
**Workplace air — Determination
of particulate cadmium and
cadmium compounds — Flame and
electrothermal atomic absorption
spectrometric method**

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

This second edition cancels and replaces the first edition (ISO 11174:1996), which has been technically revised.

The main changes are as follows:

- a reference for handling of sampler wall deposits has been added;
- references and definitions have been updated;
- additional editorial changes have been made.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The health of workers in many industries, such as mining, metal refining, battery manufacture, foundries, electronics and construction, is at risk through exposure by inhalation of particulate cadmium and cadmium compounds. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure, and this is generally achieved by making workplace air measurements. This document provides a method for making valid exposure measurements for cadmium. It is of benefit to: agencies concerned with health and safety at work, industrial hygienists and other public health professionals, analytical laboratories, industrial users and workers of metals and metalloids, etc.

The execution of its provisions and the interpretation of the results obtained is entrusted to appropriately qualified and experienced people.

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Workplace air — Determination of particulate cadmium and cadmium compounds — Flame and electrothermal atomic absorption spectrometric method

WARNING — Cadmium and cadmium compounds are toxic and are suspected human carcinogens [1]. Avoid any exposure by inhalation. Personal protection (e.g. an effective respirator) shall be used in all cases where exposure to cadmium or cadmium compounds is possible.

1 Scope

This document specifies a method for the determination of the mass concentration of particulate cadmium and cadmium compounds in workplace air, using either flame or electrothermal atomic absorption spectrometry.

The sample digestion procedure specified in 10.2.2 has been validated [2,3] for a selection of cadmium compounds and pigments and glass enamels containing cadmium.

The analytical method has been validated [2] for the determination of masses of 10 ng to 600 ng of cadmium per sample using electrothermal atomic absorption spectrometry, and 0,15 µg to 96 µg of cadmium per sample using flame atomic absorption spectrometry. [3] The concentration range for cadmium in air for which this procedure is applicable is determined in part by the sampling procedure selected by the user.

The method is applicable to personal sampling of the inhalable or respirable fraction of airborne particles, as defined in ISO 7708, and to stationary sampling.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3585, *Borosilicate glass 3.3 — Properties*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 7708, *Air quality — Particle size fraction definitions for health-related sampling*

ISO 8655-1, *Piston-operated volumetric apparatus — Part 1: Terminology, general requirements and user recommendations*

ISO 8655-2, *Piston-operated volumetric apparatus — Part 2: Pipettes*

ISO 8655-5, *Piston-operated volumetric apparatus — Part 5: Dispensers*

ISO 8655-6, *Piston-operated volumetric apparatus — Part 6: Gravimetric reference measurement procedure for the determination of volume*

ISO 13137, *Workplace atmospheres — Pumps for personal sampling of chemical and biological agents — Requirements and test methods*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 18158, *Workplace air — Terminology*

ISO 20581, *Workplace air — General requirements for the performance of procedures for the measurement of chemical agents*

ISO 21832, *Workplace air — Metals and metalloids in airborne particles — Requirements for evaluation of measuring procedures*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <https://www.electropedia.org/>

3.1 exposure by inhalation

situation in which a chemical agent is present in air which is inhaled by a person

3.2 sample dissolution

process of obtaining a solution containing all analytes of interest from a sample, which might or might not involve complete dissolution of the sample

[SOURCE: ISO 15202-2:2020^[10], 3.1]

3.3 sample solution

solution prepared from a sample by the process of *sample dissolution* (3.2)

[SOURCE: ISO 15202-2:2020^[10], 3.2]

3.4 test solution

blank solution or *sample solution* (3.3) that has been subjected to all operations required to bring it into a state in which it is ready for analysis

[SOURCE: ISO 15202-2:2020^[10], 3.3, modified — Note 1 to entry has been deleted.]

4 Principle

4.1 Particulate cadmium and cadmium compounds are collected by drawing a measured volume of air through a sampling substrate (8.2), such as a filter or foam, mounted in a sampler (8.1) designed to collect either the inhalable fraction of airborne particles or the respirable fraction of airborne particles, as appropriate.

4.2 A test solution is prepared by treating the sampling substrate (8.2) and collected sample with 5 ml of nitric acid diluted 1 + 1 (7.3), heating on a hotplate until about 1 ml of concentrated nitric acid (7.2) solution remains, allowing the solution to cool and then diluting to 10 ml with water (7.1).

4.3 The test solution is analysed for cadmium by aspirating into the oxidizing air/acetylene flame of an atomic absorption spectrometer (8.6.5) equipped with a cadmium hollow cathode lamp or electrodeless discharge lamp. Absorbance measurements are made at 228,8 nm and results are obtained by the analytical curve technique.

4.4 For accurate determination when the concentration of cadmium in the solution is low, the analysis can be repeated using electrothermal atomic absorption spectrometry. Aliquots of the test solution and a matrix-modifier solution are injected onto a solid, pyrolytic graphite platform mounted in a pyrolytically coated graphite tube, and after the drying and sample ashing stages the sample is atomized electrothermally. Absorbance measurements are made at 228,8 nm with background correction and results are obtained by the analytical-curve technique.

5 Reactions

In general, the majority of particulate cadmium compounds which are commonly found in samples of workplace air are converted to water-soluble cadmium ions (Cd^{2+}) by the sample digestion procedure specified in [10.2.2](#). However, if there is any doubt about the effectiveness of this procedure for digestion of particulate cadmium compounds which can be present in the test atmosphere, investigate this before proceeding with the method. For instance, other digestion methods described in ISO 15202-2^[10] can be suitable.

6 Requirement

The measuring procedure shall comply with the performance requirements specified in ISO 20581, ISO 21832, and any relevant international, European or national standard which specifies performance requirements for procedures for measuring chemical agents in workplace air.

7 Reagents

During the analysis, use only reagents of analytical grade, and only water as specified in [7.1](#).

7.1 Water, complying with the requirements for ISO 3696 grade 2 water (electrical conductivity less than 0,1 mS/m and resistivity greater than 0,01 M Ω ·m at 25 °C).

The concentration of cadmium in the water shall be less than 0,01 $\mu\text{g/ml}$.

It is recommended that the water used be obtained from a water purification system that delivers ultrapure water having a resistivity greater than 0,18 M Ω ·m (usually expressed by manufacturers of water purification systems as 18 M Ω ·cm).

7.2 Nitric acid (HNO_3), concentrated, $\rho = 1,42 \text{ g/ml}$, 69 % (m/m) to 71 % (m/m).

The concentration of cadmium shall be less than 0,01 $\mu\text{g/ml}$.

WARNING — Concentrated nitric acid is corrosive and oxidizing, and nitric acid fumes are irritant. Avoid exposure by contact with the skin or eyes, or by inhalation of fumes. Personal protective equipment (e.g. gloves, face shield or safety spectacles) shall be used when working with the concentrated or diluted nitric acid, and concentrated nitric acid shall be used in a fume hood.

7.3 Nitric acid, diluted 1 + 1.

Carefully add 500 ml of concentrated nitric acid ([7.2](#)) to 450 ml of water ([7.1](#)) in a 2 l beaker. Swirl to mix, allow to cool and quantitatively transfer to a 1 000 ml one-mark volumetric flask ([8.6.1.7](#)). Dilute to the mark with water, stopper and mix thoroughly.

7.4 Nitric acid, diluted 1 + 9.

Pour approximately 800 ml of water ([7.1](#)) into a 1 000 ml one-mark volumetric flask ([8.6.1.7](#)). Carefully add 100 ml of concentrated nitric acid ([7.2](#)) to the flask and swirl to mix. Allow to cool, dilute to the mark with water, stopper and mix thoroughly.

7.5 Hydrofluoric acid (HF), concentrated, $\rho \approx 1,16$ g/ml (about 48 % mass fraction), if required (see [10.2.2](#)), for digestion of samples containing lead silicates.

The concentration of cadmium in the HF shall be less than 0,1 µg/ml.

WARNING — Concentrated hydrofluoric acid and hydrogen fluoride vapour are extremely toxic and intensely corrosive. Diluted hydrofluoric acid can also cause serious and painful burns, and it is possible that these burns will not be felt until up to 24 h after contact. Avoid exposure by contact with the skin or the eyes, or by inhalation of the vapour. Use of personal protection (e.g. impermeable gloves, face shield or safety glasses) is essential when working with concentrated or diluted hydrofluoric acid, and concentrated hydrofluoric acid should be used in a fume hood. It is essential that hydrofluoric acid antidote gel containing calcium gluconate is readily available to workers, both during and for 24 h after use of hydrofluoric acid.

7.6 Cadmium stock standard solution, corresponding to 1 000 mg of Cd per litre.

7.6.1 Use a commercially available cadmium standard solution at a concentration of 1 000 mg/l. Observe the manufacturer's expiry date or recommended shelf-life.

Alternatively, prepare a cadmium standard solution according to the procedure specified in [7.6.2](#).

7.6.2 Accurately weigh $1\,000\text{ g} \pm 0,001\text{ g}$ of cadmium metal, 99,9 % of Cd, into a 50 ml beaker ([8.6.1.1](#)), add 20 ml of the nitric acid diluted 1 + 1 ([7.3](#)), cover with a watch glass ([8.6.1.4](#)) and heat to approximately 150 °C on the hotplate ([8.6.4](#)) in a fume hood until the metal is completely dissolved. Remove the beaker from the hotplate, allow to cool, quantitatively transfer the solution to a 1 000 ml one-mark volumetric flask ([8.6.1.7](#)), dilute to the mark with water ([7.1](#)), stopper and mix thoroughly.

This solution can be stored in a polypropylene bottle ([8.6.2.2](#)) for up to one year.

7.7 Cadmium working standard solution A, corresponding to 100 mg of Cd per litre.

Using a pipette ([8.6.3.1](#)), accurately add 10,0 ml of stock cadmium solution ([7.6](#)) to a 100 ml one-mark volumetric flask ([8.6.1.7](#)). Add 1 ml of concentrated nitric acid ([7.2](#)), dilute to the mark with water ([7.1](#)), stopper and mix thoroughly.

This solution can be stored in a polypropylene bottle ([8.6.2.2](#)) for up to one month.

7.8 Cadmium working standard solution B, corresponding to 1 mg of Cd per litre.

Using a pipette ([8.6.3.1](#)), accurately add 100 µl of cadmium stock solution ([7.6](#)) to a 100 ml one-mark volumetric flask ([8.6.1.7](#)). Add 1 ml of nitric acid ([7.2](#)), dilute to the mark with water ([7.1](#)), stopper and mix thoroughly.

This solution can be stored in a polypropylene bottle ([8.6.2.2](#)) for up to one month.

7.9 Matrix-modifier solution, corresponding to 10 g of $\text{NH}_4\text{H}_2\text{PO}_4$ per litre of water ([7.1](#)).

Weigh 1,00 g of ammonium dihydrogen phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$) into a 250 ml beaker ([8.6.1.1](#)). Add 50 ml of water ([7.1](#)) and swirl to dissolve. Add 10 ml of concentrated nitric acid ([7.2](#)), swirl to mix, and quantitatively transfer the solution to a 100 ml one-mark volumetric flask ([8.6.1.7](#)). Dilute to the mark with water, stopper and mix thoroughly.

If modifications are needed to optimize the matrix-modifier solution, these modifications should be described in the test report ([Clause 13](#)).

7.10 Laboratory detergent solution, suitable for cleaning samplers ([8.1](#)) and laboratory apparatus, diluted with water ([7.1](#)) according to the manufacturer's instructions.

7.11 Air, high-purity, compressed and filtered.

7.12 Acetylene, in a compressed gas cylinder.

7.13 Argon, supplied in a cylinder or as a cryogenic fluid.

This gas is required if the analysis is carried out by electrothermal atomic absorption spectrometry (see [10.3.4.6](#)).

8 Apparatus

8.1 Samplers, for collection of the inhalable fraction or the respirable fraction of airborne particles (see [9.1.1](#)) as defined in ISO 7708, suitable for use with the sampling substrates ([8.2](#)) and compatible with the sampling pumps ([8.3](#)) used.

NOTE 1 A number of different terms are used to describe samplers designed for collection of the inhalable fraction of airborne particles, for example, sampling heads, filter holders, filter cassettes and air monitoring cassettes (see ISO 15202-1[9]).

NOTE 2 In general, the collection characteristics of inhalable samplers are such that particulate material collected on the filter sampling substrate is the inhalable or respirable fraction of airborne particles, and any deposited on the internal surfaces of the sampler is not of interest. However, some samplers are designed such that airborne particles which pass through the entry orifice(s) constitute the inhalable or respirable fraction; in which case any particulate material deposited on the internal surfaces of the sampler is part of the sample. Certain samplers of this type incorporate an internal filter cassette or cartridge which can be removed from the sampler to enable this material to be easily recovered. See ASTM D8358[4] for further information on inclusion of sampler internal wall deposits.

NOTE 3 Cyclone samplers are typically used for collection of personal samples of the respirable fraction of airborne particles.

NOTE 4 Samplers manufactured in non-conducting material have electrostatic properties which can influence representative sampling. Samplers manufactured from conducting material can reduce electrostatic influences.

8.2 Sampling substrates, such as filters or foams, soluble using the sample digestion procedure specified in [10.2.2](#), and with a retentivity not less than 99 % for particles of median aerodynamic diameter 0,3 µm (see ISO 7708).

The cadmium content shall be less than 0,001 µg per sampling substrate.

NOTE Mixed cellulose ester membrane filters of 0,8 µm to 1,2 µm pore size are generally the most suitable. Cellulose (paper) filters can have a retentivity below 99 % and are therefore unsuitable. Neither glass-fibre nor quartz-fibre filters are dissolved by the sample digestion procedure specified in [10.2.2](#), but this can be modified to permit their use. Furthermore, such filters can have high metal background which can cause interference in highly sensitive analysis.

8.3 Sampling pumps, conforming to the specifications of ISO 13137.

8.4 Flowmeter, portable, capable of measuring the appropriate flow rate (see [9.1.1](#)) to within ±5 %, and calibrated against a primary standard, i.e. a flowmeter of which the accuracy is traceable to national standards.

The calibration of the flowmeter shall be checked against a primary standard, i.e. a flowmeter whose accuracy is traceable to national standards. If appropriate (see [9.1.3.2](#)), record the atmospheric temperature and pressure at which the calibration of the flowmeter was checked.

It is recommended that the flowmeter used be capable of measuring the volumetric flow rate to within ±2 % or better.

8.5 Ancillary equipment

8.5.1 Flexible tubing, of a diameter suitable for making a leak-proof connection from the samplers (8.1) to the sampling pumps (8.3).

8.5.2 Belts or harnesses, to which the sampling pumps (8.3) can conveniently be fixed for personal sampling (except where the sampling pumps are small enough to fit inside worker's pockets).

8.5.3 Flat-tipped forceps, for loading and unloading sampling substrates (8.2) into samplers (8.1).

8.5.4 Transport cassettes, or similar, if required to transport samples for laboratory analysis.

To avoid potential contamination after sample collection, it is advisable to leave sampling substrates (8.2) within the sample holders during transportation of samples.

8.5.5 Barometer, suitable for measurement of atmospheric pressure, if required (see 9.1.3).

8.5.6 Thermometer, minimum temperature range of 0 °C to 50 °C, with graduated divisions of 1 °C or less, for measurement of atmospheric temperature.

For applications at temperatures below freezing, the range of the thermometer shall extend to the appropriate desired range.

A hygrometer can also be used for measurement of relative humidity, if desired.

8.6 Analytical or laboratory apparatus

Ordinary laboratory apparatus, and:

8.6.1 Glassware, made of borosilicate glass, 3.3 complying with the requirements of ISO 3585.

It is preferable to reserve a set of glassware for analysis of cadmium by this method. It is possible that heavily contaminated glassware in general usage will not be satisfactorily cleaned using the procedure specified in 10.1.4.

8.6.1.1 Beakers, capacity 50 ml, for digestion of sampling substrates (8.2), and for preparation of the cadmium stock standard solution (7.6).

If hydrofluoric acid (7.5) is used to assist in sample dissolution, use of plastic heatable beakers (8.6.2.1) that are resistant to corrosion by HF should be used.

8.6.1.2 Beakers, capacity 250 ml, for preparation of the matrix-modifier solution (7.9).

8.6.1.3 Beakers, capacity 2 l, for preparation of nitric acid diluted 1 + 1 (7.3).

8.6.1.4 Watch glasses, to fit the 50 ml beakers (8.6.1.1).

8.6.1.5 Single volume pipettes, complying with the requirements of ISO 648, as an alternative to piston-operated volumetric apparatus (8.6.3).

8.6.1.6 Measuring cylinders, of capacities between 10 ml and 1 l.

8.6.1.7 One-mark volumetric flasks, of capacities between 10 ml and 1 000 ml, complying with the requirements of ISO 1042.

8.6.2 Plastic labware, including the following:

8.6.2.1 Heatable beakers, beaker covers, etc., if required, made of a material that is resistant to corrosion by hydrofluoric acid (7.5); e.g. a fluorocarbon polymer such as polytetrafluoroethylene (PTFE), and suitable for performing dissolutions using hydrofluoric acid.

NOTE If resistance to corrosion by HF is not needed, beakers and beaker covers can be made of glassware (8.6.1).

8.6.2.2 Polypropylene bottles, of capacities from 100 ml to 1 000 ml.

Bottles made of alternative plastics can be used, provided that they are suitable for the intended use (see 7.6.2, 7.6 and 7.7). However, the use of bottles made of coloured plastics should be avoided, since some contain cadmium pigments which can release cadmium when in contact with nitric acid solutions.

8.6.3 Piston-operated volumetric instruments, complying with the requirements of ISO 8655-1 and tested in accordance with ISO 8655-6.

8.6.3.1 Pipetters, complying with the requirements of ISO 8655-2, as an alternative to single volume pipettes (8.6.1.5), for the preparation of standard solutions, calibration solutions and dilution of samples.

8.6.3.2 Dispensers, complying with the requirements of ISO 8655-5, for dispensing acids.

8.6.4 Hot plate, thermostatically controlled, capable of maintaining a surface temperature of approximately 150 °C, for hot plate procedures.

NOTE The efficiency of thermostatted hotplates is sometimes deficient, and the surface temperature can also vary considerably with position on a hotplate with a large surface area. It can therefore be useful to characterize the performance of the hotplate before use.

8.6.5 Atomic absorption spectrometer, fitted with an air-acetylene burner supplied with compressed air (7.11) and acetylene (7.12), and equipped with either a cadmium hollow cathode lamp or electrodeless discharge lamp.

If sample dissolution is carried out with the aid of hydrofluoric acid (7.5) (see 10.2.2), the atomic absorption spectrometer shall be compatible with hydrofluoric acid.

If electrothermal atomic absorption is to be carried out (see 10.3.4.6), the atomic absorption spectrometer shall be capable of carrying out simultaneous background correction at 228,8 nm, either by using a continuous source such as a deuterium lamp to measure non-specific attenuation, or by using Zeeman or Smith-Hieftje background correction systems^[5].

8.6.6 Electrothermal atomiser, fitted with a solid, pyrolytic graphite platform mounted in a pyrolytically coated graphite tube, supplied with argon (7.13) as a purge gas, and equipped with an autosampler capable of injecting microlitre volumes onto the platform.

NOTE Some manufacturers of atomic absorption spectrometers (8.6.5) use an alternative design of electrothermal atomiser to achieve a constant temperature environment during atomisation, and some use aerosol deposition as a means of sample introduction. The use of such accessories is acceptable, but it is possible that the performance of the method will be different from that given in 11.2.

8.6.7 Disposable autosampler cups, made of polystyrene or other suitable plastics, for use in the autosampler used with the electrothermal atomiser (8.6.6).

It is recommended to avoid the use of autosampler cups made of coloured plastics, since some contain cadmium pigments which can release cadmium in contact with nitric acid solutions.

8.6.8 Analytical balance, capable, of weighing to $\pm 0,1 \mu\text{g}$.

8.6.9 Disposable gloves, impermeable, to avoid the possibility of contamination from the hands and to protect them from contact with toxic and corrosive substances. Polyvinyl chloride (PVC) gloves are suitable.

9 Sampling

9.1 Sampling procedure

9.1.1 Collection characteristics and flow rate

Select a sampler ([8.1](#)) suitable for collection of either the inhalable fraction or the respirable fraction of airborne particles, as defined in ISO 7708, and use at the flow rate at which the sampler exhibits the required collection characteristics.

National occupational exposure limits for cadmium and cadmium compounds typically apply to the inhalable or respirable fraction of airborne particles.

NOTE Both inhalable and respirable samplers are typically used at flow rates of around 2 l/min (it is advisable to refer to the manufacturer's recommendations). Some samplers enable flow rates of up to 10 l/min.

9.1.2 Sampling period

Select a sampling period of appropriate duration (2 h minimum), using any available information about the work process and test atmosphere, so that the amount of cadmium collected is within the recommended working range of the method. For example, use a sampling period that relates to the reference period (such as 8 h).

In order to estimate a sampling period of appropriate duration, it is necessary to consider the flow rate used (see [9.1.1](#)) and the anticipated concentration of cadmium in the test atmosphere. When low cadmium-in-air concentrations are anticipated, the lower limit of the working range of the method (see [11.3.2](#)) should be taken into consideration. For example, to determine cadmium in air at a concentration of $0,5 \mu\text{g}/\text{m}^3$ using flame atomic absorption spectrometry, the minimum sampling time at a flow rate of 2 l/min is 8 h. When high cadmium-in-air concentrations are anticipated, the sampling time should not be long enough to risk overloading the sampling substrate ([8.2](#)) with particulate matter.

9.1.3 Temperature and pressure effects

9.1.3.1 Consider whether it is necessary to recalculate the mass concentration of cadmium in air to reference conditions of temperature and pressure in order to comply with national standards and regulations (see ISO 8756^[6]). If appropriate, measure and record the atmospheric temperature and pressure throughout the sampling period (see [9.3.2](#), [9.3.3](#) and [9.3.5](#)) and use the equation given in [11.1.3](#) to apply the necessary correction.

NOTE Cadmium-in-air concentrations are generally stated for the actual environmental conditions (temperature, pressure) at the workplace.

9.1.3.2 The indicated flow rate of certain types of flowmeter is dependent upon temperature and pressure. Therefore, refer to the manufacturer's directions for the particular flowmeter used, and consider whether it is necessary to make a correction to take into account any difference between the atmospheric temperature and pressure at the time of calibration of the flowmeter and at the time of sampling. Make such a correction if it is considered possible that an error of greater than $\pm 5 \%$ will be introduced by not doing so. If a correction is to be made, measure and record the atmospheric temperature and pressure at which the flowmeter ([8.4](#)) was calibrated.

NOTE An example of temperature and pressure correction for the indicated flow rate is given in [11.1.2](#), for a flowmeter of variable area with constant pressure drop.

9.2 Preparation of sampling equipment

Perform the following in an area where cadmium contamination is known to be low.

9.2.1 Clean the samplers ([8.1](#)) before use. Disassemble the samplers, soak in laboratory detergent solution ([7.10](#)), rinse thoroughly with water ([7.1](#)), wipe with absorptive tissue and allow to dry before reassembly.

9.2.2 Load the sampling substrates ([8.2](#)), such as filters or foams, into clean, dry samplers ([8.1](#)) using clean, flat-tipped forceps ([8.5.3](#)). Connect each loaded sampler to a sampling pump ([8.3](#)) using flexible tubing ([8.5.1](#)), ensuring that no leaks can occur. Switch on the sampling pump, attach the calibrated flowmeter ([8.4](#)) to the sampler so that it measures the flow through the sampler inlet orifice(s), and set the appropriate flow rate (see [9.1.1](#)) with an accuracy of ± 5 %. Switch off the sampling pump and seal the sampler with its protective cover or plug to prevent contamination with cadmium during transport to the sampling position.

If necessary, based on the type of sampling pump, warm the pump up (it is recommended to refer to the manufacturer's instructions).

9.3 Collection of samples

9.3.1 For personal sampling, fix the sampler ([8.1](#)) to the lapel of the worker, in the breathing zone and as close to the mouth and nose as is reasonably practicable. Then, either place the sampling pump ([8.3](#)) in a convenient pocket or attach it to the worker in a manner that causes minimum inconvenience, for example, to a belt ([8.5.2](#)) around the waist. For stationary sampling, position the sampler at the sampling site.

NOTE The breathing zone has been defined in ISO 18158 as the space around the worker's face from where he takes his breath. For technical purposes, a more precise definition can be provided, as follows: hemisphere (generally accepted to be 0,3 m in radius) extending in front of the human face, centred on the midpoint of a line joining the ears; the base of the hemisphere is a plane through this line, the top of the head and the larynx.

9.3.2 When ready to begin sampling, remove the protective cover or plug from the sampler ([8.1](#)) and switch on the sampling pump. Record the time at the start of the sampling period and, if the sampling pump has an elapsed time indicator, set this to zero. If appropriate (see [9.1.3.1](#)), measure the atmospheric temperature and pressure at the start of the sampling period using the thermometer ([8.5.6](#)) and barometer ([8.5.5](#)) and record the measured values.

NOTE If desired, relative humidity can be measured by using a hygrometer.

9.3.3 Since it is possible for the sampling substrate ([8.2](#)) to become clogged, monitor the performance of the sampler ([8.1](#)) frequently, a minimum of once per hour. Measure the flow rate with an accuracy of ± 5 % using the calibrated flowmeter ([8.4](#)) and, if appropriate (see [9.1.3.1](#)), measure the atmospheric temperature using the thermometer ([8.5.6](#)) and the atmospheric pressure using the barometer ([8.5.5](#)). Record the measured values.

NOTE Regular observation of the flow-fault indicator is an acceptable means of ensuring that the flow rate of a flow-stabilized sampling pump is maintained satisfactorily, provided that the flow-fault indicator indicates malfunction when the flow rate is outside ± 5 % of the nominal value.

9.3.4 Terminate sampling and consider the sample to be invalid if the flow rate is not maintained to within ± 5 % of the nominal value throughout the sampling period.

9.3.5 At the end of the sampling period (see 9.1.2), measure the flow rate with an accuracy of $\pm 5\%$ using the calibrated flowmeter (8.4), switch off the sampling pump (8.3) and record the flow rate and the time. Also observe the reading on the elapsed time indicator, if fitted, and consider the sample to be invalid if the reading on the elapsed time indicator and the timed interval between switching the sampling pump on and off do not agree to within $\pm 5\%$, since this can suggest that the sampling pump has not been operating throughout the sampling period. Reseal the sampler (8.1) with its protective cover or plug and disconnect it from the sampling pump. If appropriate (see 9.1.3.1), measure the atmospheric temperature and pressure at the end of the sampling period using the thermometer (8.5.6) and barometer (8.5.5) and record the measured values.

9.3.6 Carefully record the sample identity and all relevant sampling data (see Clause 13). Calculate the mean flow rate by averaging the flow-rate measurements taken throughout the sampling period, and, if appropriate (see 9.1.3.1), calculate the mean atmospheric temperature and pressure. Calculate the volume of air sampled, in litres, at atmospheric temperature and pressure, by multiplying the mean flow rate, in litres per minute, by the sampling time, in minutes.

9.3.7 With each batch of 10 samples, submit for analysis at least two unused filters sampling substrates (8.2) from the same lot as used for sample collection. Subject these blank sampling substrates to exactly the same handling procedure as the samples, but do not draw air through them.

9.4 Transportation

Perform the following in an area where cadmium contamination is known to be low.

9.4.1 For samplers (8.1) which collect the required fraction of airborne particles on the sampling substrate (8.2), remove the sampling substrate from each sampler using clean flat-tipped forceps (8.5.3), place in a labelled transport cassette (8.5.4) and close with a lid.

9.4.2 For samplers (8.1) which have an internal cassette, remove the cassette from each sampler, fasten with the transport clip supplied by the manufacturer, and label appropriately.

9.4.3 For samplers (8.1) designed such that airborne particles which pass through the entry orifice(s) constitute the inhalable (or respirable) fraction, but which do not have an internal cassette, and for samplers of the disposable cassette type, transport samples to the laboratory in the samplers in which they were collected.

9.4.4 Transport the transport cassettes (8.5.4) or samplers (8.1) (see 9.4.3) in a container which has been designed to prevent damage to the samples in transit and which has been labelled to assure proper handling.

10 Procedure for analysis

10.1 Cleaning of glassware and polypropylene bottles

10.1.1 Before use, clean all glassware to remove any residual grease or chemicals, by soaking in laboratory detergent solution (7.10) and then rinsing thoroughly with water (7.1).

10.1.2 After initial cleaning (see 10.1.1), clean all beakers used in the sample digestion procedure specified in 10.2.2 with hot nitric acid. Fill to one-third capacity with concentrated nitric acid (7.2), cover with a watch glass (8.6.1.4), heat to approximately 150 °C on the hotplate (8.6.4) in a fume hood for 1 h, allow to cool, and then rinse thoroughly with water (7.1).

10.1.3 After initial cleaning (see [10.1.1](#)), clean all glassware other than beakers used in the sample digestion procedure specified in [10.2.2](#), by soaking in nitric acid diluted 1 + 9 ([7.4](#)) for at least 24 h, and then rinsing thoroughly with water ([7.1](#)).

10.1.4 Thoroughly rinse glassware which has been previously subjected to the entire cleaning procedure specified in [10.1.1](#), [10.1.2](#) and [10.1.3](#), and which has been reserved for analysis of cadmium by this method, first with nitric acid diluted 1 + 9 ([7.4](#)) and then with water ([7.1](#)).

10.1.5 Before use, clean the polypropylene bottle ([8.6.2.2](#)) by soaking in nitric acid diluted 1 + 9 ([7.4](#)) for at least 24 h and then rinsing thoroughly with water ([7.1](#)).

10.2 Preparation of sample solutions and blank test solutions

10.2.1 Open the filter-transport cassettes (see [8.5.4](#)), sampling cassettes (see [9.4.2](#)) or samplers ([8.1](#)) (see [9.4.3](#)) and transfer each sample, including sampling substrate ([8.2](#)) and collected particulate matter, to an individual clean, labelled 50 ml beaker ([8.6.1.1](#)) using clean, flat-tipped forceps ([8.5.3](#)). If the sampler used was of a type in which airborne particles deposited on the internal surfaces of the sampling cassette or sampler forms part of the sample, wash any particulate material adhering to the internal surfaces into the beaker using a minimum volume of water ([7.1](#)). Follow the same procedure for the blank sampling substrate (see [9.3.7](#)).

10.2.2 Add 5 ml of nitric acid diluted 1 + 1 ([7.3](#)) to each beaker, partially cover with a watch glass ([8.6.1.4](#)), and heat to approximately 150 °C on the hotplate ([8.6.4](#)) in a fume hood, until the sampling substrate ([8.2](#)) has dissolved, and the volume has been reduced to approximately 1 ml. Then remove the beakers from the hotplate and allow the solutions to cool.

If glass-fibre or quartz-fibre filters are used, it is necessary to use polytetrafluoroethylene (PTFE) laboratory apparatus and to add hydrofluoric acid ([7.5](#)) to dissolve the filters before addition of nitric acid diluted 1 + 1 ([7.3](#)). In such circumstances, the procedure described in [10.3.1](#) should be modified to prepare calibration solutions in hydrofluoric acid diluted 1 + 9. Furthermore, if test solutions are analysed using flame atomic absorption spectrometry, it is necessary to use a nebulizer that is resistant to attack by hydrofluoric acid solutions.

NOTE The exact temperature of the hotplate is not critical. A temperature of 150 °C is used because it is high enough to enable the liquid to be evaporated at an acceptable rate, whilst not being high enough to evaporate the liquid at such a rate that there is a significant risk of inadvertently evaporating all the solution and baking the residue.

10.2.3 Carefully rinse the watch glass and the sides of each beaker with water ([7.1](#)) and transfer each solution quantitatively to a 10 ml one-mark volumetric flask ([8.6.1.7](#)).

If necessary, remove any undissolved particulate matter by filtering the solution through a cellulose (paper) filter which has been prewashed with nitric acid diluted 1 + 9 ([7.4](#)) and then with water ([7.1](#)).

Finally, dilute to the mark with water ([7.1](#)), stopper and mix thoroughly.

10.3 Analysis by flame atomic absorption spectrometry

10.3.1 General

Laboratory experiments^[2] have shown that flame atomic absorption measurements of cadmium are not affected significantly by variation in nitric acid concentrations with the range 1 + 19 nitric acid to 1 + 3 nitric acid. However, it is good laboratory practice to match sample and standard matrices as far as is reasonably practicable.

10.3.2 Preparation of calibration solutions

Prepare at least six calibration solutions, including a zero-member calibration solution, to cover the range 0 µg of Cd per millilitre to 1 000 µg of Cd per millilitre. Add 20 ml of nitric acid diluted 1 + 1 (7.3) to separate, labelled 100 ml single-volume volumetric flasks (8.6.1.7). Using a pipette (8.6.1.5), accurately add the appropriate volume of the cadmium working standard solution A (7.7) to each flask, dilute to the mark with water (7.1), stopper and mix thoroughly.

Prepare fresh calibration solutions each week at a minimum.

NOTE The range of the set of calibration solutions is given as a guide. The upper limit of the working range depends upon which wavelength is used, and it is also governed by instrumental factors which affect sensitivity and the linearity of the calibration. Accordingly, the range of the set of calibration solutions can be varied as long the response of the spectrometer over the alternative range of concentrations selected complies with the limitations on curvature indicated in 10.3.3.3.

10.3.3 Calibration

10.3.3.1 Set-up the atomic absorption spectrometer (8.6.5) to make absorbance measurements at a wavelength of 228,8 nm using an oxidizing air/acetylene flame. Use background correction to correct for nonspecific attenuation, if appropriate. Follow the manufacturer's recommendations for specific parameters for the operation of instruments.

NOTE Use of the 228,8 nm cadmium line is specified in this document since the only alternative is the 326,1 nm cadmium line, which exhibits much lower sensitivity. The sensitivity, defined as the concentration required to produce a signal of 1 % absorbance or 0,004 4 absorbance units, is about 0,02 µg of Cd per millilitre for the 228,8 nm cadmium line.

10.3.3.2 Adjust the spectrometer zero whilst aspirating the zero-member calibration solution (9.3.1) into the flame. (Repeat this procedure regularly throughout the calibration and readjust the zero if the baseline drifts.) Then aspirate the calibration solutions (10.3.1) into the flame and make absorbance measurements for each solution.

NOTE Use of an autosampler to present the calibration solutions to the atomic absorption spectrometer (8.6.5) can improve the precision of the absorbance measurements and reduce the volume of solution consumed.

10.3.3.3 For instruments controlled by a microprocessor or personal computer, use a suitable algorithm to generate the calibration function. For instruments without this possibility, prepare a calibration graph by plotting the absorbance of the calibration solutions (10.3.1) versus the concentration of cadmium, in micrograms per millilitre, in the respective solutions.

NOTE In general, it is best to work in the linear range of an atomic absorption calibration, where absorbance is proportional to the concentration of cadmium in solution. However, a certain amount of curvature can be tolerated, but ideally the slope of the top 20 % of the calibration curve should be not less than 70 % of the slope of the bottom 20 % calculated in the same manner. If the calibration obtained does not comply with these limitations on curvature, it is advisable to prepare a new set of calibration standards with cadmium concentrations lower than those recommended in 10.3.1.

10.3.4 Determination

10.3.4.1 Adjust the spectrometer zero whilst aspirating the zero-member calibration solution (see 10.3.1) into the flame. Repeat this procedure regularly throughout the determination and readjust the zero if the baseline drifts. Then aspirate the sample solution and blank test solution into the flame and make absorbance measurements for each solution (see 10.2.3). For instruments controlled by a microprocessor or personal computer, use the calibration function (see 10.3.3.3) to calculate the concentration of cadmium in the sample solutions, and obtain a direct read-out of the results in concentration units. For instruments without this possibility, determine the concentration of cadmium in the sample solutions from the calibration graph (see 10.3.3.3).

NOTE Use of an autosampler to present the sample solutions to the atomic absorption spectrometer (8.6.5) can improve the precision of the results and reduce the volume of solution used.

10.3.4.2 Aspirate a mid-range calibration solution after each five to ten samples and make an absorbance measurement. If this indicates that the sensitivity has changed by more than $\pm 5\%$, take one of the following corrective measures: either use the available software facilities of the microprocessor or personal computer to correct for the sensitivity change (re-slope facility); or suspend analysis and recalibrate the spectrometer as specified in 10.3.3.2 and 10.3.3.3. In both cases, reanalyse the sample solutions which were analysed during the period in which the sensitivity change occurred.

10.3.4.3 If low concentrations of cadmium are found, consider using scale expansion, since this can, in some instances, improve the precision of measurements made near the detection limit.

10.3.4.4 If high concentrations of cadmium are found, dilute the sample solutions to bring the concentration within the calibration range (see 10.2.3). Make all dilutions so that the final nitric acid concentration is 1 + 9 (7.4), and record the dilution factor F .

10.3.4.5 Calculate the mean cadmium concentration in the blank test solutions.

10.3.4.6 If the concentration of cadmium in the sample solutions is less than 0,05 $\mu\text{g/ml}$ (see 10.2.3), consider repeating the analysis using electrothermal atomic absorption spectrometry (see 10.4), since this technique gives more precise measurements at low concentrations.

10.4 Analysis by electrothermal atomic absorption spectrometry

10.4.1 General

Cadmium is present at a low level in the environment and therefore it is essential that strict standards of cleanliness are observed to avoid contamination of laboratory apparatus. This is particularly important when carrying out electrothermal atomic absorption spectrometry, since the technique exhibits a very low detection limit. It should be ensured that all glassware is cleaned thoroughly before use in accordance with 10.1, and that autosampler cups (8.6.7) are stored in nitric acid diluted 1 + 9 (7.4) until required.

10.4.2 Preparation of working calibration solutions

10.4.2.1 Prepare a working calibration solution at a concentration of 2,5 ng of Cd per millilitre. Using a pipette (8.6.1.5), accurately add 250 μl of cadmium working standard solution B (7.8) to a 100 ml one-mark volumetric flask (8.6.1.7). Add about 50 ml of water (7.1) and 20 ml of nitric acid diluted 1 + 1 (7.3), and swirl to mix. Allow to cool, dilute to the mark with water (7.1), stopper and mix thoroughly.

Prepare a fresh solution each week at a minimum.

10.4.2.2 Prepare a working calibration blank solution following the procedure in 10.4.2.1, but omitting the 250 μl of cadmium working standard solution B (7.8).

Prepare a fresh solution each week at a minimum.

10.4.3 Calibration and determination

10.4.3.1 Set up the atomic absorption spectrometer (8.6.5) and electrothermal atomiser (8.6.6) to determine cadmium at a wavelength of 228,8 nm, using background correction to correct for non-specific attenuation. Follow the manufacturer's recommendations for specific operating parameters. Additional information can be found in Annex A.

NOTE The operating parameters for electrothermal atomic absorption spectrometry vary considerably between different instruments, much more so than for flame atomic absorption spectrometry. Typical operating parameters are given in [Tables A.1, A.2, and A.3](#). The characteristic mass, defined as the number of picograms required to give 0,004 4 absorbance seconds, is typically about 1 pg for cadmium. This is equivalent to a sample solution concentration of 0,05 ng of Cd per millilitre of cadmium for a 20 µl sample solution injection volume.

10.4.3.2 Program the autosampler to prepare matrix-modified reference solutions, sample solutions and blank test solutions in situ on a pyrolytic graphite platform mounted in the pyrolytically coated graphite tube of the electrothermal atomiser ([8.6.6](#)). Prepare at least six matrix-modified calibration solutions to cover the range 0 ng of Cd per millilitre to 2,5 ng of Cd per millilitre using the working calibration solution (see [10.4.2.1](#)), the working calibration blank solution (see [10.4.2.2](#)) and the matrix-modifier solution ([7.9](#)). Prepare matrix-modified sample and blank test solutions using the sample solutions, blank test solutions (see [10.2.3](#)) and matrix-modifier solution. (See [Table A.3](#) for typical autosampler injection volumes.)

Sample solutions should be diluted (see [10.4.2.6](#)) before analysis by electrothermal atomic absorption spectrometry (see [10.3.4.6](#)), if results obtained by flame atomic absorption spectrometry indicate that the cadmium concentration is above the upper limit of the calibration range for electrothermal atomic absorption spectrometry (see [10.4.2.2](#)).

NOTE 1 The procedure described above can be varied to accommodate the use of electrothermal atomisers of alternative design.

NOTE 2 Matrix-modified calibration and sample solutions can be prepared in one-mark volumetric flasks ([8.6.1.7](#)) as an alternative to preparation in situ using the autosampler.

10.4.3.3 Set up the analytical sequence in the microprocessor or personal computer. Specify an appropriate number of replicate analyses for each solution and insert a calibration blank solution and a mid-range calibration solution after each five to ten sample solutions to monitor for baseline drift and sensitivity change, respectively.

10.4.3.4 Place the working calibration solution (see [10.4.2.1](#)), the working calibration blank solution (see [10.4.2.2](#)), the matrix-modifier solution ([7.9](#)), and the sample solutions in separate acid-washed autosampler cups ([8.6.7](#)), and position as appropriate in the autosampler carousel (see [10.2.3](#)). Analyse the matrix-modified calibration and sample solutions, using the microprocessor or personal computer software to generate a calibration and obtain a direct read-out of sample and blank results in nanograms of cadmium per millilitre.

10.4.3.5 If significant baseline drift is observed during the course of the analysis, or if the sensitivity has changed by more than +5 %, take one of the following appropriate corrective measures: either use the available software facilities of the microprocessor or personal computer to correct the sensitivity change (re-slope facility); or suspend analysis and recalibrate the spectrometer as specified in [10.4.3.4](#). In both cases, reanalyse the sample solutions which were analysed during the period in which the sensitivity change occurred.

10.4.3.6 If concentrations of cadmium above the upper limit of the calibration range (see [10.4.2.2](#)) are found, dilute the sample solutions to bring them within the calibration range, and repeat the analysis. Make all dilutions so that the final nitric acid concentration is 1 + 9 ([7.4](#)), and record the dilution factor *F*.

Alternatively, the cadmium concentration can be brought within the calibration range by using a reduced aliquot of sample solution. See [Table A.2](#).

10.4.3.7 Calculate the mean cadmium concentration in the blank test solutions.

10.5 Estimation of the instrumental detection limit

10.5.1 Estimate the instrumental detection limit under the working analytical conditions, following the procedure specified in [10.5.2](#) and [10.5.3](#). Repeat this exercise whenever these conditions are changed.

10.5.2 Prepare a reference solution at a concentration of 0,01 µg of Cd per millilitre for flame atomic absorption spectrometric analysis and 0,1 ng of Cd per millilitre for electrothermal atomic absorption spectrometric analysis by diluting working cadmium standard solution B ([7.8](#)). Make this dilution so that the final nitric acid concentration is 1 + 9 ([7.4](#)).

10.5.3 Make at least 20 absorbance measurements on the reference solution (see [10.5.2](#)) and calculate the instrumental detection limit using standard statistical methods.

The limit of detection of an atomic absorption method is defined as the concentration of analyte for which the absorbance has a value equal to k times that of the standard deviation of a series of readings measured on a solution, the concentration of which is distinctly detectable above, but close to, that of the blank (k is generally taken as either 2 or 3). For the purposes of this document, k should be taken as 3.

NOTE 2 The limit of detection calculated from results obtained using the procedure specified in [10.5.2](#) and [10.5.3](#) is an instrumental detection limit. This is of use in identifying changes in instrument performance, but it is not a detection limit of the method and is likely to be unrealistically low because it only takes into account the variability between instrumental readings. Determinations made on one solution do not take into consideration variability from the matrix or sample variability. A more realistic detection limit for the analytical procedure specified in this document can be obtained by making measurements on at least 10 blank test solutions, i.e. solutions of blanks (see [9.3.7](#)). The standard deviation of such measurements, made over a longer time interval than between successive calibrations, can be used to obtain an estimate of the detection limit of the method.

11 Expression of results

11.1 Calculations

11.1.1 Calculate the mass concentration of cadmium in the air sample, ρ_{Cd} , in milligrams per cubic metre at ambient conditions, using the formula:

$$\rho_{Cd} = \frac{(\rho_{Cd,1} \times V_1 \times F) - (\rho_{Cd,0} \times V_0)}{V}$$

where

$\rho_{Cd,0}$ is the mean concentration of cadmium, in micrograms per millilitre, in the blank test solutions (see [10.3.4.5](#) or [10.4.3.7](#));

$\rho_{Cd,1}$ is the concentration of cadmium, in micrograms per millilitre, in the sample solution (see [10.3.4.1](#) or [10.4.3.4](#));

V is the volume, in litres, of the air sample;

V_0 is the volume, in millilitres, of the blank test solution; i.e. 10 ml (see [10.2.3](#));

V_1 is the volume, in millilitres, of the sample solution; i.e. 10 ml (see [10.2.3](#));

F is the dilution factor used in [10.3.4.4](#) or [10.4.3.6](#) ($F = 1$ in the absence of dilution).

For low concentrations of cadmium-in-air determined by electrothermal atomic absorption spectrometry, it is recommended to calculate results in micrograms per cubic metre by using solution concentrations in nanograms per millilitre in the above equation.

11.1.2 In some instances, it is necessary to apply a temperature and pressure correction for the indicated sampling flow rate (see 9.1.3.2). A typical example of when such a correction is necessary is when the sampling pump used incorporates a flowmeter of variable area with constant pressure drop, which was calibrated and used to measure the flow rate in 9.3.2, 9.3.3 and 9.3.5. In this instance, use the following formula to calculate the volume of air samples:

$$V_{corr} = q_v \times t \sqrt{\frac{p_1 \times T_2}{p_2 \times T_1}}$$

where

V_{corr} is the corrected volume, in litres;

q_v is the mean flow rate, in litres per minute;

t is the sampling time, in minutes;

p_1 is the atmospheric pressure, in kilopascals, during calibration of the sampling pump flowmeter;

p_2 is the mean atmospheric pressure, in kilopascals, during the sampling period;

T_1 is the temperature, in kelvins, during calibration of the sampling pump flowmeter;

T_2 is the mean temperature, in kelvins, during the sampling period.

It is possible that other flowmeters will also require correction for variation in temperature and pressure. Follow the manufacturer's instructions for such corrections.

11.1.3 If appropriate (see 9.1.3.1), calculate the mass concentration of cadmium in the air sample at reference temperature and pressure (273 K and 101,3 kPa, respectively), $\rho_{Cd,corr}$ using the formula:

$$\rho_{Cd,corr} = \rho_{Cd} \times \frac{101,3 T_2}{p_2 \times 273}$$

where

ρ_{Cd} is the mass concentration of cadmium in the air sample, in milligrams per cubic metre, at ambient conditions, as calculated in 10.1.1;

T_2 is the mean temperature, in kelvins, during the sampling period;

t is the sampling time, in minutes;

p_2 is the mean atmospheric pressure, in kilopascals, during the sampling period;

101,3 is the standard atmospheric pressure, in kilopascals;

273 is the reference temperature, in kelvins.

11.2 Performance of the method

11.2.1 Laboratory experiments indicate that the analytical method does not exhibit significant bias. The mean analytical recovery for spiked cellulosic filters in the range 0,15 µg to 96 µg of cadmium was determined^[2] to be 100,3 % using flame atomic absorption spectrometry; and the mean analytical