



Occupational Medicine and  
Hygiene Laboratory

## MDHS 3

Methods for the  
Determination of  
Hazardous Substances

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# Generation of test atmospheres of organic vapours by the syringe injection technique

Portable apparatus for laboratory and field use

## INTRODUCTION

1 Organic vapours are, in general, toxic and may also be flammable. Handle pure chemicals and concentrated solutions in a ventilated fume cupboard. The apparatus or vapour exit should be placed in a ventilated fume cupboard or suitably vented to the outside atmosphere. Consult suitable source reference for toxicity and first-aid advice.

## SCOPE

### Range

2 This method is suitable for the generation of atmospheres of gases or low- to medium-boiling liquids from the volume percent level down to 0.1 ppm in one to three dilution steps. The atmosphere may be generated at a rate between 1 and 100 litres/min. The apparatus is suitable for liquids with boiling points of up to about 140°C, but the range may be extended by solvent dilution of the injected liquid.

### Accuracy

3 Assuming there are no leaks and the (dynamic) system has reached equilibrium, there are three main sources of error:

### Atomisation

4 The liquid may not be evaporating in a smooth and predictable manner, particularly if it is relatively high-boiling.

### Syringe

5 The motor-driven injector is expected to have precision of 1%, measured as a relative standard deviation and, if calibrated, should have a bias of less than 1%.

### Rotameters

6 The rotameters have a specified precision of 2%, measured as a relative standard deviation. A bias may occur if temperature and pressure corrections are not made (para 35).

### Stability

7 There will be some short-term variation in the

concentration of the delivered atmosphere, owing to slight irregularities in the syringe drive, atomisation and pressure control. This variation occurs over time periods of the order of a few seconds and should not exceed  $\pm 5\%$  of the mean delivered concentration. Some smoothing of the atmosphere may be achieved by incorporating a large (about 10 litres) glass vessel at the output stage of the design.

## Construction

8 The construction is of glass with ball and socket joints to avoid strain and facilitate interchangeability of parts. The apparatus is mounted on a board of height 560 mm and width 660 mm, which is sufficiently compact to be placed in a small fume cupboard. The apparatus can be made fully portable, requiring only a source of compressed air and, for toxic vapours, fume extraction.

## PRINCIPLE

9 Organic gases or liquids are loaded into a gas-tight syringe connected by PTFE tubing to an atomisation chamber.

10 The organic material is injected into the atomisation chamber by means of a motor-drive on the syringe.

11 A regulated supply of air (or nitrogen, or calibrated gas mixture) is simultaneously applied to the atomisation chamber.

12 If desired, the atmosphere generated in the atomisation chamber may be diluted with a second or third regulated supply of air.

13 The concentration of atmosphere generated is calculated from the rate of injection of the gas or vapour and the rate of supply of the air or diluent gas.

## REAGENTS

### Compressed air

14 A compressor or compressed air cylinder may be used. The apparatus includes a purification unit.

### Bulk gas or liquids

15 These should be analytical grade of known purity.

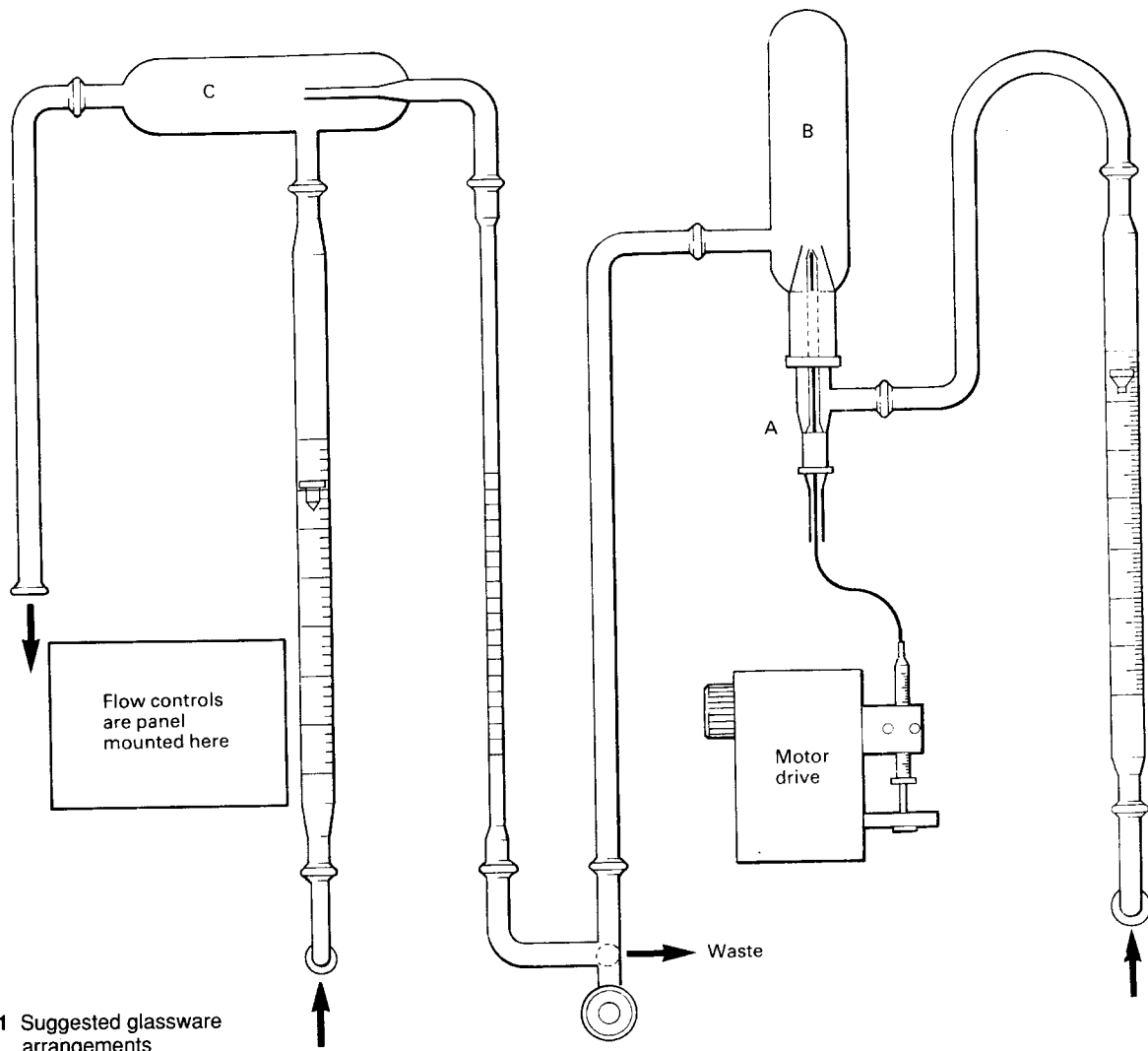


Fig 1 Suggested glassware arrangements

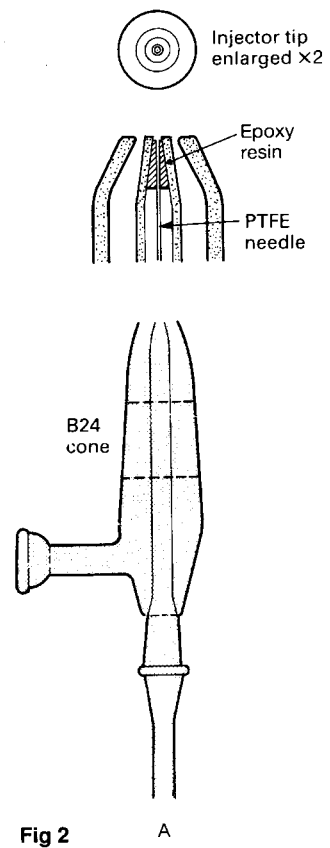


Fig 2

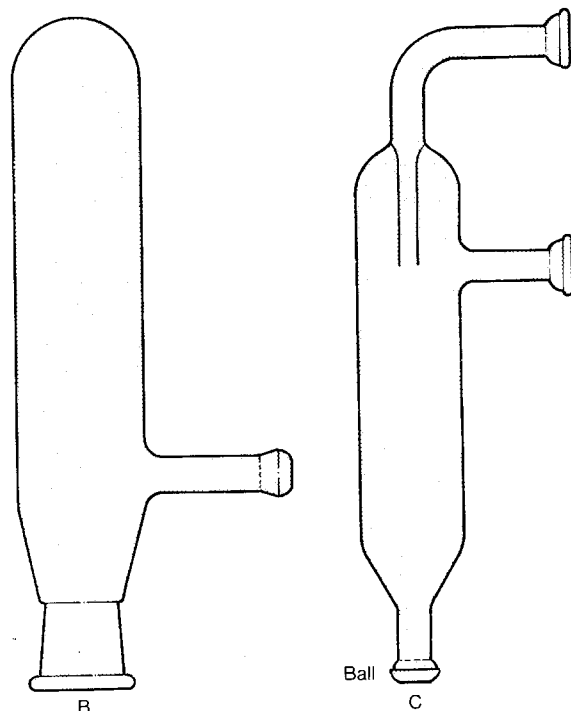


Fig 3

## APPARATUS

16 The apparatus is illustrated in Fig 1. The main items required are:

### Glassware (Figs 2 and 3)

17 Borosilicate glass is used for the mixing chambers and connecting tubing. Joints are all ball and socket. The connecting tubing should be at least 9 mm ID to minimise resistance to air flows.

### Flow meters

18 A range of rotameters from 30-350 ml/min to 5-50 litres/min.

### Motor-driven syringe unit

19 Suitable for injecting liquids in the range 3-30  $\mu\text{l}/\text{min}$  from a 1-ml glass syringe.

### Regulators

20 Pressure regulators, needle valves, on-off valves, 3-way valves, and an air filtration and purification unit.

## PROCEDURE

21 The apparatus is set up as in Fig 1 (for a two-stage dilution system).

22 Calculate the gas flows required for generating the specified atmosphere, selecting syringe size, syringe drive speed, and rotameters for gas flows to atomiser and to second or third dilution stage as appropriate.

23 A 1-ml gas-tight precision glass syringe is generally used for liquids; a 10-ml gas-tight precision glass syringe is generally used for pure gas.

24 The syringe drive speed should be a minimum of 0.2 mm/min to avoid irregularities in the test atmosphere due to thermal expansion or contraction of the syringe reservoir.

25 Individual dilutions should not exceed a ratio of 1:100 v/v. It may be necessary to include a further dilution stage to achieve generated concentrations in the sub ppm range.

26 Switch on and adjust syringe drive, pressure and flow regulators.

27 Checks should be made to ensure even evaporation of liquid (if used) in the atomiser.

28 Allow system to stabilise; dynamic equilibrium should be reached within 10 min.

## CALIBRATION

### Syringe drive

29 Fill the 1-ml glass syringe with distilled water. Operate the motor-drive syringe at the drive setting to be used and collect the dispensed water into a previously

weighed 10-ml flask. Run the motor for 60 minutes. Reweigh the flask and calculate the volume of water collected.

$$q_l = \frac{v}{t}$$

where  $q_l$  = injection rate ( $\mu\text{l}/\text{min}$ )  
 $v$  = volume of water collected ( $\mu\text{l}$ )  
 $t$  = time (min)

### Rotameters

30 The true flow rate of a gas passing through a rotameter calibrated at  $T_1$  K and  $P_1$  mm Hg for air may be obtained by multiplying the flow indicated on the rotameter by the factor  $f = \left[ \frac{P_2 \times T_1}{d_g \times P_1 \times T_2} \right]^{1/2}$

where  $T_2$  = operating temperatures (K)  
 $d_g$  = density of gas relative to air  
 $P_2$  = operating pressure (mm Hg)

### Atmosphere

31 The concentration of vapour delivered by the apparatus may be checked by independent analysis, eg infra-red gas analyser, gas chromatography (after collection and desorption from adsorbent of known desorption efficiency), wet chemical methods.

## CALCULATIONS

32 To calculate diluent gas flows for standard atmospheres of liquid vapours, the following expression may be used:

$$q_g = \frac{d_l \times q_l \times 1000}{C \times f}$$

where  $q_g$  = indicated gas flow (litres/min)  
 $d_l$  = liquid density (g/ml)  
 $q_l$  = liquid injection rate ( $\mu\text{l}/\text{min}$ )  
 $C$  = required vapour concentration ( $\text{mg}/\text{m}^3$ ) at operating temperature and pressure  
 $f$  = rotameter correction factor (paras 30 and 35)

33 To calculate diluent flows for gases, the following expression may be used:

$$q_2 = \frac{q_1}{C'}$$

where  $q_2$  = diluent gas flow (litres/min)  
 $q_1$  = primary gas flow of pure vapour ( $\mu$ l/min)  
 $C'$  = required vapour concentration (vol. ppm)

34 Concentrations in vol. ppm and mg/m<sup>3</sup> are related by:

$$C' = C \times \frac{24.45}{MW} \times \frac{760}{P_2} \times \frac{T_2}{298}$$

where  $C'$  = vapour concentration (ppm)  
 $C$  = vapour concentration (mg/m<sup>3</sup>)  
 24.45 = molar volume (litres) at 25°C and 760 mm Hg  
 MW = molecular weight of substance used

35 For temperatures and pressures within 10°C or 30 mm Hg of calibration conditions, rotameter corrections are less than 2% and may be ignored. Care should be taken, particularly in the design of the atomiser, to avoid significant back pressure which might introduce further rotameter pressure corrections. For flows greater than 15 litres/min a larger orifice than that shown in Fig 2 (A) may be required.

#### ADVICE

Advice on this method and the equipment used may be obtained from the Health and Safety Executive, Occupational Medicine and Hygiene Laboratory, 403-405 Edgware Road, London NW2 6LN (telephone 081-450 8911).

A number of methods are available for generating standard atmospheres, including others described in this series.<sup>1</sup> The use of other methods not included in this series is acceptable provided they have the accuracy and reliability appropriate to the application.

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. Methods for the Determination of Hazardous Substances. *Generation of Test Atmospheres by the Permeation Tube Method*. MDHS 4/81. HSE: London, 1981.

#### OTHER USEFUL REFERENCES

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Further copies of this publication can be obtained from the Health and Safety Executive Sales Point, St Hugh's House, Stanley Precinct, Bootle, Merseyside L20 3QY.

