

## IX. REFERENCES

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## X. APPENDIX I

### METHOD FOR SAMPLING NICKEL IN AIR

The following sampling method is adapted from that described in Method No. 173 of the NIOSH Manual of Analytical Methods [187].

#### General Requirements

Collect breathing-zone samples representative of the individual employee's exposure. At the time of sample collection, record a description of sampling location and conditions, equipment used, date, time and rate of sampling, and any other pertinent information.

#### Equipment

The sampling train consists of a filter in a filter cassette, a suitable length of tubing, and a vacuum pump.

(a) Filters and cassettes: a 2- or 3-piece filter cassette containing a 37-mm cellulose mixed-ester membrane filter with a 0.8- $\mu$ m pore size.

(b) Pump: A battery-operated vacuum pump, complete with clip for attachment to the employee's belt, capable of operating at 2.0 liters/minute for up to 8 hours.

## Calibration

Accurate calibration of a sampling pump is essential to obtain a correct value for the volume of air sampled. The frequency of calibration depends on the use, care, and handling to which the pump is subjected. If the pump receives hard use, more frequent calibration may be necessary. Pumps should be recalibrated if they have been subjected to misuse or if they have just been repaired or received from a manufacturer. Maintenance and calibration should be performed and recorded on a regular schedule, for example, after every 40 hours of use or once a month regardless of use.

Ordinarily, pumps should be calibrated in the laboratory both before they are used in the field and at intervals after they have been used to collect a large number of field samples. The accuracy of calibration depends on the type of instrument used as a reference. The choice of calibration instrument will depend largely upon where the calibration is to be performed. For laboratory testing, a 1-liter soapbubble meter is recommended, although other standard calibrating instruments, such as the wet-test meter, can be used.

Instructions for calibration with the soapbubble meter follow. If another calibration device is used, equivalent procedures should be used. The calibration setup for a personal sampling pump with a filter is shown in Figure XV-2. Since the flow rate obtained with a pump depends on the pressure drop across the sampling device, in this case a cellulose mixed-ester membrane filter, the pump must be calibrated while operating with a representative filter in line.

(a) Check the voltage of the pump battery with a voltmeter to assure adequate voltage for calibration. Charge the battery if necessary.

- (b) Place a membrane filter in the holder.
- (c) Assemble the sampling train as shown in Figure XV-2.
- (d) Turn the pump on and moisten the inside of the soapbubble meter by immersing the buret in the soap solution. Draw bubbles up the inside until they are able to travel the entire buret length without bursting.
- (e) Adjust the pump rotameter to provide the desired flowrate.
- (f) Check the water manometer to insure that the pressure drop across the sampling train does not exceed 12 inches of water (approximately 1 inch of mercury) at 2.0 liters/minute.
- (g) Start a soapbubble up the buret and measure with a stopwatch the time it takes for the bubble to move from one calibration mark to another. If, for the pump being calibrated, the number of strokes is used to determine volume, count the number of strokes required to move the bubble from one marker to another.
- (h) Repeat the procedure in (g) at least three times, average the results, and calculate the flowrate by dividing the volume between the preselected marks by the time required for the soapbubble to traverse the distance.
- (i) Record calibration data, including the volume measured, elapsed time, pressure drop, air temperature, atmospheric pressure, serial number of the pump, date, and name of the person performing the calibration.
- (j) Use graph paper to record the airflow as the ordinate and the rotameter readings as the abscissa.

### Sampling Procedure

(a) Open a filter cassette and connect the back of the cassette to the pump with tubing. The filter cassette is supported or clipped in a vertical position, facing down, in the employee's breathing zone.

(b) Sample a minimum of 700 liters of air. For example, 2.0 liters/minute for about 6 hours.

(c) Measure and record the temperature and pressure of the atmosphere being sampled.

(d) Treat at least one filter in the same manner as sample filters (break open, reseal, and ship), but draw no air through it. This filter serves as a blank.

(e) Immediately after the samples are collected, recap the filter cassette, label, and ship for analysis. Include a blank filter with each batch of filters sent to be analyzed.

## XI. APPENDIX II

### ANALYTICAL METHOD FOR AIRBORNE NICKEL

The recommended atomic absorption spectrophotometric method is based on Method No. 173, described in the NIOSH Manual of Analytical Methods [187].

#### Principle of the Method

Particles are collected on a cellulose mixed-ester membrane filter. The filter is ashed in nitric acid, and the residue is dissolved in dilute nitric acid. The absorbance of nickel at 232 nm is determined by atomic absorption spectrophotometry in an oxidizing flame and compared to the absorbance of standards.

#### Range and Sensitivity

The working range of the method is 1.0-20  $\mu\text{g}$  nickel/ml of the redissolved ash solution. This range is somewhat instrument dependent and can be extended to higher concentrations by dilution of the sample or measurement of a less sensitive line.

The sensitivity of the method has been determined to be 0.2  $\mu\text{g}$  nickel/ml of the redissolved ash solution. This value will vary somewhat depending on individual instrumentation.

### Interferences

No interferences are listed in the NIOSH Manual of Analytical Methods [187], although another report [209] has indicated that a hundredfold excess of iron, manganese, chromium, copper, cobalt, or zinc may decrease the absorbance recorded for nickel by as much as 12%. However, it also was noted [209] that this effect can be minimized by proper burner elevation and by the use of an oxidizing flame. In addition, a high solids content in the aspirated solution will cause increased nonspecific absorbance if the 232-nm line is used [250].

### Precision and Accuracy

The coefficient of variation for analysis by atomic absorption is approximately 2% for absorbances greater than 1%. An absorbance of less than 1% increases the coefficient of variation. At the lowest end of the working range, a reading error of as much as 3% can occur with an instrument equipped with a digital readout.

No data on accuracy are available at this time.

### Apparatus

- (a) Atomic absorption spectrophotometer with nickel hollow-cathode lamp and a burner head for an air-acetylene flame.
- (b) Hotplate.
- (c) Volumetric flasks.
- (d) Pipets.

(e) Oxidant: air which has been filtered to remove oil, water, and other foreign materials.

(f) Fuel: commercially available acetylene.

(g) Pressure-reducing valves.

(h) Beakers.

(i) Watchglasses.

### Reagents

All reagents used must be ACS certified reagent grade or better.

(a) Doubly distilled or deionized water.

(b) Redistilled concentrated nitric acid.

(c) Distilled 1:1 hydrochloric acid.

(d) Commercially prepared aqueous stock standard (1,000  $\mu\text{g}$  of nickel/ml): serially dilute commercial standards in 5% nitric acid to cover a range of nickel concentrations from 1.0  $\mu\text{g}/\text{ml}$  to 20  $\mu\text{g}/\text{ml}$ . The diluted standards should be stored in polyethylene bottles which have been soaked in 5% nitric acid and should be stored for no more than 1 week.

### Procedure

(a) Clean glassware if it has never been used by soaking it for 24 hours in 5% nitric acid. Rinse thoroughly with warm tap water and then with doubly distilled water. After glassware has been used, wash it first in detergent and rinse with tap water. Then soak it in 5% nitric acid for 1 hour, rinse it thoroughly with doubly distilled water, and allow it to dry. Use plastic containers for washing and do not handle the glassware without

gloves which are impervious to sweat.

(b) Transfer the sample and the blank filter to 125-ml beakers and add 5 ml of concentrated nitric acid to each. Cover each beaker with a watchglass and heat it on a hotplate in a ventilation hood until only a few drops of solution remain in the beaker. Several additions of nitric acid may be needed for complete ashing.

(c) Dissolve the ash in 5% nitric acid with heating. If solids are present in the solution, repeat the ashing procedure with an additional 5 ml of a 1:4 mixture of hydrochloric and nitric acids. Quantitatively transfer the solution to a volumetric flask. Increase the total volume to 10 ml with 5% nitric acid. Dilution may be necessary if the amount of nickel in the sample exceeds the upper limit of the range of detection.

(d) Aspirate the sample solutions and the standard solutions into an oxidizing flame. Measure and record the absorbance of each sample at 232 nm. It is advisable to run the set of standards before, during, and after a sample run to ensure that conditions have not changed. Correction for nonspecific absorption can be made for measurements at 232.0 nm by the use of a hydrogen or deuterium lamp, or by subtracting the absorbance at the 231.7-nm nonabsorbing line of nickel.

### Calculations

Prepare a calibration curve by plotting the absorbance of the standards versus the concentration ( $\mu\text{g/ml}$ ) of each standard on linear graph paper. Subtract the absorbance of the filter blank from the absorbance of each sample. Read the nickel concentration corresponding to this

absorbance value from the corrected calibration curve. The concentration of nickel in air is calculated as:

$$\mu\text{g nickel/cu m} = \frac{\mu\text{ g nickel/ml (from graph)} \times \text{total ml of solution}}{\text{cu m of air sampled}}$$