

# MDHS

## *Methods for the Determination of Hazardous Substances*

Health and Safety Laboratory



# 86

## Hydrazine in air

Laboratory method using sampling either onto acid-coated glass fibre filters followed by solvent desorption or into specially constructed impingers. Final analysis by derivatisation and HPLC

August 1997

### INTRODUCTION

#### Requirements of the COSHH regulations

1 The Control of Substances Hazardous to Health (COSHH) Regulations 1994<sup>1</sup> are designed to ensure that the exposure of people at work to substances which could cause damage to health is either prevented, or where this is not reasonably practicable, adequately controlled. Employers are required to make an assessment of the health risk created by such work, and to prevent or control exposure to the substances involved. The COSHH Regulations also require that people 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 MDHS.<sup>1</sup>

#### Properties and uses

2 Hydrazine is a colourless liquid, chemical formula  $N_2H_4$ . In the UK it is almost always found as some form of hydrazine hydrate, an aqueous solution. Commonly, solutions of 35% w/w and 64% w/w are to be found. It is a highly reactive reducing agent, decomposing slowly in air, and is strongly adsorbed onto most solid surfaces. Although none is manufactured in the UK, approximately 1000 tonnes are imported annually. Around half of that amount is used as an oxygen scavenger in boiler feedwater in the electricity generation industry. Most of the remainder is used in chemical synthesis, mainly in the agrochemical and pharmaceutical industries.

#### Toxicity

3 The toxicity of hydrazine has been reviewed by HSE.<sup>2</sup> Hydrazine is readily absorbed by all routes of exposure. It is rapidly metabolised and excreted from the body. The target organs following both single and repeated exposure are the liver, kidney and central nervous system. Hydrazine is irritating to the skin, eyes and respiratory tract and is a skin sensitiser. It has been shown to be genotoxic in mammalian cell *in vitro*; the potential for genotoxicity *in*

*vivo* has not been adequately investigated. It has been shown to be carcinogenic in animals and this carcinogenic potential is considered to be relevant to humans, although there are inadequate human data to confirm this.

4 Readers should be aware of the carcinogen classification awarded to hydrazine, its aqueous solutions and its salts under the Chemicals (Hazard Information and Packaging for Supply) (CHIP) regulations<sup>3</sup> when working with these substances.

#### 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 (eg other methods in the MDHS series). 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 validated<sup>4</sup> 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 Normalisation (CEN) in the European Standard prEN 482.<sup>5</sup> If an alternative method is used it is necessary to demonstrate that it also meets these performance requirements.

#### Quality control

7 An appropriate method of quality control should be employed when using this method. For guidance see MDHS 71.<sup>6</sup> Analyse freshly prepared quality control samples as a quality check.

#### SCOPE

8 Two sampling methods are described for airborne hydrazine. Both are suitable for the determination of time-weighted average concentration of hydrazine vapour in

workplace and environmental atmospheres. The first method, sampling onto acid-coated glass fibre filters, is applicable to sampling over periods of a few minutes up to two hours. Under no circumstances should samples be taken over periods of greater than two hours since it is possible for significant losses due to oxidation to occur, leading to a false, low result. This method is suitable for personal sampling. The alternative sampling method, using specially constructed impingers, is applicable to long-term sampling, up to 8 hours. However, this technique may be unsuitable for personal sampling since the liquid is easily spilled from the sampler.

**Detection limits**

9 Both methods are suitable for the measurement of hydrazine vapour in the concentration range 0.002 to 2 ppm using the recommended sampling conditions. The actual detection limit for an individual sample is dependent on the sample volume.

**Overall uncertainty**

10 The overall uncertainty for a measuring procedure is defined in BS EN 482<sup>7</sup> as ‘the quantity used to characterise as a whole the uncertainty of the result given by a measuring procedure’, and is quoted as a percentage combining bias and precision using the following equation:

$$\text{Overall uncertainty} = \frac{|\bar{x} - x_{\text{ref}}| + 2s}{x_{\text{ref}}} \times 100$$

where:

$\bar{x}$  is the mean result obtained from a number *n* of repeated measurements;

$x_{\text{ref}}$  is the true or reference result;

*s* is the standard deviation of the results

11 The above equation may be used to calculate overall uncertainty of a method providing a reference method exists against which the test method may be compared. No reference method was available in this case; however, sufficient data were generated in the method development stages by sampling from standard atmospheres to calculate overall uncertainty values for both the filter and impinger techniques. Typical overall uncertainty for both methods was found to be within 15%, and never exceeded 25% when sampling from standard atmospheres. This is sufficient to demonstrate compliance with BS EN 482.

**Interferences**

12 The most likely interferent is ammonia, which is used in close proximity to hydrazine in the power industry. These methods are not affected by the presence of ammonia at concentrations normally found in the working environment. The effects of substituted hydrazines (monomethyl hydrazine (MMH) and unsymmetrical dimethyl hydrazine (UDMH)) have not been investigated. However, these compounds are rarely encountered in the UK.

**PRINCIPLE**

13 *Method 1 (filter sampling).* Air is drawn through a phosphoric acid-coated glass fibre filter at a measured flow rate for a known time. Upon completion of sampling the filter is desorbed into 1.0 ml of 0.1M sulphuric acid.

14 *Method 2 (impinger sampling).* Air is drawn through a specially modified impinger containing 10 ml of 0.1M sulphuric acid. Upon completion of sampling the volume of liquid in the impinger is topped up to 10 ml with de-ionised water if necessary. 1.0 ml of the acid is then removed from the impinger for analysis.

15 In either case the analysis procedure is identical. Benzaldehyde is added to the sample solution which reacts with any hydrazine in the sample (when heated to 80°C for one hour in the presence of a basic buffer) to form benzalazine. This compound is detected by HPLC.

**APPARATUS**

**Filter method**

16 Filter holder - Delrin filter holders, obtained from Gelman, were used for filter sampling. The open faced, chemically inert nature of this type of filter holder is necessary for hydrazine sampling.

17 Filters - binder-free glass fibre filters (eg Whatman GF/A), 25 mm diameter are suitable.

**Impinger method**

18 Impingers - specially modified impingers are required for hydrazine sampling. These are made from standard 25 ml midget impingers, 1-2 mm i.d. Teflon tubing and a suitable gas-tight sealant, such as PTFE tape. A schematic diagram of such an impinger is shown in Figure 1.

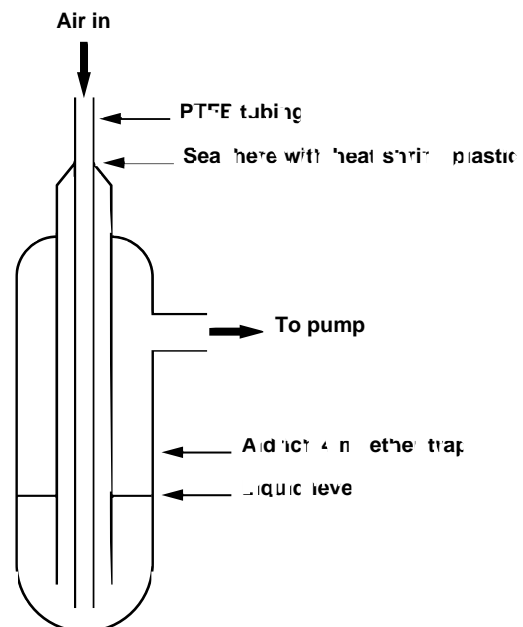


Figure 1: Impinger modified for hydrazine sampling

## General

### Sampling pumps

19 Sampling pumps, with an adjustable flow rate, incorporating a flow-meter or flow fault indicator, capable of maintaining the appropriate flow rate (paragraph 34) to the level of accuracy specified in MDHS 14<sup>8</sup> or its successors throughout the sampling period, 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 sampling head 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.

### Flow-meter

20 Flow-meter, portable, capable of measuring the appropriate flow rate to the level of accuracy specified in MDHS 14<sup>8</sup> or its successors, and calibrated against a primary standard.

### Laboratory apparatus

#### Glassware

21 A selection of laboratory glassware, including beakers; pasteur pipettes; 4 ml vials (with screw-top caps and teflon-silicone septa); and volumetric flasks, class A, complying with the requirements of BS1792<sup>9</sup> and class B.

#### Disposable gloves

22 PVC or natural rubber disposable gloves are suitable.

## Balance

23 A balance, calibrated against a primary standard, for the preparation of the internal standard solution and calibration standards. The balance should be capable of weighing to  $\pm 0.1$  mg over the range 0 to 100 g.

### Heater block

24 A heater block, capable of maintaining samples (in 4 ml vials) at 80°C for 30 minutes.

### HPLC system

25 An HPLC system fitted with a uv/vis spectrophotometric detector is required. Chromatographic conditions are as follows:

Mobile phase - 70% acetonitrile/30% water  
@ 2.5 ml/min  
Column type - 5 micron, reverse phase C18,  
15 cm long, 4.6 mm internal diameter  
Injection volume - 20  $\mu$ l  
Detector wavelength - 313 nm

26 Since the filter samples contain particulate matter (loose fibres from the filters) it is recommended to fit the HPLC system with a pre-column filter to prolong column life.

27 With the HPLC parameters set up as above, the excess reagent (benzaldehyde) peak elutes after approximately one minute, the hydrazine (benzalazine) peak elutes after approximately 2.5 minutes. The peaks should be well resolved; if this is not the case renew the column. A total run time of 4 minutes is sufficient. A sample chromatogram is shown in Figure 2.

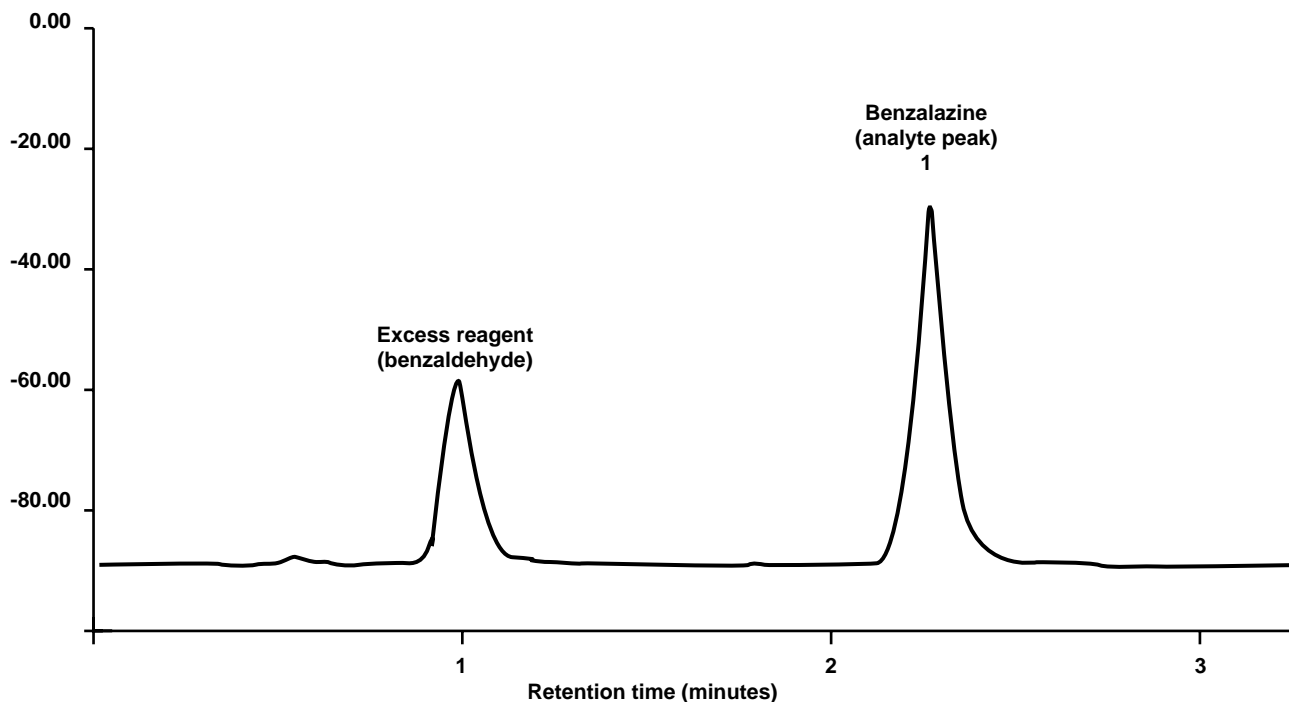


Figure 2: Typical chromatogram from hydrazine sample

## REAGENTS

### Phosphoric acid in acetonitrile

28 Dilute 0.5 ml phosphoric acid (85% aqueous solution) to 25 ml with acetonitrile.

### 0.1M sulphuric acid

29 Dilute 2.7 ml concentrated sulphuric acid to 500 ml with water.

### Reagent solution

30 Dilute 0.5 ml benzaldehyde to 100 ml with methanol.

### Buffer solution

31 Dissolve 2 g sodium tetraborate in 100 ml water (a magnetic stirrer is useful at this stage since the sodium tetraborate takes a while to dissolve at room temperature).

## PROCEDURE

### Preparation of sampling equipment

#### *Filter preparation*

32 Lay out clean filters, in a fume cupboard, so that they are supported with a minimum amount of their surface area in contact with any solid surface. With 25 mm GF/A filters this may be achieved by resting the filter on the top rim of a standard 10 ml laboratory beaker. Add 0.25 ml of the phosphoric acid in acetonitrile solution to each filter and allow 30 minutes for the solvent to evaporate. The filters are then ready for use. If kept clean, the acid-coated filters may be stored for several weeks before use.

#### *Impinger preparation*

33 Remove the lower portion of the glass stem from a standard 25 ml midjet impinger and replace it with Teflon tubing, which is sealed in place with PTFE tape. On no account must the air sample pass through any glass tubing before bubbling through the collecting solution, since this can lead to significant errors due to hydrazine adsorbing onto the glass. Place 10 ml of 0.1M sulphuric acid in the impinger body for sample collection. A diagram of a modified impinger is shown in Figure 1.

#### *Pump preparation*

34 Samples are taken using battery operated portable sampling pumps producing a smooth airflow (see paragraph 19). Check the flow rate before and after sampling, ideally using a bubble flow-meter, although a rotameter is acceptable. The flow rate for filter sampling should be  $2.0 \pm 0.1$  litres/min. The back pressure due to the filters is negligible, therefore the flow rate may be set without a sampler in place. The flow rate for impinger sampling should be  $1.0 \pm 0.05$  litres/min. Again, this may be set without the sampler in place.

## Collection of samples

### *Filter sampling*

35 Attach an acid-coated filter, mounted in an appropriate sampling head, to each pump with a length of flexible tubing. If personal sampling is being performed, the filters should be mounted on the sample subject's lapel, as close as possible to the breathing zone. The sampling period should not exceed two hours under any circumstances. Do not allow filters to be exposed to test atmospheres before sampling commences since it is possible for the samplers to collect hydrazine diffusively without the aid of a pump. For this reason, samplers should be sealed before and immediately after sampling, using an appropriate cap. The filters are not suitable for sampling in extremely dry air, although they have been shown to perform adequately at humidities as low as 20% RH.

### *Impinger sampling*

36 The acid-filled, Teflon stemmed impinger is connected to a sampling pump by a length of flexible tubing. This sampling arrangement is best suited to static sampling, since the liquid is easily spilled from the impinger.

### *Blanks*

37 A minimum of three field blanks should be included with a batch of samples. This is applicable to both sampler types.

### Preparation of calibration standards

38 Calibration standards are prepared from hydrazine sulphate, a stable, solid salt of hydrazine containing 24.6% w/w hydrazine. A range of calibration solutions may be prepared by weighing a known amount of hydrazine sulphate into a volumetric flask and diluting with 0.1M  $H_2SO_4$ . It will usually be necessary to make one or more further dilutions of this concentrated solution before a solution of suitable strength to prepare a range of calibration standards from is achieved. In order to make the calculations for calibration standards it is necessary to know that for hydrazine 1 ppb = 1.3 ng/litre (at 20°C, atmospheric pressure). When an appropriate range of calibration standards has been prepared, transfer 1.0 ml of each to a 4 ml, clear glass, screw top septum vial and proceed in the same manner as for the actual samples.

39 As a salt of hydrazine, hydrazine sulphate carries the same carcinogen classification under the CHIP and COSHH regulations (see paragraphs 1, 3 and 4 of this document).

## Analysis

### *Filter samples*

40 Remove the filters from the sampling heads using tweezers. Avoid handling the filter as far as is possible.

Place each filter in a 4 ml, clear glass, screw top septum vial and desorb by adding 1.0 ml of 0.1M H<sub>2</sub>SO<sub>4</sub> to each vial. (If the samples are not to be analysed immediately, the vials may be sealed at this stage. In this state the samples may be stored at room temperature for up to 4 weeks. Undesorbed filters are not stable for more than a few hours.)

### **Impinger samples**

41 Upon completion of sampling it may be necessary to make the liquid in the impinger back to the original volume (10 ml, using de-ionised water) if significant evaporation losses have occurred. Take 1.0 ml of the solution from the impinger and place in a 4 ml, clear glass, screw top septum vial.

42 The remainder of the analysis procedure is common to samples taken using either technique. Add 1.0 ml of reagent (benzaldehyde in methanol) to each vial, followed by 1.0 ml of buffer solution. Seal the vials with screw tops fitted with PTFE-lined septa, and heat the samples at 80°C for one hour. The samples are then cooled back to room temperature and analysed by HPLC. The amount of hydrazine in each sample may be calculated by referencing against the calibration factor determined by analysis of a series of calibration standards, prepared as described above. It is important not to shake the vials containing filter samples at any time since this will disturb the loose fibres from the filters. These will damage the HPLC column (or prematurely block the pre-column filter, if fitted) if they are injected into the system, causing a deterioration in peak shape, resolution and sensitivity. It should also be noted that excess acidity inhibits the reaction between hydrazine and benzaldehyde. A significant peak on the chromatogram, eluting before benzaldehyde, is evidence that this has occurred.

### **Blanks**

43 These should be analysed identically to samples.

## **CALCULATIONS**

### **Calibration factor**

44 Plot a graph of HPLC peak area (y-axis) against hydrazine concentration (µg/ml) in calibration standard (x-axis). The slope of the best fit line is equal to the calibration factor.

### **Samples**

45 Multiply the peak area from the sample by the calibration factor. This gives the hydrazine concentration, in µg/ml, in the sample solution. Since filters are desorbed into 1.0 ml of liquid this is the absolute amount of hydrazine in the air sample. However, for impinger samples this value must be multiplied by 10 since 1 ml of liquid is taken for analysis from the 10 ml which were actually used to take the sample.

46 Divide the absolute amount of hydrazine (in µg) by the sample volume in litres. This gives the hydrazine concentration of the atmosphere sampled, in µg/l. Divide this value by 1.3 to convert to ppm.

### **Detection limits**

47 Limits of detection and quantification may be calculated from the blanks using the following formula:

Limit of detection (LOD) = mean blank value + 3 \* standard deviation

Limit of quantification (LOQ) = mean blank value + 10 \* standard deviation

## **TEST REPORT**

48 Appendix 2 gives recommendations for information to be included in the test report.

### **APPENDIX 1 - Suppliers of equipment**

25 mm open face Delrin filter holder - Gelman Sciences Ltd, 10 Harrowden Road, Brackmills, Northampton, NN4 0EZ.

Whatman 25 mm GF/A filters, Phase Sep Nucleosil Columns, 1.6 mm i.d. Teflon tubing (for modifications to impingers) - Fisher Scientific Equipment, Bishop Meadow Road, Loughborough, Leicestershire, LE11 0RG.

25 ml glass impingers - SKC Ltd, Unit 11 Sunrise Park, Higher Shaftesbury Road, Blandford Forum, Dorset, DT11 8ST.

### **APPENDIX 2 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 (for personal samples) 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 of sampler used;
- (d) the type of sampling pump and flow-meter used, the primary standard against which it was calibrated, and the range of flow rates for which the flow-meter was calibrated;
- (e) the duration of the sampling period in minutes and/or the time at the start and at the end of the sampling period;
- (f) the volume of air sampled, in litres;

- (g) the name of the person who collected the sample;
- (h) the time-weighted average hydrazine concentration found in the air sample, in ppm and/or the mass of hydrazine collected by the sampler, in micrograms;
- (i) the overall uncertainty of the method;
- (j) the name of the analyst;
- (k) the date of the analysis.

#### ADVICE

Advice on the method and equipment used in it can be obtained from the Health and Safety Laboratory, Broad Lane, Sheffield, S3 7HQ (tel: 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.

#### REFERENCES

- 1 Health and Safety Executive *General COSHH ACoP (Control of Substances Hazardous to Health) and Carcinogens ACoP (Control of Carcinogenic Substances) and Biological Agents ACoP (Control of Biological Agents). Control of Substances Hazardous to Health Regulations 1994: Approved Codes of Practice* (6th edition) HSE Books 1997 ISBN 0 7176 1308 9
- 2 Health and Safety Executive *Hydrazine. Criteria document for an Occupational Exposure Limit* EH65/28 HSE Books 1996 ISBN 0 7176 1099 3

3 Health and Safety Commission *Approved guide to the classification of substances and preparations dangerous for supply: Chemicals (Hazard Information and Packaging for Supply) Regulations 1994: Guidance on Regulations* HSE Books 1994 ISBN 0 7176 0860 3\*

4 Keen C and Pengelly MI *Development of a method for measuring airborne hydrazine* HSE internal report IR/L/SP/95/07 1995

5 Comité Européen de Normalisation *Workplace atmospheres - General requirements for the performance of procedures for the measurement of chemical agents* prEN 482 CEN 1992\*

6 Health and Safety Executive *Methods for the Determination of Hazardous Substances. Analytical quality in workplace air monitoring* MDHS 71 HSE Books 1991 ISBN 0 11 885976 5\*

7 British Standards Institution *Workplace atmospheres - General Requirements for the performance of procedures for the measurement of chemical agents* BS EN 482 ISBN 0 580 23644 7

8 Health and Safety Executive *Methods for the Determination of Hazardous Substances. General methods for sampling and gravimetric analysis of respirable and total inhalable dust* MDHS 14/2 HSE Books 1997 ISBN 0 7176 1295 3\*

9 British Standards Institution *Specification for one-mark volumetric flasks* BS 1792 BSI 1993 ISBN 0 580 12754 0

\* These documents are amended occasionally. Readers should ensure that they are using the current edition.



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|------|---|------|--|
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| 10/2 | Cadmium AA  | 58   | Mercury vapour   |
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| 12/2 | Chromium AA   | 60   | Mixed hydrocarbons   |
| 13   | Chromium XRF  | 61   | Total hexavalent chromium compounds in air colorimetric  |
| 14   | Total inhalable and respirable dust gravimetric   | 62   | Aromatic carboxylic acid anhydrides  |
| 15   | Carbon disulphide charcoal tube/GC  | 63   | Butadiene diffusive/thermal desorption/GC  |
| 16   | Mercury adsorbent tube (Hydrar) AA  | 64   | Toluene charcoal diffusive/solvent desorption/GC   |
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| 21   | Glycol ethers charcoal tube/GC  | 69   | Toluene diffusive/solvent desorption/GC  |
| 22   | Benzene thermal desorption/GC   | 70   | General methods for sampling airborne gases and vapours  |
| 23   | Glycol ethers thermal desorption/GC   | 71   | Analytical quality in workplace air monitoring   |
| 24   | Vinyl chloride charcoal tube/GC   | 72   | Volatile organic compounds in air  |
| 25/2 | Organic isocyanates reagent bubbler/HPLC  | 73   | Measurement of air change in factories and offices   |
| 26   | Ethylene oxide charcoal tube/GC   | 74   | n-Hexane in air diffusive/solvent desorption/GC  |
| 27   | Diffusive sampler evaluation protocol   | 75   | Aromatic amines solid sorbent/thermal desorption/GC  |
| 28   | Chlorinated hydrocarbons charcoal tube/GC   | 76   | Cristobalite in respirable dusts X-ray diffraction (direct method)                             |
| 29/2 | Beryllium AA  | 77   | Asbestos in bulk materials   |
| 30/2 | Cobalt AA   | 78   | Formaldehyde diffusive/solvent desorption/liquid chromatography                                |
| 31   | Styrene pumped thermal desorption/GC  | 79   | Peroxodisulphate salts mobile phase ion chromatography   |
| 32   | Phthalate esters solvent desorption/GC  | 80   | Volatile organic compounds diffusive/thermal desorption/GC                                     |
| 33   | Adsorbent tube standards  | 81   | Dustiness of powders and materials   |
| 34   | Arsine colorimetric (diethyldithiocarbamate)  | 82   | The dust lamp  |
| 35   | HF and fluorides ion-selective electrode  | 83   | Resin acids GC   |
| 36   | Toluene charcoal tube/GC  | 84   | Oil mist from mineral oil-based metalworking fluids  |
| 37   | Quartz in respirable airborne dust direct infra-red   | 85   | Triglycidyl isocyanurate in air pumped filter/desorption/liquid chromatography                 |
| 38   | Quartz in respirable airborne dust KBr disc technique   |      |  |
| 39/4 | Asbestos fibres light microscopy (European reference version)                                     |      |  |
| 40   | Toluene thermal desorption/GC   |      |  |
| 41/2 | Arsenic AA  |      |  |
| 42/2 | Nickel AA   |      |  |
| 43   | Styrene diffusive/thermal desorption/GC   |      |  |
| 44   | Styrene diffusive/solvent desorption/GC   |      |  |
| 45   | Ethylene dibromide solvent desorption/GC  |      |  |
| 46   | Platinum AA   |      |  |
| 47   | Rubber fume in air measured as total particulates and cyclohexane soluble material                |      |  |
| 48   | Newspaper print rooms: measurements of total particulates and cyclohexane soluble material in air |      |  |

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