

VII. COMPATIBILITY WITH OTHER STANDARDS

The Environmental Protection Agency has not classified cadmium as a hazardous pollutant and neither emission nor ambient air standards have been issued.

EPA has not promulgated solid waste regulations for cadmium. However, it has reaffirmed the 1962 PHS drinking water standard of 0.01 mg/liter²⁷⁹ as an interim primary drinking water

regulation, applicable to community water supplies (*Federal Register* 40:59570, December 24, 1975). While this standard is for community water supplies, and thus is not directly applicable to discharge of cadmium into streams, it seems clear that disposal of cadmium waste, liquid or solid, should be in a manner not leading to its introduction into drinking water.

VIII. REFERENCES

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IX. APPENDIX I

AIR SAMPLING METHOD

Apparatus

(a) A cellulose ester membrane filter of nominal pore size $0.8 \mu\text{m}$ is recommended.

(b) The filter should be mounted in a holder with a cover to protect it during sampling. One or more holes of approximately 4 mm in diameter must be provided in the cover for airflow during sampling (eg, a Millipore Field Monitor in which the inlet plug has been removed).

(c) Connections between the filter holder and the sampling pump must be made in a manner to prevent leakage.

(d) The air sampling pump must be equipped with a means of indicating flow, either directly by a rotameter or indirectly by a suitably geared motor revolution counter.

(e) The air sampling pump should be calibrated so that flow may be determined to within 5%. A spirometer, wet-test meter, bubble meter, or the equivalent, can be used for calibration (see discussion of calibration). A rotameter may be used as flow indicator but should not be used for calibration of pumps with pulsating flow.

(f) Battery-operated pumps must be capable of at least 4 and preferably 8 hours of continuous operation without recharging.

Sampling Procedure for Determining Worker Exposure

(a) Samples should be taken within 50 cm of the worker's nose and mouth.

(b) Each sample for TWA concentration estimation should be taken for 2 or more hours and combined to obtain a TWA concentration for an entire shift. The most meaningful results will be obtained by combining four 2-hour samples (for an 8-hour shift). A single full-shift sample (8-10

hours), two 4-hour samples, or other combinations of full-shift consecutive samples, are also acceptable.

(c) Partial-period consecutive or intermittent samples, for example a number of 15-minute samples for estimation of TWA and ceiling concentrations, are acceptable if they adequately represent full-shift exposure.

(d) Air samples for TWA determinations should have sample volumes of at least 50 liters. A sampling rate of 1.0-2.0 liter/min for personal samples is recommended. Personal samples may be taken at rates of 0.5-3.0 liters/min.

(e) For each air sample, there should be a record of: the date, time, and place of sampling; the operation(s) being carried out during the sampling; the device used to obtain the sample; the sampling rate and sampling period; the name of the person collecting the sample; and other relevant information. Each sample must be clearly labeled for identification.

Sampling Procedure for Determining Control Effectiveness or Need for Personal Protection

(a) Area samples may be taken with the apparatus used for determining worker exposure, or with electrostatic precipitators.

(b) The sample should be taken at a site representative of the area being contaminated. The site should be identified for future sampling.

Calibration of Sampling Trains

The accurate calibration of a sampling pump is essential for the correct interpretation of the volume indicator. The necessary frequency of calibration is dependent on the use, care, and handling to which the pump has been subjected. In addition to the normally scheduled calibration, pumps should be recalibrated if they have been subjected to misuse, just received from a manufac-

turer, or just repaired. If the pump receives hard usage, more frequent calibration may be necessary.

Ordinarily, pumps should be calibrated in the laboratory before they are used in the field and at frequent intervals if they are used to collect numerous field samples. The accuracy of calibration is dependent on the type of calibrating instrument used as a reference. The choice of calibrating instrument may depend largely upon where the calibration is to be performed. For laboratory testing, a 1- or 2-liter buret or a wet-test meter is recommended, although other standard calibrating instruments, such as a spirometer, Marriott's bottle, or dry-gas meter, can be used.

Instructions for calibration with the soapbubble flowmeter follow. However, if an alternative calibration device is selected, equivalent procedures should be used. The calibration setup for personal sampling pumps with a cellulose filter is shown in Figure XIV-1. Since the flowrate indicated by the flowmeter of the pump is dependent on the pressure drop across the sampling device, a membrane filter with appropriate backup pad, the pump flowmeter must be calibrated while operating with a representative filter and backup pad in the line.

(1) While the pump is running, the voltage of the pump battery is measured with a voltmeter to assure that the battery is charged adequately for calibration.

(2) Place the cellulose membrane filter with backup pad in the filter cassette.

(3) The calibration setup is assembled as shown in Figure XIV-1.

(4) The pump is turned on and the inside of the soapbubble meter is moistened by immersing the buret in the soap solution and drawing bubbles up the tube until they are able to travel the entire length of the buret without bursting.

(5) The pump is adjusted to provide a flowrate of 2.0 liters/min.

(6) The water manometer is checked to ensure that the pressure drop across the sampling train does not exceed 13 conventional inches of water (3.23 kPa) at 2 liters/min.

(7) A soapbubble is started up the buret and the time it takes the bubble to travel a minimum of 1.0 liter is measured with a stopwatch.

(8) The procedure in (7) above is repeated at least three times, the results are averaged, and the flowrate is calculated by dividing the volume between the preselected marks by the time required for the soapbubble to travel the distance.

(9) Data recorded for the calibration should include the volume of air measured, elapsed time, pressure drop, air temperature, atmospheric pressure, serial number of the pump, date, and name of the person performing the calibration.

X. APPENDIX II

ENVIRONMENTAL ANALYSIS

Determination of Cadmium in Air Samples by Atomic Absorption Spectroscopy

(a) Principle of the Method

Airborne cadmium dust and fume samples are collected on cellulose ester membrane filters or other collection systems as described in Appendix I. The filter samples are wet-ashed, using nitric acid. Analysis is performed by aspiration of the ashed sample solution into the flame of an atomic absorption spectrophotometer, and comparing the instrument response to that of a standard.

(b) Range and Sensitivity

The sensitivity in conventional flame atomic absorption may be 10-25 ng Cd/ml/0.0044 absorbance units, with detection limits as low as 1 ng Cd/ml, depending on operating conditions and instrumental variations. Using a sensitivity of 20 ng Cd/ml with an air sample of 100 liters (eg, 100 minutes at 1 liter/min) and a final liquid volume of 5 ml, the sensitivity is 1 μg Cd/cu m. For these same air and liquid volumes, the linear response range extends up to 100 μg Cd/cu m (final liquid concentration of 2 μg Cd/ml). Samples may be diluted to provide cadmium concentrations in this range.

(c) Interferences, Precision, and Accuracy

Interferences with this method are not normally encountered in the analysis of air samples. The precision for spiked samples has been determined to be 4% relative standard deviation; a larger deviation could be expected for "real" samples, using a similar method.²⁵⁵ The accuracy has not been determined; however, recoveries of 90% were reported, using 20 replicates.²⁵¹ Collaborative testing has not been performed. The dependability of a similar procedure has been tested²⁵⁵ and is discussed in Chapter IV.

(d) Advantages and Disadvantages of the Method

Analysis can be performed quickly and accurately by a technician experienced in trace analysis. Atomic absorption instrumentation, although not available in all laboratories, is becoming increasingly popular. The speed of the analysis by this method is a distinct advantage.

(e) Reagents and Apparatus

American Chemical Society (ACS) reagent grade chemicals or materials of similar quality are required.

(1) Concentrated nitric acid: redistilled or of trace metal quality. Use in subsequent steps where nitric acid is designated.

(2) Water: double distilled or distilled deionized.

(3) Standard cadmium solution: 1000 $\mu\text{g}/\text{ml}$. Dissolve 1.000 g of metallic cadmium in nitric acid and dilute to 1 liter in a volumetric flask with sufficient water and nitric acid to yield a solution containing 10% nitric acid. This solution is commercially available.

(4) Atomic absorption apparatus with a cadmium hollow cathode lamp, readout accessory, and gas supply system. Operating conditions recommended by the manufacturer should be followed for gas flow rates and other instrumental variables. The resonance line for cadmium is 2,288 Angstroms.

(5) Hot plate, capable of 300 C.

(f) Quality Control

Establishment and maintenance of total analytical quality control systems to assure continued precision and accuracy of laboratory reports include, as appropriate, these requirements:

(1) Each test must be checked on each day of use.

(2) At least one standard (it may be an instrument standard) and one control sample (working value established and run through the entire analytical procedure) should be included with each run of unknown samples. Where the

control sample is not subject to the interferences in the unknown samples, a previously run unknown should be included as a blind check sample. A blank sample (no added amount of the constituent being determined) should also be run to aid in detecting reagent contamination and other problems important near the lower limit of operation of the method.

(3) If the results on the standard, control, blank, or recycle samples are not within acceptable limits, the entire batch of analyses must be repeated and consideration should be given to the nonacceptance of samples where there is only enough material for a single analysis. There may be situations where this policy is waived. Consideration of the consequences of reporting results when the analytical system is apparently "out of control" should minimize such waivers.

(g) Procedure

(1) Trace Analysis Precautions

All glassware should be thoroughly cleaned and rinsed. Cleaned glassware should be soaked overnight in 10% nitric acid or one-half hour in 50% nitric acid prior to use. Glassware taken from routine laboratory use should be checked for cadmium leachability before it is integrated into the trace analysis system. Note that it is sometimes the case that previous use will make glassware unsuitable for trace analysis.

Analysts are cautioned concerning the potential for contamination of samples by smoking. Hands should be washed and smoking forbidden in the trace analysis facility.

(2) Standardization

Standards are prepared from the 1,000 $\mu\text{g/ml}$ standard solution by serial dilution with 1% nitric acid. Because of the loss of trace metals to glassware or plastic, dilute standards should be made daily unless in-house data show no depletion when compared with fresh standards. Standards are prepared in the 0.02 to 2 $\mu\text{g/ml}$ range; standards should preferably bracket the samples. Aspirate the standards into the flame and record the instrument response. Prepare a graph of the results by plotting the absorbance vs concentration for each standard. Other instrumental output, such as percent absorption, may be used over a narrow range of concentrations. Instruments with devices that read directly in terms of concentration are also suitable.

(h) Analysis of Samples

Sample, control sample (working value previously established by carrying through entire analytical procedure), and blank are placed in suitable acid-washed vessels. For personal samplers employing 37 mm or similar filters, 50-ml Griffen beakers are suitable. Next, 3 ml of concentrated nitric acid is added and the vessel is covered with a watch glass. Each sample is heated on a hot plate in an exhaust hood until the volume of nitric acid is reduced to approximately 0.5 ml and is pale yellow or water white. Further additions of nitric acid may be necessary for complete oxidation of the filter. The cooled sample is transferred to a suitable volumetric vessel, after rinsing the watch glass. A 5-ml graduated cylinder has been employed successfully. The transfer and volume adjustment are effected with 1% nitric acid solution.

The resulting blank, control and sample solutions are aspirated into the flame of the instrument and the instrument response recorded. Samples may be diluted or concentrated to correspond to the standards. If concentration is necessary, the aspiration flow rate should be checked to assure that it is comparable to those for the standards.

(i) Calculations

Absorbances of sample and blank solutions are converted to concentration values by comparison with a curve of absorbance vs concentration prepared from the standards. The blank, which represents the level of contamination in the system, is subtracted from the sample value. The concentration of cadmium in the environmental sample is determined by the following formula:

$$\mu\text{g Cd/cu m} = \frac{\text{Cd} \times \text{S}}{\text{V}}$$

where Cd = Sample concentration minus blank concentration from standard curves, in $\mu\text{g Cd/ml}$

S = Solution volume, in ml

V = Volume of air sample, in cubic meters