, except place 100 mL of 0.1 N HNO_3 in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. Assemble the train as shown in Figure 1.

6.1.4   Leak-Check Procedures. Follow the general leak-check procedures given in Method 5, Sections 4.1.4.1 (Pretest Leak-Check), 4.1.4.2 (Leak-Checks During the Sample Run), and 4.1.4.3 (Post-Test Leak-Check).

6.1.5   Sampling Train Operation. Follow the same general procedure given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in CARB Method 5, Figure 5-2.

6.1.6   Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

6.2   Sample Recovery

Begin proper clean-up procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing liquid from the impingers into the filter.

Before moving the sampling train to the cleanup site, remove the probe from the sampling train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the glassware inlet where the probe was fastened and cap the inlet. Remove the umbilical cord from the last impinger and cap the impinger. The tester may use ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and filter-impinger assembly to a cleanup area, which is clean and protected from the wind so that the chances of contaminating or losing the sample are minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the sample as follows:

6.2.1 Container No. 1 (Filter). Carefully remove the filter from the filter holder and place it in its identified petri dish container. If it is necessary to fold the filter, make sure that the sample-exposed side is inside the fold. Carefully transfer to the petri dish any visible sample matter and/or filter fibers that adhere to the filter holder gasket by using a dry Nylon bristle brush and/or nonmetallic scraper. Seal the container.

6.2.2 Container No. 2 (Probe). Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, clean all surfaces that have been exposed to the sample (including the probe nozzle, probe fitting, probe liner, and front half of the filter holder) with 0.1 N HNO_3. Place the wash in a glass sample storage container. Measure and record (to the nearest 2 mL) the total amount of 0.1 N HNO_3 used for each rinse.

Perform the rinses with 0.1 N HNO_3 as follows:

Carefully remove the probe nozzle and rinse the inside surface with

0.1 N HNO_3 from a wash bottle. Brush with a nylon-bristle brush, and rinse until the 0.1 N HNO_3 rinse shows no visible particles, after which, make a final rinse of the inside surface. Brush and rinse the inside nonmetallic parts of any couplings with 0.1 N HNO_3 in a like manner until no visible particles remain.

Rinse the probe liner with 0.1 N HNO_3. While squirting the nitric acid rinse into the upper end of the probe, tilt and rotate the probe so that all inside surfaces are wetted with the nitric acid. Let the 0.1 N HNO_3 drain from the lower end into the sample container. The tester may use a glass funnel to aid in transferring liquid washes to the container. Follow the rinse with a probe brush. Hold the probe in an inclined position, and squirt 0.1 N HNO_3 into the upper end as the probe brush is being pushed with a twisting action through the probe.

Hold the sample container underneath the lower end of the probe, and catch any liquid and particulate matter brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the 0.1 N HNO_3 and none remains on the probe liner on visual inspection. Rinse the brush with 0.1 N HNO_3 and quantitatively collect these washings in the sample container. After the brushing, make a final rinse of the probe as described above. It is recommended that two people clean the probe to minimize loss of sample: Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints have been wiped clean of silicone grease, brush and rinse with 0.1 N HNO_3 the inside of the front half of the filter holder. Brush and rinse each surface three times or more, if needed, to remove visible particulate matter. Make a final rinse of the brush and filter holder. After all 0.1 N HNO_3 washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that the liquid will not leak out when it is shipped to the laboratory.

Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container clearly to identify its contents.

Rinse the glassware a final time with water to remove residual HNO_3 before reassembling. Do not save the final rinse water.

6.2.3 Container No. 3 (Silica Gel). Check the color of the indicating silica gel to determine if it has been completely spent and note its condition. Transfer the silica gel from the fourth impinger to the

original container and seal. The tester may use a funnel to pour the silica gel and rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of particles that may adhere to the impinger walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, the tester may follow procedure for Container No. 3 under Section 6.4 (Analysis).

6.2.4 Container No. 4 (Impingers). If the volume of liquid is large, the tester may place the impinger solutions in several containers. Clean each of the first three impingers and connecting glassware in the following manner:

Wipe the impinger ball joints free of silicone grease and cap the joints.

Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.

Transfer the contents of the impingers to a 500 mL graduated cylinder. Remove the outlet ball joint cap and drain the contents through this opening. Do not separate the impinger parts (inner and outer tubes) while transferring their contents to the cylinder. Measure the liquid volume to within $\pm$2 mL.

Alternatively, determine the weight of the liquid to within $\pm$ 0.5 g. Record in the log the volume or weight of the liquid present, along with a notation of any color or film observed in the impinger catch. The liquid volume or weight is needed, along with the silica gel data, to calculate the stack gas moisture content. (See Method 5, Figure 5-3.)

Transfer the contents to Container No. 4.

Note: In the steps below, measure and record the total amount of 0.1 N HNO_3 used for rinsing. Pour approximately 30 mL of 0.1 N HNO_3 into each of the first three impingers and agitate the impingers. Drain the 0.1 N HNO_3 through the outlet arm of each impinger into Container No. 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

Wipe the ball joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1 N HNO_3; transfer this rinse into container No. 4. (Do not rinse or brush the glass-fritted filter support.) Mark the height of the fluid

level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

6.2.5 Blanks. Save 200 mL of the 0.1 N HNO_3 used for sampling and cleanup as a blank. Take the solution directly from the bottle being used and place into a glass sample container.

6.3 Sample Preparation

6.3.1 Container No. 1 (Filter). Cut the filter into strips and transfer the strips and all loose particulate matter into a 125 mL Erlenmeyer flask. Rinse the petri dish with 10 mL of 50 percent HNO_3 to insure a quantitative transfer and add to the flask. (Note: If the total volume required in Section 6.3.3 is expected to exceed 80 mL, use a 250 mL Erlenmeyer flask in place of the 125 mL flask.

6.3.2 Containers No. 2 and No. 4 (Probe and Impingers). Check the liquid level in Containers No. 2 and/or No. 4 to determine whether any sample was lost during shipment. Record observations on the analysis sheet. If a noticeable amount of leakage has occurred, either void the sample or take steps, subject to the approval of the Executive Officer to adjust the final results. Combine the contents of Containers No. 2 and No. 4 and evaporate to near dryness on a hot plate.

6.3.3 Sample Extraction for Nickel. Using the approximate stack gas particulate concentration and the total volume of stack gas sampled, estimate the total weight of particulate sample collected. Then transfer the residue from containers No. 2 and No. 4 to the 125-mL Erlenmeyer flask that contains the filter using a rubber policeman and 10 mL of 50 percent HNO_3 for every 100 mg of sample collected in the train or a minimum of 30 mL of 50 percent HNO_3 whichever is larger.

Place the Erlenmeyer flask on a hot plate and heat with periodic stirring for 30 minutes at a temperature just below boiling. If the sample volume falls below 15 mL, add sufficient 50 percent HNO_3 to bring volume back up to 15 mL. Add 10 mL of 3 percent H_2O_2 and continue heating for 10 min. Add 50 mL of hot ($80^{o}C$) deionized distilled water and heat for 20 min. Remove the flask from the hot plate and allow to cool. Filter the sample through Whatman No 42 filter paper (or equivalent) and transfer the filtrate to a 250 mL volumetric flask. Dilute to volume with deionized distilled water. The diluted sample has an approximate acid concentration of 3% (v/v) HNO_3 and is now ready for analysis.

Depending upon the total weight estimate above and the working range of nickel concentrations for the analytical device, adjustment of the volumetric flask size and reagent volumes may be necessary.

6.3.4 Filter Blank. Determine a filter blank using two filters from each lot of filters used in the sampling train. Cut each filter into strips and place each filter in a separate 125 mL Erlenmeyer flask. Add 15 mL of 50 percent HNO_3 and treat as directed in Section 6.3.3 beginning with the sentence "Add 10 mL of 3 percent H_2O_2 and continue heating for 10 min". Filter and dilute to a total volume of 100 mL using deionized distilled water.

6.3.5 0.1 N HNO_3 Blank. Take the entire 200 mL of 0.1 N HNO_3 to near dryness on a steam bath. Add 15 mL of 50 percent HNO_3 and treat as directed in Section 6.3.3 beginning with the sentence "Add 10 mL of 3 percent H_2O_2 and continue heating for 10 min". Filter and dilute to a total volume of 100 mL using deionized distilled water.

6.4 Analysis

6.4.1 Nickel Determination. Calibrate the spectrophotometer as described in Section 7.2. Aspirate each sample, the filter blank, and 0.1 N HNO_3 blank, and determine the absorbance or read the concentration directly from the readout system. Analyze each sample three times in this manner. Use a minimum of one blank per sample batch to determine if contamination or memory effects are present.

If the absorbance of a sample is above the linear range of the standards, dilute the sample with 3% HNO_3, solution, (5.4.10), reanalyze and use the appropriate dilution factor in calculations. If the Ni concentration of a sample is at the low end of the calibration curve and high accuracy is required, the sample can be taken to near dryness on a hot plate and the residue dissolved in the appropriate volume of 3% HNO_3 solution to bring it into the linear range of the calibration curve.

6.4.2 Mandatory Check for Matrix Effects. The analysis for Ni by atomic absorption is sensitive to the chemical composition and to the physical properties e.g., viscosity, pH, of the sample (matrix effects). As the Ni procedure described here will be applied to many different sources, many sample matrices will be encountered. Therefore, check (mandatory) at least one sample from each source using the Method of Additions to ascertain that the chemical composition and physical properties of the sample have not caused

erroneous analytical results.

Three acceptable "Method of Additions" procedures are described in the General Procedure Section of the Perkin Elmer Corporation Manual (Ref. 10.6). If the results of the Method of Additions procedure on the source sample do not agree within 5 percent of the value obtained by the conventional atomic absorption anlaysis, then the tester must reanalyze all samples from the source using the Method of Additions procedure.

6.4.3 Container No. 3 (Silica Gel). The tester may conduct this step in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g; record this weight.

7 CALIBRATION

7.1 Sampling Train Calibration

Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Section 5.5); Leak-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

7.2 Spectrophotometer

Use the 232.0-nm wavelength line and background correction. An oxidizing air-acetylene flame is required. Follow the manufacturer's operating instructions for all other instrument parameters.

Measure the absorbance of the standard solutions. Repeat until good agreement ($\pm$3 percent) is obtained between two consecutive readings. For those instruments which read directly in concentrations, set the curve corrector to read out the proper concentration.

Either (1) construct a calibration curve by plotting the absorbance of the standards (y-axis) versus concentration in ug Ni/mL (x-axis), or (2) for the method of standard additions, plot absorbance versus added concentration. Draw or compute a straight line through the linear portion of the curve. Do not force the calibration curve through zero, but if the curve does not pass through the origin or at least lie closer to the origin than $\pm$ 0.003 absorbance units, check for incorrectly prepared standards and for curvature in the calibration curve.

To determine stability of the calibration curve, run a blank and a standard after every five samples and recalibrate, as necessary. Calibration curves

must be composed of a minimum of a blank and three standards.

Spiked samples or standard reference materials should be run periodically to ensure that correct procedures are being followed, and that all equipment is operating properly.

8   CALCULATIONS

8.1   Dry Gas Volume

Using the data from this test, calculate $V_{m(std)}$ the total volume of dry gas metered corrected to standard conditions (20 ^{o} and 760 mm Hg), by using Equation 5-1 of Method 5. If necessary, adjust $V_{m(std)}$ for leakages as outlined in Section 6.3 of Method 5. Check the field data sheet for the average dry gas meter temperature and average orifice pressure drop.

8.2   Volume of Water Vapor and Moisture Content

Using data obtained in this test and Equations 5-2 and 5-3 of Method 5, calculate the volume of water vapor $V_{w(std)}$ and the moisture content B_{ws} of the stack gas.

8.3   Total Nickel in Source Sample

For each source sample correct the average absorbance for the contribution of the filter blank and the 0.1 N HNO_3 blank. Use the calibration curve and this corrected absorbance to determine the Ni concentration in the sample aspirated into the spectrophotometer. Calculate the total Ni mass M^o_{Ni} (in ug) in the original source sample.

Correct for all the dilutions that were made to bring the Ni concentration of the sample into the linear range of the spectrophotometer.

8.4   Nickel Concentration in Stack Gas

Calculate the stack nickel concentration C_{Ni} in mg/dscm as follows:

$$C_{Ni} = \frac{K\,M^o_{Ni}}{V_{m(std)}} \qquad \textbf{1}$$

Where:

K   =   0.001 mg/ug for metric units.
      =   2.205 x 10^{-9} lb/ug for English units.

8.5   Isokinetic Variation and Acceptable Results

Same as Method 5, Sections 6.11 and 6.12, respectively. To calculate V_s the average stack gas velocity, use Equation 2-9 of Method 2 and the data from this field test.

9   OTHER METHODS

9.1   Furnace AA

Properly conducted, this method can be equivalent to the flame AA, based on current technology. This method is recommended for measurement of lower nickel concentrations. The typical working range, 5 to 100 ug/L, is about 100 times smaller than for flame AA. Intralaboratory and interlaboratory studies have demonstrated that the method will yield results with acceptable precision, accuracy, and specificity. However, this method requires greater skill and experience. (Ref. 10.1.)

9.2   Argon Plasma Atomic Emission Spectrometry

Properly conducted, this method can be equivalent to the flame AA, based on current technology. The sensitivity and working range is similar. This is a more expensive analytical technique that requires much skill to interpret results and obtain valid data. (Ref. 10.3.)

9.3   Neutron Activation Analysis

Properly conducted, this method can be equivalent to the flame AA, based on current technology. This is the most scientifically sophisticated method, it is slightly more sensitive than furnace AA. This method requires the use of radioisotopes and is more of a research method than one which is routinely available. (Ref. 10.4.)

9.4   X-Ray Fluorescence

This method is used for ambient nickel measurements, with low filter loadings. It is not suited for the high heterogeneous filter loadings commonly found in source sampling. (Ref. 10.5.)
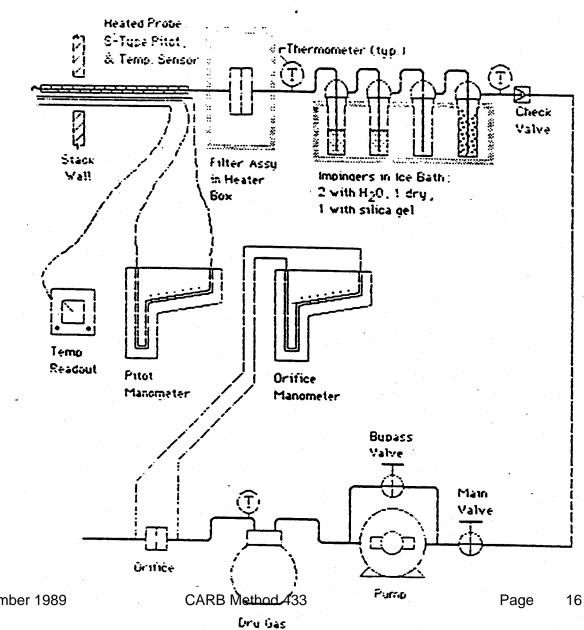
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Figure 1

Sampling Train