

MDHS

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



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Measurement of oil mist from mineral oil-based metalworking fluids

June 1997

INTRODUCTION

1 This method describes the measurement of personal exposure to oil mist. Oil mist is the aerosol which can be formed by a mineral oil as it is used as a coolant or lubricant during the machining of metal components. This method is suitable for measuring the airborne aerosol formed from mineral oil-based metalworking fluids, known as neat cutting oils or straight oils. HSE intends to publish a separate method for the measurement of the aerosol from water-mix metalworking fluids (soluble oils, synthetic and semi-synthetic oils). In this document the phrase 'metalworking fluid' will apply to the mineral oil-based variety. This method is suitable only for oils with viscosities greater than 18 cSt (or mm²/s) at 40°C. Measurement of the oil mist concentration from less viscous (more volatile) oils may under-estimate the true value due to loss of volatile components from the sample by evaporation during sampling. This method may be suitable to measure oil mist from other sources, such as from quench oils during the heat treatment of metal components, providing that there is no significant loss of sample from the filter by evaporation.

Properties and uses

2 Properties and uses of metalworking fluids are described in Guidance Note EH62.¹

Health effects

3 The health effects of metalworking fluids are summarised in Guidance Note EH62¹ and in leaflet IND(G)169L.² The use of unrefined mineral oil has been associated with the development of skin cancer - further details can be found in Guidance Note EH58.³

Health precautions

4 Leaflet IND(G)169L² summarises the potential risks involved in working with metalworking fluids and what can be done to control them. Prevention and control of exposure, emergency procedures and health surveillance are described more fully in leaflets IND(G)165L,⁴ IND(G)167L⁵ and IND(G)168L,⁶ and on wall chart IND(G)166L.⁷

Exposure limits

5 The Health and Safety Commission (HSC) has approved an occupational exposure standard (OES) of 5 mg m⁻³, 8-hour time-weighted average reference period for oil mist. This was based on the American Conference of Governmental Industrial Hygienists' (ACGIH) threshold limit value (TLV), which was established in 1964. This limit is published in Table 2 of HSE Guidance Note EH40.⁸ The limit applies to the aerosol only and not to any accompanying hydrocarbon vapour. The limit is currently under review.

Analytical methods

6 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. The method should be able to distinguish aerosol and vapour phases.

Requirements of the COSHH Regulations

7 The Control of Substances Hazardous to Health (COSHH) Regulations 1994⁹ are designed to ensure that the exposure of people at work to substances which could cause health damage 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. Persons who may be exposed to substances hazardous to health should 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. The text of the COSHH Regulations 1994 plus three supporting Approved Codes of Practice (ACOPs) can be found in the following

publication: *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.*¹⁰

SCOPE

Applicability

8 This MDHS describes a gravimetric method for measurement of personal exposure to oil mist. It is applicable to the aerosol formed by mineral oils with viscosities greater than 18 cSt at 40°C. Lower viscosity oils contain more volatile components. The aerosol of a low viscosity oil may lose these volatile components when it is trapped on a filter during sampling, and consequently the analysis may under-estimate the correct airborne concentration.

9 The sampling time for which the method is suitable is dependent upon the sensitivity of the analytical technique used for measurement. A sampling time in the range 2 to 8 hours is recommended to achieve an adequate lower limit for the working range of the method (see paragraph 10).

METHOD PERFORMANCE

Working range

10 The working range of the method is dependent upon the detection limit of the analytical technique used for measurement (see paragraphs 47 and 52), and the volume of air sampled. The detection limit should be an order of magnitude less than the OES (ie 0.5 mg/m³). A detection limit of 0.1 mg/m³ should be achievable using the analytical method described here, with samples taken over an eight hour sampling time at a rate of 2 litres/min.

Overall uncertainty

11 Preliminary investigations indicate that the analytical method does not exhibit significant bias. The analytical recovery from filters spiked with a variety of metalworking fluids has been shown to be between 94 and 98%, with a relative standard deviation of 1%. If the analytical measurements are made within the working range of the method, the overall uncertainty of the method should be within the specifications prescribed by CEN¹¹ for measurements for comparison with limit values.

Interferences

12 The method may be subject to interferences. As well as collecting oil from the aerosol, there is the possibility of the filter also collecting oil droplets by impaction from spray formed in the course of operations involving the metalworking fluid. In such circumstances the effects may be reduced by choosing an inhalable sampler which minimises the occurrence of direct

impaction of metalworking fluid droplets by shielding the sample filter to a certain extent. If this interference is perceived to be a potential problem, its magnitude can be estimated by collecting two filter samples adjacent to each other, but with only one connected to a sampling pump. Comparison of the analytical results from both filters should reveal if there has been significant interference.

13 In some workplace atmospheres there may be other aerosols present which may contribute to the analytical result (such as that from water-mix metalworking fluids). Such interferences will affect this analytical method if they are present at significant concentration and are soluble in cyclohexane.

PRINCIPLE

14 Personal exposure to oil mist is determined gravimetrically. A measured volume of air is drawn through a pre-weighed filter mounted in a sampler attached to the lapel of the metalworking machine operator. The exposed filter is reweighed to obtain a total inhalable particulate concentration. If the total inhalable particulate concentration is greater than 2.5 mg/m³ (half the OES), the oil mist concentration is estimated by measuring the filter's weight loss after the oil is extracted into cyclohexane. Potential interference from oil spray may influence sampling (see paragraph 12).

REAGENTS

15 Analytical grade cyclohexane.

SAMPLING EQUIPMENT

Samplers for collection of the total inhalable fraction of the airborne particles

16 Samplers, with protective covers, for collection of the inhalable fraction of the airborne particles, as defined in European Standard EN 481.¹² Inhalable dust samplers suitable for personal sampling are described in MDHS 14/2.¹³

17 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.

Filters

18 Binder-free glass fibre filters or mixed cellulose ester membrane filters (0.8 µm mean pore diameter) are recommended for use in the samplers.

Sampling pumps

19 Sampling pumps should have as a minimum the following features:

- an automatic flow control which keeps the volumetric flow rate constant in the case of changing back-pressure;
- 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 if the pump flow is reduced or interrupted; and
- a facility for adjustment of the flow rate such that it can only be actuated with the aid of a tool (eg a screw-driver) or requires special knowledge for operation (eg software), so as to prevent inadvertent adjustment of the flow rate during use.

In addition, compliance with the requirements of draft European Standard prEN 1232¹⁴ is recommended.

Flowmeter

20 A portable flowmeter, capable of measuring the desired volumetric flow rate to within 0.1 litre/min, and calibrated against a primary standard (ie a flowmeter whose accuracy is traceable to national standards).

21 Bubble flowmeters are preferred for measuring the volumetric flow rate because the readings they give are independent of temperature and pressure. For other flowmeters, it may be necessary to measure the temperature and pressure at the time of use and apply corrections if these differ from the conditions under which the flowmeter was calibrated.

Ancillary equipment

22 Flexible plastic tubing, of a diameter suitable for ensuring a leakproof fit, to connect the sampler to the pump; belts or harnesses to which the sampling pumps can conveniently be fixed, unless they are small enough to fit inside the worker's pockets

23 A means to transport the samples from the workplace to the laboratory, which minimises the possibility of accidental transfers of material to or from the filter; flat-tipped tweezers for handling the filters.

LABORATORY APPARATUS

Microbalance

24 An electronic microbalance with readability (ie the finest division on the scale) of 0.01 mg or better.

Glassware

25 A selection of laboratory glassware including beakers, measuring cylinders and covered petri dishes.

SAMPLING

Air sampling procedure

26 Use the samplers at the designated flow rate, so that they exhibit the required collection characteristics. Refer to the manufacturer's instructions.

27 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 EH42.¹⁵)

Preparation of air sampling equipment

28 Perform the following in a clean area, and wear disposable gloves to prevent the possibility of contamination from the hands.

29 Allow the filters (or filter cassettes) to equilibrate with the laboratory air overnight before weighing. Calibrate the microbalance and weigh a check weight to ensure that the reading is correct. Weigh the filters (or filter cassettes) to be used for sampling. Set aside some to be used as blank filters. A minimum of three filters should be used as blanks in every twenty filters used. Store the filters in labelled sample tins. The sample tins should be cleaned to avoid any contamination of the filter.

30 Clean the samplers before use. Disassemble the samplers, rinse thoroughly with water, wipe with absorptive tissue and allow to dry thoroughly before reassembly.

31 Load the filters into the clean, dry samplers using clean, flat-tipped tweezers. Connect each loaded sampler to a sampling pump using plastic tubing, ensuring that no leaks can occur. Switch on the pump and measure the flow through the sampler inlet orifice with the calibrated flowmeter. Set the appropriate flow rate with an accuracy of $\pm 5\%$.

32 If the pump needs to be warmed up before use, run it for an appropriate period with an unweighed spare filter in the sampler (following the manufacturer's recommendations). After the flow rate has stabilised, discard the used filter and load a pre-weighed filter into the sampler for collection of the sample. Readjust the flow rate using the calibrated flowmeter to the appropriate value with an accuracy of $\pm 5\%$.

33 Switch off the pump and seal the sampler with its protective cover to prevent contamination with oil mist during transport to the sampling position.

Collection of air samples

34 Fix the sampler to the lapel of the operator, in the breathing zone and as close to the mouth and nose as is reasonably practicable. Then, either place the sampling pump in a convenient pocket or attach it to the operator in a manner that causes minimum inconvenience, eg to a belt 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.

35 Since it is possible for a filter to become clogged, monitor the performance of the sampler frequently during sampling. Measure the flow rate with an accuracy of $\pm 5\%$ using the calibrated flowmeter 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.

36 At the end of the sampling period, measure the flow rate with an accuracy of $\pm 5\%$ using the calibrated flowmeter, switch off the sampling pump, and record the flow rate and the time. Also note 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.

37 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.

38 The blank filters (see paragraph 29) should be subjected to exactly the same handling procedure as the sample filters, but should not have air drawn through them.

Transportation

39 Perform the following in a clean area, where oil mist is absent, and wear disposable gloves to prevent the possibility of contamination from the hands. Prepare the filters for transportation back to the laboratory. Discard any samples that display evidence of either splash contamination or physical damage.

40 For samplers that collect the inhalable fraction of airborne particles on the filter alone (see paragraph 17), remove the filter from each sampler using clean flat-tipped tweezers, and place in the labelled sample filter tin.

41 For samplers that use an internal cassette (see paragraph 17), remove the cassette from each sampler and fasten with the transport clip supplied by the manufacturer. Alternatively, seal the samples within the sampler.

42 Transport the samples back to the laboratory in a container which has been designed to prevent damage in transit and which has been labelled to assure proper handling.

43 There are no data on the effect of storage time on oil mist samples, but it is thought unlikely that the higher viscosity oils sampled in this method will lose any material. In the laboratory, store the samples in a refrigerator if they are not to be analysed immediately.

ANALYSIS

Cleaning of glassware

44 Before use, clean all glassware to remove any residual substances. Rinse with acetone and allow to dry.

Determination of total inhalable particulate concentration

45 Allow the filters (or filter cassettes) to equilibrate with laboratory air overnight before weighing. Calibrate the balance and weigh a check weight to ensure that the reading is correct.

46 Weigh the samples and blanks and calculate the total inhalable particulate airborne concentration (oil mist and other airborne particles) using the formula below:

$$\text{TIP} = \frac{((M_2 - M_1) - (B_2 - B_1)) \times 1000}{F \times T}$$

where TIP = total inhalable particulate (mg/m^3)
 M_1 = weight of sample filter before sampling (mg)
 M_2 = weight of sample filter after sampling (mg)
 $B_2 - B_1$ = average blank filter weight change (mg)
 F = average flow rate during sampling (l/min)
 T = sampling time (min).

47 Calculate a detection limit by multiplying the standard deviation of the blank filter weight changes by three. If the blank-corrected sample filter weight changes are less than this detection limit, then the values calculated should not be relied upon. Record as being less than the limit of detection. The airborne value for the limit of detection (the analytical value divided by the air volume sampled) should be an order of magnitude less than the OES, otherwise the analysis should be deemed invalid.

48 If the total inhalable particulate airborne concentrations exceed $2.5 \text{ mg}/\text{m}^3$ (half the OES), the filters should be analysed further by extracting the oil from the filters and re-weighing the dry filters to determine the mass of oil removed.

Determination of oil mist concentration

49 Extract each filter in a covered glass petri dish with a 10 ml aliquot of cyclohexane, for a period of one hour. Carefully decant the cyclohexane and perform a second extraction with another 10 ml aliquot of cyclohexane.

50 Remove the filter from the petri dish using clean flat-tipped tweezers. Allow the filters to dry in a fume cupboard, ensuring that they cannot pick up airborne dust or be damaged.

51 Re-weigh the filters after equilibration overnight in the laboratory, and calculate the oil mist concentration using the formula below:

$$\text{OM} = \frac{((M_2 - M_3) - (B_2 - B_3)) \times 1000}{F \times T}$$

where OM = oil mist concentration (mg/m^3)
 M_2 = sample filter weight before solvent extraction (mg)

- M_3 = sample filter weight after solvent extraction (mg)
 $B_2 - B_3$ = average blank filter weight change (mg)
 F = average flow rate during sampling (l/min)
 T = sampling time (min).

52 Calculate a limit of detection by multiplying the standard deviation of the blank filter weight changes by three. If the blank-corrected sample filter weight changes are less than this detection limit, then the values calculated should not be relied upon. Record as being less than the limit of detection. The airborne value for the limit of detection (the analytical value divided by the air volume sampled) should be an order of magnitude less than the OES, otherwise the analysis should be deemed invalid.

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.

APPENDIX A

Recommendations for the test report

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

- a complete identification of the air sample, including the date of sampling, the place of sampling, the identity of the machinery in use, and the identity of the operator whose breathing zone was sampled;
- a reference to this MDHS and a description of any deviation from the procedures described;
- the type of filter used;
- the type of sampler used;
- the type of sampling pump used;
- 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;
- the time at the start and at the end of the sampling period, and sampling time in minutes;
- the volume of air sampled, in litres;
- the name of the person who collected the sample;
- the time-weighted average mass concentration of

total inhalable particulate to which the operator was exposed, in milligrams per cubic metre, and a limit of detection for the analysis;

- the time-weighted average mass concentration of oil mist to which the operator was exposed, in milligrams per cubic metre, and a limit of detection for the analysis;
- the name of the analyst;
- the date of the analysis.

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