

General methods for sampling and gravimetric analysis of respirable, thoracic and inhalable aerosols

MDHS14/4

Methods for the
Determination of
Hazardous Substances

Health and Safety
Laboratory

Introduction

- 1 This procedure aims to guide those who wish to collect the respirable, thoracic and inhalable aerosol fractions in air for the purpose of monitoring workplace exposure. It also describes analysis of the fractions using the gravimetric technique.
- 2 Materials hazardous to health often occur in the workplace in the form of aerosols. The term 'aerosol' is used to describe any suspension of particles in air, whether they constitute dust, fibres, fume, smoke or liquid droplets. Most aerosols consist of a wide range of particle diameters.
- 3 The behaviour, deposition and fate of any particle after entry into the human respiratory system are determined by the chemical nature and size of the particle. For occupational hygiene purposes it is important to consider the concentration and the size fractions present.
- 4 It is possible to define aerosol size fractions that relate to the region of the respiratory tract where they deposit. The convention for these size fractions are described in ISO 7708¹ or BS EN 481.² These are the inhalable, thoracic and respirable size fractions:
 - (a) **Inhalable fraction** – this approximates to the fraction of airborne material that enters the nose and mouth during breathing, and is therefore available for deposition anywhere in the respiratory tract.
 - (b) **Thoracic fraction** – this is the fraction of inhaled airborne material penetrating beyond the larynx.
 - (c) **Respirable fraction** – this is the inhaled airborne material that penetrates to the lower gas exchange region of the lung.
- 5 Advice on the relevant size fraction to be measured for a particular material hazardous to health may be obtained from *EH40/2005 Workplace exposure limits*³ and the Approved Code of Practice on the COSHH Regulations⁴.

Scope

- 6 The methods described in this MDHS are suitable for the measurement of exposure to the health-related concentrations of most aerosols in the workplace. In some instances alternative methods exist (eg welding fume,⁵ colophony⁶ and isocyanates⁷) and you should refer to these specific methods. For some materials a specific sampler is required (eg IOM sampler is the preferred sampler for cotton dust⁴) to reliably perform the analysis. The use of alternative methods is acceptable provided that the accuracy and reliability appropriate to the application can be demonstrated.

7 This procedure describes the analysis of the collected aerosol using the gravimetric technique. After drawing a measured volume of air through the pre-weighed collection medium (eg filter or foam) mounted in a suitable particle size-selective sampler, the mass concentration can then be determined from the mass of the aerosol collected and the sampled air volume.

8 Where further analysis for specific constituents is required, refer to the appropriate methods to ensure the sampling medium is compatible with the analysis technique.

Recommended sampling

9 Air monitoring should be representative of the working periods of the individuals exposed. General guidance on workplace monitoring is given in *Monitoring strategies for toxic substances* (HSG173).⁸

10 A longer sampling time ensures a heavier deposit and reduces potential weighing inaccuracies. So sampling times should be as long as is reasonably practicable:

- (a) The maximum sampling time should be the entire shift, and 15 minutes for short-term samples. Task-specific sampling should cover the period of the task being performed.
- (b) For an 8-hour time-weighted average (TWA) estimation of exposure, the minimum sampling period should be at least 25% of the shift,⁸ though it is preferable if sampling times are no less than four hours.

11 To avoid sampler overloading where dust concentrations are high, several consecutive samplers should be employed for comparison with workplace exposure limits (WELs), though enough information may be obtained from one sample.

12 When employing fixed-point sampling, the samplers should be positioned at approximately head height, away from obstructions, fresh-air inlets or strong winds. The sampling procedures are otherwise the same as for personal sampling.

Prerequisites

13 Users of this procedure will need to be familiar with the content of Occupational exposure limits (EH40),³ *Monitoring strategies for toxic substances* (HSG173)⁸ and BS ISO 15767.⁹

Safety

14 Users of this procedure should carry out a suitable risk assessment. It is the user's responsibility to establish appropriate health and safety practices and to ensure compliance with regulatory requirements.

Equipment

Aerosol sampling equipment

15 Use a suitable sampler (see the Appendix) capable of performing according to the required size fraction of interest (inhalable, thoracic or respirable).^{1,2} Samplers

should be pre-cleaned, checked for defects and operated according to the manufacturer's instructions.

16 Some samplers are designed to sample multiple size fractions within one sampler and are termed multi-fraction samplers. Commercially available samplers and references to published reports on their performance are given in PD CEN/TR 15230.¹⁰

17 In instances where workers wear face visors, lapel- or collar-mounted samplers are effectively outside the breathing zone of the worker. The face level sampler¹¹ is designed to measure manganese in welding aerosol according to ISO 10882.⁵ It allows exposure measurements to be made close to the worker's mouth and can also be worn comfortably inside face visors. It can also be used for the analysis of other metals in welding aerosol and gravimetric analysis of welding aerosol, but with reduced sampling efficiency for particles larger than 20 µm.

Collection media

18 The choice of collection medium (eg filter, foam or impaction plate) will be dictated by the type of sampler, the sampler flow rate and by analytical considerations.

19 In some sampler designs the collection media are held in cassettes and these can be weighed together (see the Appendix for additional information on plastic cassettes). In other samplers the collection medium may be held within a holder that is not intended to be weighed. The manufacturer's operating instructions should be consulted on which parts of the sampler should be included in the gravimetric procedure.

Filters

20 For gravimetric measurements, glass fibre filters are commonly used, but if further chemical analysis of the collected material is required then this may determine the type of collection medium selected.

21 Fibre loss from glass fibre filters may occur during handling and could be significant if not weighed within a cassette. Consider using membrane filters to alleviate this issue.

22 Some filter materials (eg cellulose nitrate) can show excessive weight change due to moisture absorption, and other types (eg PVC, PTFE) can show excessive static build-up. Filters of mixed esters of cellulose have reduced susceptibility to these factors.

23 For membrane filters, the choice of filter pore size will depend on the size fraction of the aerosol being collected and the sampler flow rate. It is recommended that the largest pore size possible be used to minimise the pressure drop across the filter. Consult the manufacturer or analyst for the optimum filter for the particular application.

Other equipment

24 A balance should be calibrated against a primary standard for weighing the sampling media. The balance should be capable of weighing to a precision of at least 10 µg, and preferably 1 µg. The balance should be checked against a calibrated standard weight traceable to International Standards at the intervals recommended by the manufacturer and immediately before weighing sampling media. Ideally, the balance should be placed on an anti-vibration worktop.

- 25 The balance and sampling media to be weighed should be placed in a room with temperature and humidity controls within the range specified by the balance manufacturer. BS ISO 15767⁹ states that the humidity should be constant within $\pm 5\%$ and the temperature $\pm 2^\circ\text{C}$.
- 26 Use a static eliminator when weighing filters.
- 27 Use flat-tipped forceps for handling filters.
- 28 Use filter tins or sampler cassettes, transport clips and a transport container to safely transport samples from the site to the analytical laboratory.
- 29 Personal sampling pumps that meet the requirements of BS EN ISO 13137¹² should be operated according to the manufacturer's instructions. Sampling pumps should have the following features as a minimum:
- (a) an automatic flow control which keeps the volumetric flow rate within $\pm 0.1 \text{ l}\cdot\text{min}^{-1}$ in case of changing back pressure caused by filter loading;
 - (b) either a malfunction indicator, which following the completion of sampling, indicates that the air flow has been reduced or interrupted during sampling, or an automatic cut-out, which stops the pump flow if it is reduced or interrupted;
 - (c) a facility for adjustment of the flow rate that prevents inadvertent adjustment during use;
 - (d) pulsation damped flow for cyclone samplers.
- 30 Flexible plastic tubing should have a suitable diameter for making a leak proof connection from the sampling head to the pump. For personal sampling, belts or harnesses should allow sampling apparatus to be attached.
- 31 There should be a portable flow meter, calibrated against a primary standard at the flow rates of interest, with a measurement uncertainty less than $\pm 2.5\%$.¹⁰
- 32 A suitable adapter needs to connect the sampling head and the flow meter in order to set the flow rate through the sampler accurately.
- 33 A timer should determine the pump on/off times.

Preparation and sampling

Substrate weighing

Procedure for pre- and post-sampling media (in cassettes where used)

- 34 It is recommended that the weighing procedure be documented to ensure consistency of approach.
- 35 Prior to sampling, sufficient sample collection media should be placed in suitable containers, with lids ajar and left to equilibrate overnight in the balance room. Some substrates or cassettes may take longer to equilibrate. For the majority of air samples and media, overnight conditioning is satisfactory but some substrates or cassettes may take much longer to equilibrate. Further guidance is given in BS ISO 15767.⁹
- 36 As a minimum, one field (site) blank is required for every ten samples collected, with a minimum of three blanks for each batch of samples. Field blanks are used to correct for any weight changes caused by atmospheric conditions and

the handling of the media during sampling. For this reason, it is essential that field blanks are exposed to the same conditions as the samples apart from the period of sampling. In addition to field blanks, the analyst has the option of preparing laboratory blanks which may be used to assess the weighing precision.

37 Similarly, the post-run samples and blanks should be equilibrated in the balance room on receipt. The transport containers (cassettes, clips etc) should be visually checked for signs of disturbance of material from the filters. It may be necessary to develop and document special transport procedures for the aerosol samples to minimise transport losses.¹³ There may be some material on the sampling cassettes which will form part of the sample, so these should be handled carefully to avoid any disturbance of the deposit. Similarly, there may be deposits on the outside of the sampling cassettes which do not form part of the sample and should be removed before weighing.

38 Before weighing any sampling media, confirm satisfactory balance operation using the calibrated standard weight.

39 Pass the media over a static eliminator before weighing to dissipate any electrostatic charge.

40 It is important to ensure the balance has stabilised before recording each weight and that that the balance returns to zero after each weighing.

41 Blanks should be interspersed with samples, before and after sampling, so as to detect systematic variations in weighing or substrate mass (eg due to sorption or evaporation of a contaminant during weighing).

42 The limit of detection (LOD) of the weighing procedure can be determined from three times the standard deviation of the weight changes of all the field blanks.

43 Weight changes of samples that are less than the limit of detection should be reported as less than the LOD.

44 The inhalable convention is undefined for aerosols with aerodynamic diameters above 100 µm. Where these large particles are observed on the sampling media, this should be noted in the analyst's report. Similarly, observations such as unusual particle deposition patterns should be noted in the analyst's report.

Sample collection

45 The type of sampler to be used depends on many factors including the size fraction of interest and the suspected aerosol concentrations. In most cases an accurately measurable mass may be collected for long-term samples (greater than 4 hours) using a sampler that operates in the region of 2 l.min⁻¹. A higher flow rate sampler will provide a lower LOD for any given sampling period and may be preferred for shorter sampling periods or if the aerosol concentration is low.

46 Set up the sampling equipment in an uncontaminated area (ideally within one hour of the start of sampling).

47 Connect with the sampling device and collection medium to a sampling pump (excluding blanks).

48 Remove any protective cover or cap from the sampler, switch on the sampling pump and allow the pump flow to stabilise according to the manufacturer's

instructions. Attach the calibrated flow meter to the inlet of the sampler (using an adaptor if required) so that it measures the flow through the inlet. Set the flow rate of the sampler to within $\pm 0.1 \text{ l}\cdot\text{min}^{-1}$ of the prescribed flow rate.

49 Perform a leak test by covering the sampler's inlet or 'kinking' its tube. If the pump does not stall this could indicate a leak and should be rectified. Once the checks are completed, switch off the pump and recap the sampler.

50 If the temperature, humidity and pressure in the environment where the samplers are to be used differ significantly from where the flow rate was set, the volumetric flow rate may change and need to be readjusted just before sampling.

51 The sampler should be attached to the worker's upper chest or lapel, not more than 30 cm away from the nose-mouth region. Fixed-point sampling may be used to determine background levels of aerosol in the workplace although it is not appropriate to compare background samples with workplace exposure limits.

52 Samplers are not generally sensitive to orientation, but cyclones should, if possible, be attached with the grit-pot at the base as shown in Figure 2 on page 11.

53 Attach the pump to a belt or harness so that it causes minimum inconvenience to the worker, and safely secure any tubing used to connect the sampler to the pump.

54 Ensure, as far as possible, that the position the sampler is mounted in reflects the exposure of the worker. This should include consideration as to the nature of the processes being undertaken and whether these may cause non-uniform aerosol concentrations within the breathing zone.

55 When ready to begin sampling, remove the protective cap. If the pump is fitted with an integral timer, ensure that this is reset to zero. Switch on the pump and record the time.

56 Check the sampler and pump periodically during sampling to ensure that the equipment is still working, and, if appropriate, re-measure the flow rate and record the new values.

57 For each sample, record the sample identity, time on and off (and when flow rates were checked), the volumetric flow rate and other relevant sampling information.

58 At the end of the sampling period measure the flow rate, switch off the pump, record the reading of the pump timer, or record the time and attach the protective cap. Carefully remove the sampling equipment without subjecting it to mechanical shocks. Cyclones must be retained upright when switched off to avoid the contents of the grit pot falling onto the filter.

59 Ideally, in the clean area, at the end of the sampling period the sampling media should be removed from the samplers for transportation.

- (a) For samplers that use an internal cassette (eg the IOM sampler), remove the cassette from each sampler and fasten with the transport clip supplied by the manufacturer.
- (b) For other types of sampling media (eg filters) remove the filter using flat-tipped forceps and place in a labelled filter transport container. Take particular care to prevent dust being dislodged from heavily loaded filters.

60 Alternatively, it may be practical to cap the samplers and return to the laboratory for disassembly.

61 Transport the samples and blanks to the laboratory in a labelled container suitable to prevent damage or disturbance in transit.

62 Calculate the duration of the sampling period and check this against the recorded time on the reading from the pump. Consider the sample to be invalid if the two sampling times differ by more than 5% since this may indicate that the pump did not operate for the entire period.

63 For cyclone samplers the sample is invalid if the final flow rate differs by more than $0.1 \text{ l}\cdot\text{min}^{-1}$ or 5% (whichever is larger) from the initial flow rate. Where the sample is valid, assume that the mean volumetric flow rate is exactly equal to the recommended flow rate.

64 There are various factors that may affect the validity of the collected aerosol sample, such as:

- (a) presence of projectile particles (eg metal fragments from grinding processes) or splashes (eg mineral oil) entering the sampler;
- (b) large particles entering the sampler that are outside the inhalable definition (ie particles with aerodynamic diameters greater than $100 \mu\text{m}$);
- (c) transportation losses (eg particles falling off the filter).

65 In some instances where the aerosol concentrations are unusually high, variable or there are significant projectile particles present, it is reasonable to assume that the sampler may be unrepresentative of the personal exposure. This should be noted during the sampling and either disregard the result, or treat it as a 'worst-case' estimate of personal exposure. If projectile particles are present then an unpumped sampler positioned next to the pumped sampler maybe used to correct for unaspirated particles.

66 For samples that may be used for further analysis (eg metals), consider the effect of sample losses (eg wall losses onto the internal walls of the IOM cassette) and mitigate these where possible.

Calculation of airborne aerosol concentration

Volume of air sample

67 Calculate the sampled air volume, V_s , in m^3 , of each air sample by multiplying the mean volumetric flow rate in cubic metres per minute (litres per minute divided by 1000) by the sampling time in minutes.

68 Where the flow rate was checked during the sampling period the above calculation may be performed for each time period and V_s determined by summing the sampled volume during each time period.

Aerosol concentration

69 The measured aerosol concentration, C , in $\text{mg}\cdot\text{m}^{-3}$, can be calculated according to the following equation:

$$C = \frac{(M_2 - M_1 - B)}{V_s}$$

Where:

M_1 = mass of filter (plus cassette where used) before sampling (mg)

M_2 = mass of filter (plus cassette where used) after sampling (mg)

B = average mass change of blanks (mg)

V_s = volume of air sampled (m^3)

Method performance

70 The aerosol sampling methods described in this procedure are suitable for the determination of most types of aerosols in workplace air and have been evaluated in laboratory and field trials^{10,14-23} to demonstrate compliance with the required sampling conventions.²

Limit of detection and limit of quantification

71 Both the limit of detection (LOD) and the limit of quantification (LOQ) of the methods covered in this document depend on the volume of air sampled, the sensitivity of the balance and the weight stability of the sampling media used within the sampling instruments described. Detailed information on the determination of the LOD and LOQ for gravimetric analysis of airborne dust samples can be found in ISO 15767.⁹ In practice, it is convenient for the LOD of the gravimetric analysis to be determined as three times the standard deviation of the weight changes of the blank samples.

Sampling errors

Concentration bias and precision

72 Aerosols are often non-uniform in the workplace and the concentration to which the sampler is exposed is not necessarily the same as the concentration to which the person is exposed. This is particularly the case for larger-sized inhalable particles. It is usually the main source of measurement error, compared to instrument and analytical bias and precision. Sampling errors caused by concentration non-uniformity can be minimised by siting the sampler within the breathing zone of the worker, close to the nose and mouth.

Instrument bias and precision

73 No sampling instrument will exactly match the target specifications defined in ISO 7708¹ under all workplace conditions. The samplers listed in this procedure meet the required specifications over a reasonable range of conditions as laid down in EN 13205.¹³

Analytical bias and precision

74 Compared to sampling errors caused by variability in concentration of the workplace aerosol the analytical bias and precision are generally low. Details are given in ISO 15767⁹ and should be consulted to minimise substrate weighing errors.

Expanded uncertainty

75 Samplers meeting the requirements of EN 13205¹³ will have accuracy better than or equal to 30%.

76 For the complete measurement procedure, the expanded uncertainty combines the uncertainty of:

- (a) the sampled volume;
- (b) the sampled fraction;
- (c) the transportation, storage, sample preparation etc;
- (d) the analytical method employed.²⁴

Appendix: Types of aerosol samplers

Inhalable samplers

1 Suitable samplers and typical operational flow rates are: the Institute of Occupational Medicine sampler (IOM, 2 l.min⁻¹, Figure 1), conical inhalable sampler (CIS, 3.5 l.min⁻¹), the button sampler (4 l.min⁻¹) and the multi-orifice sampler (2 l.min⁻¹).

2 The IOM sampler has been shown to give the best agreement with the inhalable convention¹ under the widest range of workplace conditions, and is the preferred method of sampling the inhalable aerosol. This sampler typically has a sampling bias of less than $\pm 5\%$. Other samplers may exhibit a larger bias under certain workplace conditions.¹⁴⁻¹⁶

3 It should be noted that the inhalable dust may not be determined by weighing the respirable cyclone and grit pot unless this has been demonstrated to be valid.

Thoracic samplers

4 The GK2.69 cyclone sampler (1.6 l.min⁻¹) and the PPI2 (2 l.min⁻¹) impactor are two types of thoracic sampler. The GK2.69 has shown good agreement with the thoracic convention.²⁵

Respirable samplers

5 The cyclone sampler of the generic Higgins-Dewell type (2.2 l.min⁻¹, Figure 2)²⁶ is recommended for use in the UK for optimal agreement with the respirable convention.^{1,2} Other suitable cyclone types that also agree with the respirable convention include the GS-3 cyclone (2.75 l.min⁻¹) and the GK2.69 cyclone (4.2 l.min⁻¹).

6 Higher flow rate samplers are the PGP10 (10.0 l.min⁻¹),²⁷⁻²⁹ the BGI GK4.162 (8.5 to 9.5 l.min⁻¹) cyclone samplers³⁰ and the PPI8 impactor (8.0 l.min⁻¹).

Multi-fraction samplers

7 These samplers allow the concentration of several health-related size fractions to be measured simultaneously.

8 Examples of samplers that directly measure the size fractions are:

- (a) IOM 'multidust' dual-fraction respirable sampler (inhalable and respirable fraction) which operates at 2 l.min⁻¹;
- (b) CIS multi-fraction respirable sampler (inhalable, thoracic and respirable fractions) which operates at 3.5 l.min⁻¹;
- (c) Respicon sampler (inhalable, thoracic and respirable fractions).

9 The IOM and CIS samplers aspirate the inhalable fraction and then use porous polyurethane foam inserts to select the respirable or thoracic fraction.

10 Some samplers determine the aerosol size distribution which is then processed by the application of the appropriate sampling convention (eg respirable) to determine the mass of inhaled aerosol. Examples of these samplers are the Marple and Mini-Moudie personal multistage cascade impactor samplers.

Additional information on samplers

11 The PPI8 impactor sampler removes larger particles by impacting and collecting them onto oiled disks. The respirable-sized particles are collected onto a backup filter. Care should be taken not to overload the disks as they will become less effective at capturing large particles and could result in over sampling as they are collected on the backup filter.

12 When using CIS multi-orifice sampler types, it is essential to handle the loaded samplers with great care before the filters are removed. Ideally, filters should be removed before transport to the laboratory in order to prevent movement of loose material within the sampling heads. If this is not carried out, material can fall from the insides of the sampler onto the filter, causing a positive sampling bias.

13 Some samplers utilise a metal or plastic cassette that is weighed together with the filter. Plastic cassettes may show large weight variations due to moisture absorption. They should therefore be conditioned and weighed in an environment where the temperature and humidity is carefully controlled. They may need to be left to condition for up to several weeks before stable weights are obtained. For the IOM sampler it is recommended that metal filter cassettes are used as these are much more weight stable.

14 The samplers should be regularly maintained and any O-rings checked on a regular basis to ensure they have not perished. Some samplers (eg IOM) have several versions, which may require O-rings in different locations. The location of any O-rings should be checked with the manufacturer's instructions to ensure they are correctly placed.

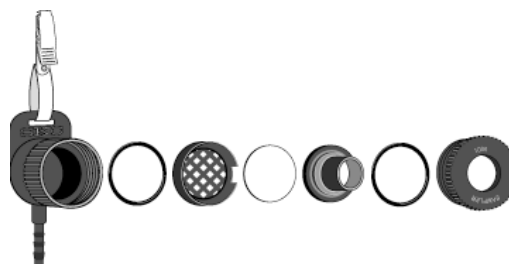


Figure 1 IOM inhalable dust sampler (parts from left to right: sampler body, body o-ring, cassette bottom, filter, cassette top, front plate o-ring, front plate)

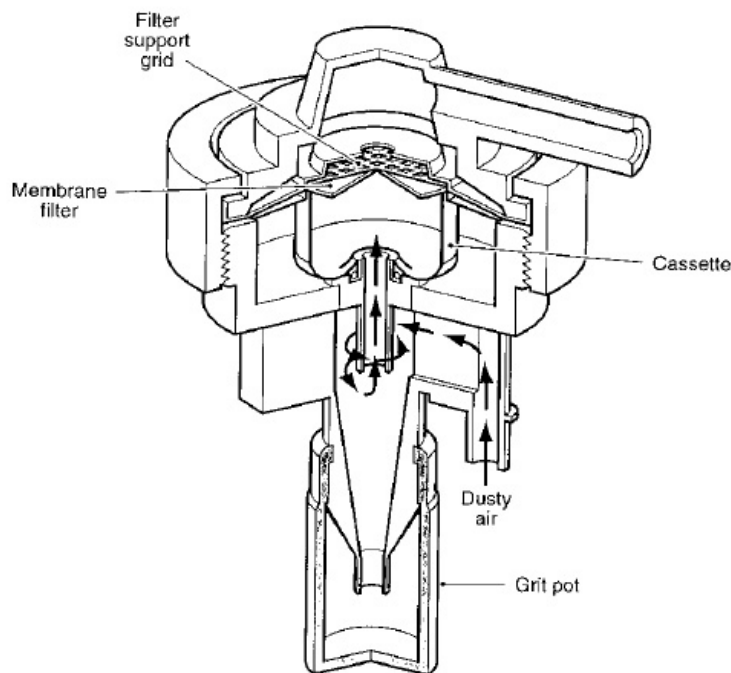


Figure 2 Cyclone respirable dust sampler

References

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- 2 BS EN 481:1993 *Workplace atmospheres: Size fraction definitions for measurement of airborne particles* British Standards Institution
- 3 EH40/2005 *Workplace exposure limits: Containing the list of workplace exposure limits for use with the Control of Substances Hazardous to Health Regulations 2002 (as amended)* Environmental Hygiene Guidance Note EH40 (Second edition) HSE Books 2011 ISBN 978 0 7176 6446 7 www.hse.gov.uk/pubns/books/eh40.htm
- 4 *Control of substances hazardous to health (COSHH). The Control of Substances Hazardous to Health Regulations 2002 (as amended). Approved Code of Practice and guidance L5* (Sixth edition) HSE Books 2013 ISBN 978 0 7176 6582 2 www.hse.gov.uk/pubns/books/l5.htm
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You should use the most current edition of any standards listed.

Further information

For information about health and safety, or to report inconsistencies or inaccuracies in this guidance, visit www.hse.gov.uk/. You can view HSE guidance online and order priced publications from the website. HSE priced publications are also available from bookshops.

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