

MDHS

*Methods for the Determination of
Hazardous Substances*
Health and Safety Laboratory



29/2

Beryllium and beryllium compounds in air

Laboratory method using flame atomic
absorption spectrometry or electrothermal
atomic absorption spectrometry

April 1996

INTRODUCTION

Note 1: This method updates and replaces MDHS 29.¹ The principal changes which have been made are (i) to recommend the use of filters that are soluble using the dissolution technique described, and (ii) to describe the use of electrothermal atomic absorption spectrometry for the analysis of sample solutions with a low beryllium concentration.

Occurrence, properties and uses

1 Occurrence, properties and uses of beryllium and beryllium compounds are fully covered in HSE Guidance Note EH 13.²

Effects on health

2 The health effects of beryllium and beryllium compounds are summarised in HSE Guidance Note EH 13² and are fully covered in HSE Toxicity Review TR 27.³

Health and safety precautions

3 HSE leaflet MS(A)18⁴ summarises the risks involved in working with beryllium and what can be done to control them. Prevention and control of exposure, emergency procedures and health surveillance are described more fully in HSE Guidance Note EH 13.²

Exposure limits

4 Schedule 1 of the *Control of Substances Hazardous to Health (COSHH) Regulations*⁵ specifies a maximum exposure limit (MEL) of 0.002 mg m⁻³, 8-hour time-weighted average reference period, for beryllium and beryllium compounds (as Be). This limit is reproduced in HSE Guidance Note EH 40⁶ and the criteria on which it was based are documented in the 1994 supplement of HSE Guidance Note EH 64.⁷

Analytical methods

5 This is not a 'reference' method in the strict analytical sense of the word. There are frequently several alternative methods available for the determination of a particular analyte. With the exception of a few cases, where an exposure limit is linked to a specific method (eg rubber fume or asbestos), the use of methods not included in the MDHS series is acceptable provided that they have been shown to have the accuracy and reliability appropriate to the application.

6 This method has been validated⁸ to demonstrate that it complies with the *General requirements for the performance of procedures for the measurement of chemical agents in workplace atmospheres* described by the Comité Européen de Normalization (CEN) in European Standard EN 482.⁹ If an alternative method is used it is necessary to demonstrate that it also meets these performance requirements.

Requirements of the COSHH Regulations

7 The *Control of Substances Hazardous to Health (COSHH) Regulations*⁵ require that employers make an assessment of the health risk created by work involving substances hazardous to health, and to prevent or control exposure to such substances. The COSHH Regulations also include a requirement that persons who may be exposed to substances hazardous to health receive suitable and sufficient information, instruction and training. Employers must ensure that their responsibilities under the COSHH Regulations are fulfilled before allowing employees to undertake any procedure described in this method. Guidance is given in the Approved Codes of Practices for the Control of Substances Hazardous to Health, the *General COSHH ACOP*, and the Control of Carcinogenic Substances, the *Carcinogens ACOP*, which are included in a single publication with the COSHH Regulations.¹⁰

SCOPE

Applicability

8 This MDHS describes a method for determination of the concentration of beryllium and beryllium compounds in workplace air using flame and electrothermal atomic absorption spectrometry. It is applicable to the determination of water-soluble beryllium salts and the majority of beryllium-containing materials in industrial use or occurring in workplace air. The method is suitable for sampling times in the range 2 to 8 hours by flame atomic absorption spectrometry and 15 minutes to 8 hours by electrothermal atomic absorption spectrometry.

Note 2: *HSE Guidance Note EH 42¹¹ advises employers about how they should conduct investigations into the nature, extent and control of exposure to substances hazardous to health which are present in workplace air. The objective of air monitoring is usually to determine worker exposure, and therefore the procedures described in this method are for personal sampling in the breathing zone. The method may be used for background or fixed location sampling, but it should be recognised that, due to aerodynamic effects, samplers designed for personal sampling do not necessarily exhibit the same collection characteristics when used for other purposes.*

METHOD PERFORMANCE

Effectiveness of sample dissolution procedure

9 The sample dissolution procedure has been tested¹² on calcined and uncalcined beryllia samples. It was found to be necessary to use sulphuric acid in addition to nitric acid to effectively dissolve samples of calcined beryllia.

Detection limits

10 The qualitative and quantitative detection limits for beryllium, defined as three times and ten times the standard deviation of a blank determination, have been determined⁸ to be 0.0036 $\mu\text{g ml}^{-1}$ and 0.012 $\mu\text{g ml}^{-1}$ for analysis by flame atomic absorption spectrometry; 0.027 ng ml^{-1} and 0.09 ng ml^{-1} for analysis by nitric acid sample dissolution and electrothermal atomic absorption spectrometry; and 0.099 ng ml^{-1} and 0.33 ng ml^{-1} for analysis by sulphuric acid sample dissolution and electrothermal atomic absorption spectrometry.

For an air sample volume of 240 litres and a sample solution volume of 10 ml this corresponds to beryllium-in-air concentrations of 0.15 $\mu\text{g m}^{-3}$ and 0.5 $\mu\text{g m}^{-3}$ for analysis by flame atomic absorption spectrometry. For an air sample volume of 30 litres and a sample solution volume of 10 ml this corresponds to beryllium-in-air concentrations of 0.009 $\mu\text{g m}^{-3}$ and 0.03 $\mu\text{g m}^{-3}$ for analysis by nitric acid sample dissolution and electrothermal atomic absorption spectrometry; and 0.033 $\mu\text{g m}^{-3}$ and 0.11 $\mu\text{g m}^{-3}$ for analysis by sulphuric acid sample dissolution and electrothermal atomic absorption spectrometry.

Overall uncertainty

11 Laboratory experiments⁸ indicate that the analytical method does not exhibit significant bias. The mean analytical recovery for 140 spiked filters in the range 0.12 μg to 3.84 μg of beryllium was determined to be $101.1 \pm 3.9\%$ using flame atomic absorption spectrometry; and the mean analytical recovery for 100 spiked filters in the range 0.012 μg to 0.192 μg of beryllium was determined to be $100.1 \pm 2.9\%$ using electrothermal atomic absorption spectrometry.

12 The component of the coefficient of variation of the method that arises from analytical variability, CV(analysis), has been determined⁸ to be less than 11% for samples in the range 0.12 μg to 3.84 μg using flame atomic absorption spectrometry; and less than 9% for samples in the range 0.012 μg to 0.192 μg using electrothermal atomic absorption spectrometry.

13 The overall uncertainty of the method, as defined by CEN,⁹ has been estimated⁸ to be less than 27% for samples in the range 0.12 μg to 3.84 μg using flame atomic absorption spectrometry; and less than 23% for samples in the range 0.012 μg to 0.192 μg using electrothermal atomic absorption spectrometry. This assumes that the coefficient of variation of the method that arises from inter-specimen sampler variability, CV(inter), is negligible and that the coefficient of variation of the method that arises from pump flow rate variability, CV(flow), is limited to 5%. The overall uncertainty is therefore within the specifications prescribed by CEN⁹ for measurements for comparison with limit values, ie <50% for measurements in the range 0.1 to 0.5 times the limit value and <30% for measurements in the range 0.5 to 2.0 times the limit value.

Interferences

14 Flame atomic absorption spectrometry is performed at a wavelength of 234.9 nm using a reducing nitrous oxide-acetylene flame. At this wavelength sodium and silicon at concentrations in excess of 1000 $\mu\text{g ml}^{-1}$ have been found to depress beryllium sensitivity. Aluminium at concentrations of 500 $\mu\text{g ml}^{-1}$ also depresses beryllium sensitivity, although this can be overcome by the addition of 8-hydroxyquinoline.

PRINCIPLE

15 A measured volume of air is drawn through a filter mounted in an inhalable dust sampler. The filter and collected sample are then treated with 5 ml of 1 + 1 nitric acid and heated on a hotplate at 150°C until about 1 ml of concentrated nitric acid solution remains. If calcined beryllium oxide is present, 1 ml of concentrated sulphuric acid is added and the beaker is returned to the hotplate at 180°C until dense white fumes are evolved. If the solution turns dark brown at this stage, more concentrated nitric acid is added and heating is continued until the reappearance of dense white fumes. The resultant solution is diluted to 10 ml with water and analysed for beryllium by aspirating into the reducing nitrous oxide-

acetylene flame of an atomic absorption spectrometer at a wavelength of 234.9 nm. For accurate determination when the concentration of beryllium in the solution is low, the analysis may be repeated using electrothermal atomic absorption spectrometry. Aliquots of the sample solution and a matrix modifier solution are injected onto a solid pyrolytic graphite platform mounted in a pyrolytically coated graphite tube, and after drying and ashing stages, the sample is atomised electrothermally. Absorbance measurements are made at 234.9 nm with background correction.

REAGENTS

16 During the analysis, use only reagents of recognised analytical grade. Use only distilled or de-ionised water, or water of equal purity (paragraph 17). Do not pipette by mouth.

Water

17 Water complying with the requirements of BS 3978¹³ grade 2 water (electrical conductivity less than 0.1 mS m⁻¹ and resistivity greater than 0.01 MΩ.m at 25°C).

Nitric acid (HNO₃), concentrated, ρ about 1.42 g ml⁻¹, 69% (m/m) to 71% (m/m)

18 The beryllium concentration of the acid shall be less than 0.001 µg ml⁻¹.

WARNING - Concentrated nitric acid is corrosive and oxidising, and nitric acid fumes are irritant. Avoid exposure by contact with the skin or eyes, or by inhalation of fumes. Personal protection (eg gloves, face shield or safety spectacles etc) should be used when working with concentrated or diluted nitric acid, and sample dissolution with nitric acid should be carried out in a fume cupboard.

Nitric acid, diluted 1 + 1

19 Carefully add 500 ml of concentrated nitric acid (paragraph 18) to 450 ml of water (paragraph 17) in a 2 litre beaker. Mix, allow to cool and quantitatively transfer to a 1000 ml volumetric flask. Dilute to the mark with water and mix thoroughly.

Nitric acid, diluted 1 + 9

20 Carefully add 100 ml of concentrated nitric acid (paragraph 18) to 500 ml of water (paragraph 17) in a 1 litre beaker. Mix, allow to cool and quantitatively transfer to a 1000 ml volumetric flask. Dilute to the mark with water and mix thoroughly.

Sulphuric acid (H₂SO₄), concentrated, ρ about 1.84 g ml⁻¹, about 98% (m/m)

21 The beryllium concentration of the acid shall be less than 0.001 µg ml⁻¹.

WARNING - Concentrated sulphuric acid is corrosive and the fumes are irritant. Avoid exposure by contact with the

skin or eyes, or by inhalation of fumes. Personal protection (eg gloves, face shield or safety spectacles etc) should be used when working with concentrated or diluted sulphuric acid, and sample dissolution with sulphuric acid should be carried out in a fume cupboard.

Sulphuric acid, diluted 1 + 9

22 Carefully add 100 ml of concentrated sulphuric acid (paragraph 21) to 500 ml of water (paragraph 17) in a 1 litre beaker. Mix, allow to cool and quantitatively transfer to a 1000 ml volumetric flask. Dilute to the mark with water and mix thoroughly.

Stock standard beryllium solution, 1000 µg ml⁻¹ of beryllium

23 Use a commercially available standard solution at a concentration of 1000 µg ml⁻¹ of beryllium. Observe the manufacturer's expiry date or recommended shelf life.

Alternatively prepare a stock beryllium standard solution by the following procedure:

24 Accurately weigh 1.000 g of beryllium metal, 99.9% Be (m/m), into a 100 ml beaker, add 20 ml of 1 + 1 nitric acid (paragraph 19), cover with a watch glass and heat on a hotplate (paragraph 32) in a fume cupboard until the metal is completely dissolved. Remove the beaker from the hotplate, allow to cool, quantitatively transfer the solution into a 1 litre volumetric flask, dilute to the mark with water (paragraph 17), stopper and mix thoroughly.

Note 3: *Beryllium standard solution prepared according to the instructions in paragraph 24 may be stored in a plastic bottle (paragraph 35) for a period of one year without deterioration.*

WARNING - Beryllium and beryllium compounds have been assigned various risk phrases in the *Approved Supply List*¹⁴ for the *Chemicals (Hazard Information and Packaging for Supply) Regulations 1994*¹⁵ (the 'CHIP 2' Regulations). Great care should be taken when working with beryllium metal and solutions containing beryllium.

Working standard beryllium solution A, 100 µg ml⁻¹ of beryllium

25 Accurately pipette 10.0 ml of stock beryllium standard solution (paragraph 23 or 24) into a 100 ml volumetric flask. Add 1 ml of concentrated nitric acid (paragraph 18), dilute to the mark with water (paragraph 17), stopper and mix thoroughly. Prepare this solution fresh weekly.

Working standard beryllium solution B, 1.00 µg ml⁻¹ of beryllium

26 Accurately pipette 100 µl of stock beryllium standard solution (paragraph 23 or 24) into a 100 ml volumetric flask. Add 1 ml of concentrated nitric acid (paragraph 18), dilute to the mark with water (paragraph 17), stopper and mix thoroughly. Prepare this solution fresh weekly.

Matrix modifier solution, 0.5% (m/v) Mg(NO₃)₂

27 Weigh 1.02 g of magnesium nitrate, Mg(NO₃)₂·6H₂O, into a 250 ml beaker. Add 50 ml water (paragraph 17) and swirl to dissolve. Add 10 ml of concentrated nitric acid (paragraph 18), swirl to mix, and quantitatively transfer the solution into a 100 ml volumetric flask. Dilute to the mark with water, stopper and mix thoroughly.

Laboratory detergent solution

28 A laboratory grade detergent suitable for cleaning of samplers and laboratory glassware, diluted with water (paragraph 17) according to the manufacturer's instructions.

SAMPLING EQUIPMENT

Samplers for collection of the inhalable fraction of the airborne particles

29 Samplers, with protective covers, for collection of the inhalable fraction of the airborne particles, as defined in European Standard EN 481.¹⁶ Inhalable dust samplers suitable for personal sampling are described in MDHS 14.¹⁷

Note 4: *In general, the collection characteristics of inhalable samplers can be such that particulate material collected on the filter is the inhalable fraction of the 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 fraction, in which case any particulate material deposited on the internal surfaces of the sampler is part of the sample. Samplers of this type incorporate an internal filter cassette which may be removed from the sampler to enable this material to be easily recovered. Refer to the manufacturer's instructions to ascertain what constitutes the inhalable fraction of the sample.*

Note 5: *Samplers manufactured in non-conducting material have electrostatic properties which may influence representative sampling. Electrostatic influences should be reduced, where possible, by using samplers manufactured from conducting material.*

Filters

30 Filters, of a diameter suitable for use in the samplers (paragraph 29), with a retentivity of not less than 99.5% for particles with a 0.3 µm diffusion diameter. The use of filters that are soluble using the sample preparation procedure described is recommended, and mixed cellulose ester filters of 0.8 µm mean pore diameter are considered to be most suitable.

The beryllium content shall be less than 0.001 µg per filter.

Note 6: *Glass fibre or other filters which do not dissolve using the sample preparation procedure described may be used, but extra care needs to be taken to ensure*

quantitative transfer of sample solutions to volumetric flasks (paragraph 63).

Sampling pumps

31 Sampling pumps, complying with the provisions of draft European Standard prEN 1232,¹⁸ with an adjustable flow rate, incorporating a flowmeter or a flow fault indicator, capable of maintaining the selected flow rate (see paragraph 42) to within ±5% of the nominal value throughout the sampling period (see paragraph 43), and capable of being worn by persons without impeding normal work activity. The pumps shall give a pulsation-free flow (if necessary, a pulsation damper shall be incorporated between the sampler and the pump, as near to the pump as possible). Flow-stabilised pumps may be required to maintain the flow rate within the specified limits.

Flowmeter

32 Flowmeter, portable, capable of measuring the appropriate flow rate (see paragraph 42) to within ±1%, and calibrated against a primary standard.

Note 7: *Flowmeters incorporated in sampling pumps are not suitable for accurate measurement of the flow rate. However, they can be useful for monitoring the performance of samplers (see paragraph 47), provided they have adequate sensitivity.*

Ancillary equipment

33 Flexible plastic tubing, of a diameter suitable for ensuring a leakproof fit, to connect the sampler to the pump; a belt to which the pump can conveniently be fixed, unless the pump is sufficiently small to fit in the worker's pocket; flat-tipped tweezers for loading and unloading the filters into samplers; and filter transport cassettes, or similar, to transport filters to the laboratory, if transport in the samplers is impracticable.

LABORATORY APPARATUS

Glassware, made of borosilicate glass

34 A selection of laboratory glassware: including beakers; watch glasses; measuring cylinders; and one-mark volumetric flasks, class A, complying with the requirements of BS 1792.¹⁹

Note 8: *It is recommended that a set of glassware is reserved for the analysis of beryllium by this method (see paragraph 58).*

Polypropylene bottle

35 A polypropylene bottle, with leakproof screw cap, for storage of stock standard solution (paragraph 24), cleaned before use by soaking in 1 + 9 nitric acid (paragraph 20) for at least 24 hours and then rinsing thoroughly with water (paragraph 17). A bottle made of an alternative plastic may be used provided that it is suitable for the intended use.

Hotplate

36 A thermostatically controlled hotplate, capable of maintaining the required surface temperatures.

Disposable gloves

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

Piston operated volumetric apparatus

38 A set of adjustable micropipettes, complying with the requirements of BS 7653-1 to BS 7653-4,²⁰⁻²³ for the preparation of solutions for calibration of the atomic absorption spectrometer (paragraphs 64 and 73) and dilution of samples (paragraphs 70 and 80). A suitable set might include micropipettes covering the ranges 10 µl to 100 µl, 100 µl to 1000 µl and 1000 µl to 5000 µl.

Atomic absorption spectrometer

39 An atomic absorption spectrometer, fitted with a nitrous oxide-acetylene burner, supplied with nitrous oxide and acetylene, and equipped with a beryllium hollow cathode lamp.

Electrothermal atomiser

40 An electrothermal atomiser, fitted with a solid, pyrolytic graphite platform mounted in a pyrolytically-coated graphite tube, supplied with argon as a purge gas, and equipped with an autosampler capable of injecting microlitre volumes onto the platform.

Note 9: Some manufacturers of atomic absorption spectrometers 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 the method performance could be different from that described in paragraphs 10 to 13.

Disposable autosampler cups

41 Disposable polystyrene autosampler cups for use in the autosampler used with the electrothermal atomiser. Soak in 1 + 9 nitric acid (paragraph 20) before use.

Note 10: Disposable polystyrene autosampler cups are also useful for containing solutions to be pipetted in microlitre quantities.

SAMPLING

Sampling procedure

42 Use the samplers (paragraph 29) at the design flow rate, so that they exhibit the required collection characteristics. Refer to the manufacturer's instructions.

43 Select a suitable sampling time, such that the filter does not become overloaded with aerosol. (An 8-hour time weighted average concentration may be derived from the results for two or more consecutive samples, as described in Guidance Note EH 42.¹¹)

Preparation of sampling equipment

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

44 Clean the samplers (paragraph 29) before use. Disassemble the samplers, soak in laboratory detergent solution (paragraph 28), rinse thoroughly with water (paragraph 17), wipe with absorptive tissue and allow to dry thoroughly before reassembly. Alternatively, use a laboratory washing machine.

45 Load the filters (paragraph 30) into clean, dry samplers (see paragraph 44) using clean, flat-tipped tweezers (paragraph 33). Connect each loaded sampler to a sampling pump (paragraph 31) using plastic tubing (paragraph 33), ensuring that no leaks can occur. Switch on the pump, attach the calibrated flowmeter (paragraph 32) to the sampler so that it measures the flow through the sampler inlet orifice, and set the appropriate flow rate (see paragraph 42) with an accuracy of $\pm 5\%$. Switch off the pump and seal the sampler with its protective cover to prevent contamination with beryllium during transport to the sampling position.

Note 11: It might be necessary to allow the pump to operate for an appropriate period to enable it to warm up and the flow rate to stabilise (refer to the manufacturer's recommendations). If this is the case, discard the used filter after the warm-up period and load a new one into the sampler for collection of the sample. Then attach the calibrated flowmeter again and readjust the flow rate to the appropriate value (see paragraph 42) with an accuracy of $\pm 5\%$.

Collection of samples

46 Fix the sampler to the lapel of the worker, in the breathing zone and as close to the mouth and nose as practicable. Then, either place the sampling pump in a convenient pocket or attach it to the worker in a manner that causes minimum inconvenience, eg to a belt (paragraph 33) around the waist. When ready to begin sampling, remove the protective cover from the sampler and switch on the pump. Record the time at the start of the sampling period, and if the pump is equipped with an elapsed time indicator, set this to zero.

47 Since it is possible for a filter to become clogged, monitor the performance of the sampler frequently, a minimum of once per hour. Measure the flow rate with an accuracy of $\pm 5\%$ using the calibrated flowmeter (paragraph 32) and record the measured value. Terminate sampling and consider the sample to be invalid if the flow rate is not maintained to within $\pm 5\%$ of the nominal value throughout the sampling period.

Note 12: Regular observation of the flow fault indicator is an acceptable means of ensuring that the flow rate of flow-stabilised sampling pumps is maintained satisfactorily, provided that the flow fault indicator indicates malfunction when the flow rate is outside $\pm 5\%$ of the nominal value.

48 At the end of the sampling period (see paragraph 43), measure the flow rate with an accuracy of $\pm 5\%$ using the calibrated flowmeter (paragraph 32), switch off the sampling pump, 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 on and switching off the sampling pump do not agree to within $\pm 5\%$, since this may suggest that the sampling pump has not been operating throughout the sampling period. Reseal the sampler with its protective cover and disconnect it from the sampling pump.

49 Carefully record the sample identity and all relevant sampling data (see Appendix A). Calculate the mean flow rate by averaging the flow rate measurements taken throughout the sampling period and calculate the volume of air sampled, in litres, by multiplying the flow rate in litres per minute by the sampling time, in minutes.

50 With each batch of ten samples, submit for analysis two unused filters from the same lot of filters used for sample collection. Subject these blank filters to exactly the same handling procedure as the samples, but draw no air through them.

Transportation

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

51 For samplers which collect the inhalable fraction of airborne particles on the filter (see note 4), remove the filter from each sampler using clean flat-tipped tweezers (paragraph 33), place in a labelled filter transport cassette (paragraph 33) and close with a lid.

52 For samplers which have an internal filter cassette (see note 4), remove the filter cassette from each sampler, fasten with the transport clip supplied by the manufacturer, and label appropriately.

53 For samplers designed such that airborne particles which pass through the entry orifice(s) constitute the inhalable fraction but which do not have an internal filter cassette (see note 4), and for samplers of the disposable cassette type, transport the samples to the laboratory in the samplers in which they were collected.

54 Transport the filter transport cassettes (see paragraph 51), sampler filter cassettes (see paragraph 52) or samplers (see paragraph 53) to the laboratory in a container which has been designed to prevent damage to samples in transit and which has been labelled to assure proper handling.

ANALYSIS

Wear disposable gloves (paragraph 37) during analysis to protect the hands from corrosive and oxidising reagents.

Cleaning of glassware

55 Before use, clean all glassware (paragraph 34) to remove any residual grease or chemicals. Firstly soak overnight in laboratory detergent solution (paragraph 28) and then rinse thoroughly with water (paragraph 17). Alternatively, use a laboratory washing machine.

56 After initial cleaning (paragraph 55), clean all beakers used in the sample dissolution procedure (paragraphs 61 and 62) with hot nitric acid. Fill to one third capacity with concentrated nitric acid (paragraph 18), cover with a watch glass, heat to approximately 150°C on the hotplate (paragraph 36) in a fume cupboard for 1 hour, allow to cool, and then rinse thoroughly with water (paragraph 17).

57 After initial cleaning (paragraph 55), clean all glassware other than beakers used in the sample dissolution procedure by soaking in 1 + 9 nitric acid (paragraph 20) for at least 24 hours and then rinsing thoroughly with water (paragraph 17).

58 Glassware which has been previously subjected to the cleaning procedure described in paragraphs 55 to 57, and which has been reserved for determination of beryllium by this method, can be adequately cleaned by rinsing thoroughly with 1 + 9 nitric acid (paragraph 20) and then with water (paragraph 17).

Preparation of sample and blank solutions

59 Open the filter transport cassettes (see paragraph 51), sampler filter cassettes (see paragraph 52) or samplers (see paragraph 53) and transfer each filter into an individual, labelled 50 ml beaker using clean flat-tipped tweezers (paragraph 33). Follow the same procedure for the blank filters (paragraph 50).

60 If the sampler used was of a type in which airborne particles deposited on the internal surfaces of the filter cassette or sampler form part of the sample (see note 4), wash any particulate material adhering to the internal surfaces into the beaker using a minimum volume of water (paragraph 17).

61 Add 5 ml of 1 + 1 nitric acid (paragraph 19) to each beaker, partially cover with a watch glass, and heat to approximately 150°C on a hotplate (paragraph 36) in a fume cupboard until the filter has dissolved and the solution has been reduced to approximately 1 ml. Remove each beaker from the hotplate and allow to cool. If calcined beryllium oxides are not present, proceed from paragraph 63 without the addition of sulphuric acid.

62 If calcined beryllium oxides could be present, add 1 ml of concentrated sulphuric acid (paragraph 21) to each beaker, cover with a watch glass, and heat to approximately 180°C on a hotplate (paragraph 36) in a

fume cupboard until white fumes are evolved (the volume should be 1 ml). If the solution turns brown during this stage, due to charring of residual organic material, carefully add a further 1 ml of concentrated nitric acid (paragraph 18), and continue heating until the reappearance of dense white fumes. Remove each beaker from the hotplate and allow to cool.

63 Carefully rinse the watch glass and the sides of each beaker with water (paragraph 17), and quantitatively transfer each solution to an individual, labelled 10 ml volumetric flask. If necessary, remove any undissolved particulate matter by filtering through a cellulose (paper) filter which has been pre-washed with 1 + 9 nitric acid (paragraph 20) and then with water (paragraph 17). Finally, dilute to the mark with water, stopper and mix thoroughly.

Analysis by flame atomic absorption spectrometry

Note 13: *Laboratory experiments⁸ have shown that flame atomic absorption measurements of beryllium are not affected significantly by variation in the nitric acid concentration within the range 1% to 25% (v/v). However, it is good laboratory practice to match sample and calibration solution matrices as far as is reasonably practicable.*

Preparation of calibration solutions

64 Prepare at least six calibration solutions to cover the range 0 $\mu\text{g ml}^{-1}$ to 1.0 $\mu\text{g ml}^{-1}$ of beryllium. Add 50 ml of water (paragraph 17) to separate, labelled 100 ml volumetric flasks. Carefully add 10 ml of concentrated nitric acid (paragraph 18) or 10 ml of concentrated sulphuric acid (paragraph 21) to each flask, according to whether sulphuric acid was used in the dissolution procedure (see paragraph 62). Accurately pipette the appropriate volume of working standard beryllium solution A (paragraph 25) into each flask and swirl to mix. Allow to cool, dilute to the mark with water (paragraph 17), stopper and mix thoroughly. Prepare these solutions fresh weekly.

Atomic absorption measurements

65 Set up the atomic absorption spectrometer (paragraph 39) to determine beryllium at a wavelength of 234.9 nm using a reducing nitrous oxide-acetylene flame. Follow the manufacturer's recommendations for specific operating parameters, and use background correction. The sensitivity, defined as the concentration required to produce a signal of 1% absorbance or 0.0044 absorbance units, is about 0.025 $\mu\text{g ml}^{-1}$ of beryllium.

66 Adjust the spectrometer zero while aspirating the 0 $\mu\text{g ml}^{-1}$ calibration solution (paragraph 64). Repeat this procedure regularly throughout the analysis and readjust the zero if the baseline drifts.

67 Aspirate the calibration solutions (paragraph 64) into the flame in order of increasing concentration and make absorption measurements for each solution. For instruments controlled by a microprocessor or personal computer, generate a calibration for beryllium by carrying out a linear regression. For instruments without this

capability, prepare a calibration graph by plotting the absorbance of the calibration solutions versus the beryllium concentration.

68 Aspirate the sample and blank solutions (paragraph 63) into the flame and make absorption measurements for each solution. For instruments controlled by a microprocessor or personal computer, use the calibration function to determine the concentration of beryllium in the sample and blank solutions and obtain a direct read-out of the results in $\mu\text{g ml}^{-1}$ of beryllium. For instruments without this capability, determine the concentration of beryllium in $\mu\text{g ml}^{-1}$ from the calibration graph (paragraph 67).

69 Aspirate a mid-range calibration solution into the flame after each five to ten sample solutions and make an absorption measurement. If this indicates that the sensitivity has changed by more than $\pm 5\%$, take one of the following appropriate corrective measures: either use the available software facilities of the microprocessor or personal computer to correct for the sensitivity change (reslope facility); or suspend analysis, recalibrate the spectrometer as described in paragraph 67, and in either case reanalyse the solutions which were analysed during the period in which the sensitivity change occurred.

70 If high concentrations of beryllium are found, dilute the sample solutions to bring the concentration within the calibration range, and repeat the analysis. Make all dilutions so that the final nitric acid or sulphuric acid concentration is 1 + 9, according to whether sulphuric acid was used in the dissolution procedure (see paragraph 62). Record the dilution factor.

71 Calculate the mean beryllium concentration of the blank solutions.

72 If the concentration of beryllium in the sample solutions is less than 0.01 mg ml^{-1} consider repeating the analysis using electrothermal atomic absorption spectrometry (see paragraphs 73 to 81) since this technique gives more precise measurements at low concentrations.

Analysis by electrothermal atomic absorption spectrometry

Note 14: *It is essential that strict standards of cleanliness are observed to avoid contamination of labware when carrying out electrothermal atomic absorption spectrometry, since the technique exhibits a very low detection limit. Ensure that all glassware is cleaned thoroughly before use in accordance with paragraphs 55 to 58, and that autosampler cups (paragraph 41) are stored in 1 + 9 nitric acid (paragraph 20) until required.*

Preparation of working calibration solutions

73 Prepare a working calibration solution at a concentration of 2.5 ng ml^{-1} of beryllium. Add 50 ml of water (paragraph 17) to a labelled 100 ml volumetric flask. Carefully add 10 ml of concentrated nitric acid (paragraph 18) or 10 ml of concentrated sulphuric acid (paragraph 21) to the flask, according to whether or not sulphuric acid was

used in the dissolution procedure (see paragraph 62). Accurately pipette 0.25 ml of working standard beryllium solution B (paragraph 26) into the flask and swirl to mix. Allow to cool, dilute to the mark with water (paragraph 17), stopper and mix thoroughly. Prepare this solution fresh daily.

74 Prepare a working calibration blank solution following the procedure in paragraph 73, but omitting the 0.25 ml of working standard beryllium solution B.

Atomic absorption measurements

75 Set up the atomic absorption spectrometer (paragraph 39) and electrothermal atomiser (paragraph 40) to determine beryllium at a wavelength of 234.9 nm using background correction. Follow the manufacturer's recommendations for specific operating parameters.

Note 15: *The operating parameters for electrothermal atomic absorption spectrometry vary considerably between different instruments, much more so than for flame atomic absorption spectrometry. A Perkin-Elmer 5100PC atomic absorption spectrometer with Zeeman HGA-600 graphite furnace module and AS-60 autosampler was used in the validation of this method,⁸ and the operating parameters used are given in Appendix B. The characteristic mass for beryllium, defined as the number of picograms required to give 0.0044 absorbance-seconds, was determined to be 1.0 pg for this analytical system. This is equivalent to a sample solution concentration of 0.05 ng ml⁻¹ of beryllium for a 20 µl sample solution injection volume.*

76 Program the autosampler to prepare matrix-modified calibration, sample solutions and blank solutions in situ on a pyrolytic graphite platform mounted in the pyrolytically-coated graphite tube of the electrothermal atomiser. Prepare at least six matrix-modified calibration solutions to cover the range 0 ng ml⁻¹ to 2.5 ng ml⁻¹ using the working calibration solution (paragraph 73), the working calibration blank solution (paragraph 74) and the matrix modifier solution (paragraph 27). Prepare matrix-modified sample and blank solutions using the sample and blank solutions (paragraph 63) and the matrix modifier solution (paragraph 27). See Table 2 for typical autosampler injection volumes.

Note 16: *The procedure described above may be varied to accommodate the use of electrothermal atomisers of alternative design (see note 9).*

Note 17: *Matrix-modified calibration and test solutions may be prepared in volumetric flasks as an alternative to preparation in situ using the autosampler.*

Note 18: *Sample test solutions should be diluted (see paragraph 80) before analysis by electrothermal atomic absorption spectrometry (see paragraph 72) if results obtained by flame atomic absorption spectrometry indicate that the beryllium concentration is above the upper limit of the calibration range for electrothermal atomic absorption spectrometry (see paragraph 76).*

77 Set-up the analytical sequence in the micro-processor 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.

78 Place the working calibration solution (paragraph 73), the working calibration blank solution (paragraph 74), the matrix modifier solution (paragraph 27), and the sample and blank solutions (paragraph 63) in separate acid-washed autosampler cups (paragraph 41) and position as appropriate in the autosampler carousel. Analyse the matrix-modified calibration, sample and blank solutions, using the microprocessor or personal computer software to generate a calibration and obtain a direct read-out of sample and blank results in ng ml⁻¹ of beryllium.

79 If significant baseline drift is observed during the course of the analysis, or if the sensitivity changes 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 for the sensitivity change (reslope facility); or suspend analysis and recalibrate the spectrometer as described in paragraph 78. In either case reanalyse the solutions which were analysed during the period in which the sensitivity change occurred.

80 If concentrations of beryllium above the upper limit of the calibration range 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 or sulphuric acid concentration is 1 + 9, according to whether sulphuric acid was used in the dissolution procedure (see paragraph 62). Record the dilution factor.

81 Calculate the mean beryllium concentration of the blank solutions.

QUALITY CONTROL MEASURES

82 Analytical quality requirements, guidance on the establishment of a quality assurance programme and details of internal quality control and external quality assessment schemes are fully described in MDHS 71.²⁴

83 If beryllium analysis is performed frequently it is recommended that internal quality control is performed. In such instances, prepare quality control filters by spiking a large batch of filters with microlitre volumes of a solution of known beryllium concentration. Analyse a random selection of at least 20 filters, each along with a different analytical batch, and calculate the mean value and standard deviation of the readings. Assuming that the distribution of these values is Gaussian, construct a Shewhart chart with warning and action limits at ±2SD and ±3SD respectively. Subsequently, analyse a quality control filter with each analytical batch and plot the result on the Shewhart chart. Compare the internal quality control result with the target value and take appropriate action if the warning or action limits are exceeded, as recommended in MDHS 71.²⁴ Take care to ensure that the quality control filters are stored under conditions which ensure maximum stability.

84 It is strongly recommended that all laboratories undertaking the determination of toxic elements in workplace air should participate in an external quality assessment scheme such as HSE's Workplace Analysis Scheme for Proficiency (WASP). Details of WASP are given in MDHS 71.²⁴ However, at present the WASP scheme does not encompass beryllium.

CALCULATIONS

Volume of air sample

85 Calculate the mean flow rate during the sampling period by averaging the flow rate measurements taken at the start and end of the sampling period. Then calculate the volume, in litres, of the air sample by multiplying the mean flow rate, in litres per minute, by the sampling time, in minutes.

Concentration of beryllium in air

86 Calculate the concentration of beryllium in air, $\rho(\text{Be})$, in milligrams per cubic metre (mg m^{-3}), using the equation:

$$\rho(\text{Be}) = \frac{[\rho(\text{Be})_1 \cdot V_1 \cdot \text{DF}_1 - (\text{Be})_0 \cdot V_0 \cdot \text{DF}_0]}{V}$$

where $\rho(\text{Be})_0$ is the mean concentration, in $\mu\text{g ml}^{-1}$, of beryllium in the blank solutions (see paragraphs 71 and 81);

$\rho(\text{Be})_1$ is the concentration, in $\mu\text{g ml}^{-1}$, of beryllium in the sample solution (see paragraphs 68 and 78);

V is the volume, in litres, of the air sample (see paragraph 85);

V_0 is the volume, in ml, of the blank solutions, ie 10 ml (see paragraph 63);

V_1 is the volume, in ml, of the sample solution, ie 10 ml (see paragraph 63);

DF_0 is the dilution factor for the blank solutions, ie 1; and

DF_1 is the dilution factor for the sample solutions (see paragraphs 70 and 80).

TEST REPORT

87 Appendix A gives recommendations for information to be included in the test report.

APPENDIX A Recommendations for the test report

It is recommended that the test report should include the following information:

- (a) a complete identification of the air sample, including the date of sampling, the place of sampling, and the identity of the individual whose breathing zone was sampled;
- (b) a reference to this MDHS and a description of any deviation from the procedures described;
- (c) the type and diameter of filter used;
- (d) the type of sampler used;
- (e) the type of sampling pump used;
- (f) the type of flowmeter used, the primary standard against which it was calibrated, and the range of flow rates for which the flowmeter was calibrated;
- (g) the time at the start and at the end of the sampling period, and the sampling time in minutes;
- (h) the volume of air sampled, in litres;
- (i) the name of the person who collected the sample;
- (j) the time-weighted average mass concentration of beryllium found in the air sample, in milligrams per cubic metre;
- (k) the name of the analyst;
- (l) the date of the analysis.

APPENDIX B Typical operating parameters for determination of beryllium by electrothermal atomic absorption spectrometry

Mode: Peak area
 Integration time: 5 seconds
 Background correction: Zeeman
 Injection volumes: 20 μl of calibration, sample or blank solution and 10 μl of matrix modifier solution

Table 1 Typical temperature profile for determination of beryllium using electrothermal atomic absorption spectrometry

Step	Ramp time (sec)	Hold time (sec)	Furnace temp ($^{\circ}\text{C}$)	Argon flow (ml min^{-1})	Read
1 Dry	50	10	125	300	
2 Ash	25	5	1500	300	
3 Cool down	1	15	20	300	
4 Atomise	0	5	2600	0	*
5 Clean	1	5	2700	300	

Table 2 Typical autosampler injection volumes for the in-situ preparation of matrix-modified calibration, sample and blank solutions

Matrix-modified solution	Volume of working calibration solution (μl)	Volume of working calibration blank solution (μl)	Volume of matrix modifier solution (μl)	Volume of sample or blank solution (μl)
0 ng ml^{-1} calibration solution	-	20	10	-
0.5 ng ml^{-1} calibration solution	4	16	10	-
1.0 ng ml^{-1} calibration solution	8	12	10	-
1.5 ng ml^{-1} calibration solution	12	8	10	-
2.0 ng ml^{-1} calibration solution	16	4	10	-
2.5 ng ml^{-1} calibration solution	20	-	10	-
Sample or blank solution	-	-	10	20
Sample solution dilution	-	(20 - x)	10	x

The beryllium concentrations in the matrix-modified calibration solutions are notional, in that they relate to the 20 μl volume of sample or blank solution (ie the 10 μl volume of matrix-modifier solution is ignored).

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ADVICE

Advice on this method and the equipment used can be obtained from the Health and Safety Executive, Health and Safety Laboratory, Broad Lane, Sheffield, S3 7HQ (telephone 0114 289 2000).

The Health and Safety Executive wishes, wherever possible, to improve the methods described in this series. Any comments that might lead to improvements would therefore be welcome and should be sent to the above address.

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