

MDHS

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



33/2

Sorbent tube standards

Preparation by the syringe injection
technique

February 1997

INTRODUCTION

Requirements of the COSHH Regulations

1 The Control of Substances Hazardous to Health (COSHH) Regulations¹ require that persons who may be exposed to substances hazardous to health receive suitable and sufficient information, instruction and training. The COSHH Regulations also include a requirement to assess the health risk created by work involving substances hazardous to health, and to prevent or control exposure to such substances. Employers must therefore ensure that the requirements of the COSHH Regulations are fully satisfied before allowing employees to undertake any procedure described in this method.

Alternative methods

2 A number of methods are available for the preparation of sorbent tube standards. The use of methods not included in the MDHS series is acceptable provided they have the accuracy and reliability appropriate to the application.

PRINCIPLE

3 Sorbent tubes are loaded with known amounts of organic compounds by injecting a known volume of a standard solution onto the sorbent. The solution is delivered by means of a microlitre syringe either directly onto the sorbent or onto a glass wool prefilter or via a gas chromatograph injection port, while simultaneously pulling air or inert gas through the tube. In the latter two cases, the organic compound is volatilised before reaching the sorbent.

4 There are two main procedures whereby standard sorbent tubes can be prepared. In one, the dynamic loading method, a standard vapour-in-air atmosphere is

generated at known concentration, and a known volume of this atmosphere is transferred to the sorbent tube. In the second, a dilution of the organic compound in solvent is prepared, and a known volume of this solution is injected by microlitre syringe into the sorbent tube. Some studies on the desorption efficiencies and analytical precisions of tubes prepared by various methods are summarised in Tables 1 and 2. Overall, the methods give similar results. In this recommendation, syringe loading is preferred to dynamic loading as being more convenient. In the syringe-loading method, the organic compound can be injected either directly as a liquid, or as a gas after volatilisation; the latter is preferred as the organic compound is transferred to the sorbent in vapour form. A further study² has shown that the performance of thermal desorption tube standards is unaffected by the presence of the excess of diluting solvent, in this case, methanol.

SCOPE AND FIELD OF APPLICATION

5 This procedure describes the preparation of sorbent tube standards to be used for calibration, determination of desorption efficiency and quality control in connection with sorbent tube/gas chromatographic methods for the determination of the time-weighted average concentrations of volatile organic compounds in workplace air. The method is suitable for a wide variety of volatile organic compounds of which those listed in paragraph 7 are examples.

REAGENTS

6 Use only reagents of recognised analytical grade.

Volatile organic compounds

7 The method is suitable for a range of volatile organic compounds, either singly or in mixture. Three

Table 1 Desorption efficiency and precision of method (charcoal/solvent desorption tubes)

<i>Loading method</i>	<i>Compounds</i>	<i>DE range (%)</i>	<i>CV mean* (%)</i>	<i>Source ref</i>
dynamic	9 solvents in NIOSH PAT scheme		3.1 ± 1.4	Hagmann (4) PAT rounds 46-51
dynamic	9 solvents in NIOSH PAT scheme	97 - 100**		PAT report, rounds 84-87
dynamic	12 solvents	73 - 99	2.9 ± 1.1	Krajewski (5)
injection of pure liquid	12 solvents	88 - 105	4.2 ± 1.6***	Krajewski (5)
injection of dilution	12 solvents	87 - 99	3.2 ± 1.6	Krajewski (5)
dynamic	7 solvents	91 - 97	6.0 ± 1.8	Larkin (6)
injection of dilution	toluene	98.9	0.7	MDHS 33/1 (7)
dynamic	benzene, toluene, xylene	83 - 104	2.2 ± 0.6	ISO (8)
dynamic	5 chlorinated solvents	88 - 102	4.5 ± 2.0****	ISO (9)

* range is ±1 standard deviation of the CV values for each solvent

** omitting ethoxyethyl acetate (86%)

*** three high values, ascribed to low injection volumes, are excluded from mean

**** one high value (low level CCl₄) excluded from mean

Table 2 Precision of method (thermal desorption tubes)

<i>Loading method</i>	<i>Sorbent</i>	<i>Compounds</i>	<i>CV range (%)</i>	<i>CV mean* (%)</i>	<i>Source ref</i>
dynamic	Tenax TA	benzene, toluene, xylene: 1 µg	1.8 to 2.6	2.1 ± 0.4	BCR (10)
injection of dilution as liquid	Tenax GR	3 chlorinated solvents plus toluene	0.5 to 14.2	3.4 ± 4.6	BCR (11)
injection of dilution as vapour	Tenax GR	3 chlorinated solvents plus toluene	0.5 to 3.3	1.4 ± 0.9	BCR (11)
dynamic	Tenax GR	3 chlorinated solvents plus toluene	0.6 to 4.9	1.9 ± 1.2	BCR (11)
injection of dilution as vapour	Tenax TA	as para 6: 10 µg, ATD-50	0.5 to 10.7**	2.0 ± 1.1	MDHS 72 (12)
injection of dilution as vapour	Chromosorb 106	16 solvents: 1 µg, ATD-50	1.3 to 5.9	3.1 ± 1.3	EC project (13)
injection of dilution as vapour	Carboxen 569	propane, methanol: 1 µg, ATD-50	1.8, 1.7		EC project (13)
injection of dilution as vapour	Chromosorb 106	benzene, tetrachloroethylene, styrene: 10 µg, ATD-400	0.9 to 1.3	1.0 ± 0.1	Table 3

* range is ±1 standard deviation of the CV values for each solvent

** one high value (for hexane) is excluded from mean

organic solvent mixtures are given as examples, which are arranged to give resolved peaks when the sorbent tube standards are analysed on either BP-1 or BP-10 columns; other mixtures may be more appropriate on other columns.

Note 1: Benzene is a recognised human carcinogen. Avoid any exposure by inhalation or skin contact.

Note 2: n-Hexane presents a serious health hazard if incorrectly handled. Avoid any exposure by inhalation or skin contact.

Mixture 1

8 These components are: n-hexane, n-heptane, n-octane, n-decane, n-undecane, n-dodecane, benzene, toluene, o-xylene, p-xylene, n-propylbenzene, iso-propylbenzene, o-ethyltoluene, m-ethyltoluene, p-ethyltoluene, 1,2,4-trimethylbenzene, 1,3,5-trimethylbenzene, n-propyl acetate, n-butylacetate, iso-butyl acetate, methoxyethyl acetate, butoxyethyl acetate.

Mixture 2

9 These components are: iso-propanol, iso-butanol, n-butanol, ethoxyethanol, methoxyethanol, propylene glycol monomethyl ether, butoxyethanol, 1,2,3-trimethylbenzene, ethylbenzene, ethyl acetate, ethoxyethyl acetate, toluene.

Mixture 3

10 These components are: acetone, 2-butanone, methyl isobutyl ketone, cyclohexanone, 2-methylcyclohexanone, 3-methylcyclohexanone, 4-methylcyclohexanone, iso-propyl acetate, isophorone, n-nonane, toluene.

Methanol

11 This should be of chromatographic quality. It must be free from compounds co-eluting with the calibration compounds. Other solvents may be used, such as carbon disulphide (for charcoal tubes, but not for Tenax) and cyclohexane.

Sorbent

One or more of the following, or equivalent.

For solvent desorption

12 Activated coconut shell charcoal, particle size 0.1-0.8 mm (20-40 mesh) should be used. The charcoal is activated in an inert atmosphere at 600°C for several hours. Suitable tubes prepacked with activated charcoal are available commercially. Other typical sorbents include silica gel, Carbotrap, Carbosieve S-III and Carboxen 564.

For thermal desorption

13 A porous polymer, carbon molecular sieve or graphitised carbon sorbent, particle size 0.18-0.25 mm (60-80 mesh) should be used. The sorbent should be preconditioned by heating in an inert atmosphere at a

temperature at least 25°C below the published maximum for that sorbent for 16 hours before packing the tubes. To prevent recontamination of the sorbent, it should be kept in a clean atmosphere during its cooling to room temperature, storage, and loading into the tubes. Typical sorbents include Carbotrap, Carbopack, Carbosieve S-III, Carboxen 569, Carboxen 1000, Chromosorb 102, Chromosorb 106, Porapak N, Porapak Q, Spherocarb, Tenax TA and Tenax GR.

APPARATUS

Ordinary laboratory apparatus and:

Sorbent tubes

For solvent desorption

14 Typically, but not exclusively, they are constructed of glass tubing, with both ends flame-sealed: 70 mm long and 6 mm OD and 4 mm ID, containing two sections of suitable sorbent (paragraph 12) separated by a 2 mm portion of polyurethane foam. The first section contains 100 mg charcoal and a back-up section 50 mg. A 3 mm portion of polyurethane foam is placed between the outlet end of the tube and the back-up section. A plug of silanised glass wool is placed in front of the first section. The pressure drop across the tube must be less than 3 kPa at an airflow of 1 l.min⁻¹.

For thermal desorption

15 These tubes should be compatible with the thermal desorption apparatus to be used. Typically, but not exclusively, they are constructed of stainless steel tubing, 6.3 mm (1/4 inch) OD, 5 mm ID and 90 mm long. One end of the tube is marked, for example by a scored ring about 10 mm from the end. The tubes are packed with preconditioned sorbent (paragraph 13) so that the sorbent bed will be within the desorber heated zone. Tubes contain typically about 200-300 mg porous polymer or 300-500 mg carbon molecular sieve or graphitised carbon sorbent. The sorbents are retained by stainless steel gauzes and/or unsilanised glass wool plugs. If more than one sorbent is used in a single tube, the sorbents should be arranged in order of increasing sorbent strength and separated by unsilanised glass wool, with the weakest sorbent nearest to the marked end of the tube. If tubes are packed with unconditioned sorbent, they must be conditioned at a temperature 10-25°C below the published maximum for that sorbent (or for the least stable sorbent in a multi-bed tube), for at least 2 hours under a flow of at least 100 ml.min⁻¹ inert gas. Tubes prepacked by the manufacturer are available for most sorbents and as such only require conditioning. After conditioning, tubes should be analysed to ensure that the thermal desorption blank is sufficiently small. If the blank is unacceptable, tubes should be reconditioned. Once a standard tube has been analysed, it may be reused for the preparation of a new standard immediately. However, it is advisable to check the thermal desorption blank if the tubes are left for an extended period before reuse, or if loading with a different analyte is envisaged. Tubes should be sealed and stored in an airtight container.

Note 3: The sorbent tube blank level is acceptable if it is no greater than the equivalent of 100 ng for any of the calibration compounds (paragraph 7). Typical levels are much less than this.

Sorbent tube end caps

16 Tubes for solvent desorption should be sealed with plastic, not rubber, seals. Thermal desorption tubes should be sealed with metal screw-cap fittings with PTFE seals.

Injection facility for preparing standards

17 A conventional gas chromatographic injection port may be used for preparing thermal desorption tube standards. This can be used in situ, or it can be mounted separately. The carrier gas line to the injector should be retained. The back of the injection port should be adapted if necessary to fit the sample tube. This can be done conveniently by means of a compression coupling with a PTFE ring seal.

Guided plunger syringe

18 Precision syringe, 5 μl , readable to 0.1 μl , or a 10 μl syringe, readable to 0.2 μl , with repeating dispenser.

Pump

19 A pump capable of operating at 100 $\text{ml}\cdot\text{min}^{-1}$.

Optical comparator

20 An optical comparator with a x3 magnifying lens and measurement graticule.

PROCEDURE

Estimation of syringe bias

21 Fit the syringe into the repeating dispenser and set to deliver 4.0 μl . The optical comparator can be used to align the plunger. Fill the syringe with mercury and weigh the delivered mercury to the nearest 50 μg . Calculate the actual volume delivered by the syringe, using the density of mercury at the temperature of the experiment.

Note 4: Mercury is not suitable as a calibrant liquid for some plunger-in-needle type syringes. Bromoform may be used instead.

Solution containing approximately 10 mg/ml of each component

22 Accurately weigh approximately 1 g of each of the substances in mixture 1 (paragraph 8) into a 100 ml volumetric flask, starting with the least volatile substance. Make up to 100 ml with methanol (paragraph 11), stopper and shake to mix.

Note 5: Other specific volatile organic components may be included in the calibration blend by adding 1 g of the relevant component before making up to volume.

23 Prepare similar calibration solutions from mixtures 2 or 3 (paragraphs 9 and 10) if required.

Solution containing approximately 1 mg/ml of each component

24 Introduce 50 ml of methanol into a 100 ml volumetric flask. Add 10 ml of solution 22 or 23. Make up to 100 ml with methanol, stopper and shake to mix.

Solution containing approximately 1 mg/ml of gas

25 Obtain pure gas at atmospheric pressure by filling a small plastic bag from a gas cylinder. Fill a 1 ml gas-tight syringe with 1 ml of the pure gas and close the valve of the syringe. Using a 2 ml septum vial, add 2 ml methanol and close with the septum cap. Insert the tip of the syringe needle through the septum cap into the methanol. Open the valve and withdraw the plunger slightly to allow the methanol to enter the syringe. The action of the gas dissolving in the methanol creates a vacuum and the syringe fills with solvent. Return the solution to the vial. Flush the syringe twice with the solution and return the washings to the vial. Calculate the weight of gas added using the gas laws, ie 1 mole of gas at STP occupies 22.4 litres.

Stability of calibration blend solutions

26 Fresh standard solutions should be prepared weekly, or more frequently if evidence is noted of deterioration, eg condensation reactions between alcohols and ketones.

Standard loaded sorbent tubes

For solvent desorption

27 Using the pre-set syringe (paragraph 18), slowly inject 4 ml of organic compound, in either the pure state or diluted in a suitable solvent (paragraph 11), onto the glass wool preceding the main section in the sorbent tube. Simultaneously, pull air through the tube at a flow rate of about 100 $\text{ml}\cdot\text{min}^{-1}$ in the same direction as for sampling, using the sampling pump (paragraph 19). After 5 minutes, disconnect the pump, cap the tube and store the tube overnight at 4°C. Calculate the weight of organic compound loaded, using the density of the bulk liquid (pure liquid or dilution). Dilutions should be prepared gravimetrically.

Note 6: Volumes other than 4 μl may be used, provided the syringe is calibrated as in paragraph 21. However, volumes of less than 1 μl are not recommended, as precision may be compromised.

28 A variant of the method can be used for loading charcoal diffusive badges.³ Inject the compound or a dilution onto a glass fibre filter paper placed inside the badge and allow the vapour to diffuse from the filter to the sorbent within the badge.

For thermal desorption

29 A sorbent tube is fitted into the injection unit (paragraph 17) through which inert purge gas is passed at 100 ml/min and a 4 μl aliquot of an appropriate standard

Table 3 Precision of method (thermal desorption tubes) - analysis by Perkin-Elmer ATD-400

<i>Loading method</i>	<i>Compounds</i>	<i>Loading (μg)</i>	<i>CV on BP 1 column (%)</i>	<i>CV on BP 10 column (%)</i>
injection of dilution	benzene	9.1	0.9	1
injection of dilution	tetrachloroethylene	17.7	0.9	1.3
injection of dilution	styrene	9.98	1	1

Table 4 Storage stability

<i>Loading method</i>	<i>Sorbent</i>	<i>Compounds</i>	<i>Loading</i>	<i>Storage time</i>	<i>Mean recovery (%)</i>	<i>Source ref</i>
dynamic	charcoal	5 chlorinated solvents	1 to 10 mg	2 weeks at 4°C	90.8 \pm 2.2	ISO (9)
dynamic	charcoal	5 chlorinated solvents	1 to 10 mg	2 weeks at -20°C	98.4	ISO (9)
dynamic	Tenax TA	benzene, toluene, xylene	1 μg	14 months* at 40°C	100-104	BCR (10)
dynamic	Tenax TA	benzene, toluene, xylene	1 μg	14 months* at 19-23°C	102-104	BCR (10)
dynamic	Tenax TA	benzene, toluene, xylene	1 μg	14 months* at 0-4°C	101-103	BCR (10)
dynamic	Tenax GR	3 chlorinated solvents	0.1 to 1 μg	27 days at 40°C	98.2	BCR (11)
dynamic	Tenax GR	toluene	4 to 36 ng	27 days at 40°C	103.3	BCR (11)
injection of dilution as vapour	Tenax TA	as para 6	10 μg	5 months	99.7**	MDHS 72 (12)
injection of dilution as vapour	Tenax TA	as para 6	10 μg	11 months	99.4***	MDHS 72 (12)
injection of dilution as vapour	Chromosorb 106	16 solvents	1 μg	2 weeks	105.6	EC project (13)
injection of dilution as vapour	Carboxen 569	propane, methanol	1 μg	2 weeks	115, 64	EC project (13)

* no instability was detected after 25 months

** excluding hexane (93.6%), methoxyethanol (87.3%)

*** excluding methoxyethanol (93.1%)

solution injected through the septum. After 5 minutes, the tube is then disconnected and sealed.

Note 7: *Volumes other than 4 μl may be used, provided the syringe is calibrated as in paragraph 21. However, volumes of less than 1 μl are not recommended, as precision may be compromised.*

30 For ambient or indoor air, use standard solution of approximately 100 $\mu\text{g}/\text{ml}^{-1}$ and 10 $\mu\text{g}/\text{ml}^{-1}$.

Effect of humidity

31 The methods described in paragraphs 27 and 28 do not take account of humidity. Tube standards loaded for desorption efficiency or storage studies may be affected by humidity. If so, tubes may be humidified either by adding an appropriate amount of water to the injected solution or by passing an appropriate volume of humidified clean air through the tube prior to the injection.

Precision

32 Precision data for solvent desorption tubes are given in Table 1 and data for thermal desorption tubes in Table 2. Historic data for solvent desorption indicate precisions of around 5% were usual, although better than 1% is achievable under carefully controlled conditions for substances with desorption efficiencies close to 100%. Precision data for thermal desorption are generally 2% or better.

Storage

33 Storage data for both types of tube are given in Table 4. Charcoal tube standards are relatively unstable and are best refrigerated. Thermal desorption standards are generally stable and if properly sealed do not require refrigeration. However, migration between sorbent beds within multi-bed tubes may occur, and such tubes are best refrigerated.

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 (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.

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