

Measurement of oil mist from mineral oil-based metalworking fluids

Laboratory method using inhalable sampler and gravimetric analysis

MDHS84/2

Methods for the
Determination of
Hazardous Substances

Health and Safety
Laboratory

Scope

1 This method describes a gravimetric procedure for the measurement of time-weighted average concentrations of oil mist. Oil mist may be formed from mineral oil used as a coolant or lubricant during the machining of metal components. It is applicable to aerosol formed by mineral oils with viscosities greater than $18 \text{ mm}^2 \text{ s}^{-1}$ (or centi-Stokes, cSt) at $40 \text{ }^\circ\text{C}$. Lower viscosity oils contain a greater proportion of volatile components. The aerosol of a low viscosity oil may be unstable and lose volatile constituents while trapped on a filter during air monitoring, consequently the gravimetric analysis may underestimate the original airborne aerosol concentration in these samples.

Summary

2 A measured volume of air is drawn through a pre-weighed filter mounted in an inhalable dust sampler. The filter is reweighed to determine the mass of inhalable aerosol deposited on the filter.

3 If the inhalable particulate concentration is greater than half of any specified target value, it is recommended that the filter should be further extracted in cyclohexane to remove any contribution from any potential interferences deposited on it, by reweighing the filter after extraction.

4 The use of alternative methods not included in the MDHS series is acceptable provided they can demonstrate the accuracy and reliability appropriate to the application.

Recommended sampling

5 Sampling should be carried out as described in MDHS14¹ for inhalable dusts for 2 to 8 hours with a recommended minimum sample volume of 240 litres at 2 litres per minute.

Prerequisites

6 Users of this method will need to be familiar with the content of MDHS14.¹

Safety

7 Users of this method should be familiar with standard laboratory practice and 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

- 8 An inhalable dust sampler, pre-cleaned as specified by the manufacturer: An IOM sampler operated as described in MDHS14¹ has been found suitable.
- 9 Binder-free glass fibre or mixed cellulose ester membrane filters (0.8 µm mean pore diameter). A 25 mm glass fibre GF/A filter has been found to be suitable for use with the IOM sampler.
- 10 Personal sampling pumps that meet the requirements of BS EN 13137.²
- 11 A portable flow meter calibrated against a primary standard, with a measurement uncertainty typically less than ±2%.
- 12 Flexible plastic tubing for making a leak-proof connection from the sampling train to the pump; belts or harnesses to facilitate attachment of sampling apparatus to subjects; flat-tipped tweezers for loading and unloading the filters into sampler cassettes.

Laboratory apparatus and reagents

- 13 During the analysis, use only reagents of a recognised analytical grade.
- 14 Cyclohexane (>99.5% purity).
- 15 A microbalance calibrated against a primary standard, capable of weighing to a precision of 10 µg or better.
- 16 A selection of laboratory glassware including beakers, measuring cylinders and covered Petri dishes cleaned with acetone and dried.

Preparation and sampling

- 17 Load the filters in a clean dust-free environment into clean filter cassettes with the flat tipped tweezers and allow equilibration with laboratory air overnight before weighing. Wear disposable gloves to assemble the filter cassettes to avoid contamination from the hands.
- 18 Weigh the filter cassettes and place in their transport clips or load into the inhalable dust sampler and cap with the protective covers.
- 19 Set aside a minimum of six pre-weighed filter cassettes to be used as blanks. Where more than forty samples are taken, a minimum of three blank filters should be used for every twenty samples. Ensure that the blanks are treated in the same way as the samples but without drawing air through them.
- 20 Sampling should be carried out in accordance with the procedures described in MDHS14¹ for inhalable dust.
- 21 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).
- 22 Connect each sampler, excluding the blanks, to a sampling pump using plastic tubing, ensuring that no leaks can occur and set the flow rate using the calibrated flow meter.

23 Attach the sampler in the breathing zone of the subject within 200 mm of the mouth and nose.

24 When ready to begin sampling, remove the protective cover from the sampler, switch on the pump and check and adjust the flow rate if necessary.

25 Record the time and sample details at the start of the sampling period.

26 At the end of the sampling period, measure the flow rate using the calibrated flow meter, switch off the sampling pump, and record the flow rate and the time. Also note the reading on the elapsed time indicator, if fitted.

27 Reseal the sampler with its protective cover and disconnect it from the sampling pump.

28 In a clean area, where oil mist is absent, and wearing clean disposable gloves, remove the filter cassette from each sampler, place in their transport clips and transport back to the laboratory in a suitable container. Alternatively, transport the samples in the capped sampling heads.

29 Store the samples in a refrigerator if they are not to be analysed immediately.

Sample analysis

30 Allow the filters cassettes to equilibrate with laboratory air overnight before weighing.

Determination of total inhalable particulate concentration

31 Weigh the samples and blanks cassettes and calculate the total inhalable particulate airborne concentration (oil mist and other airborne particles) using the equation:

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

where

TIP = total inhalable particulate (oil mist and other airborne particles) (mg.m⁻³)

M₁ = weight of sample cassette before sampling (mg)

M₂ = weight of sample cassette after sampling (mg)

B₁ = average blank filter weight change (mg)

F = average flow rate during sampling (l.min⁻¹)

T = sampling time (min)

32 Calculate the limit of detection by multiplying the standard deviation of the weight changes in the blank filters by three. Where the blank-corrected weight change of a sample filter is less than the limit of detection, record the total inhalable particulate concentration of the sample as less than the limit of detection.

33 If the total inhalable particulate airborne concentration exceeds half of any target value, the filters should be further analysed by extracting the oil from the filters and determining the oil mist concentration.

Determination of oil mist concentration

34 Extract each filter for 1 hour in a covered glass Petri dish in 10 ml of cyclohexane. Carefully decant the cyclohexane and perform a second extraction with a further 10 ml of cyclohexane.

35 Remove the filter from the petri dish using clean flat-tipped tweezers and allow to dry in a dust-free fume cupboard.

36 Reweigh the filters after equilibration overnight and calculate the oil mist concentration using the equation:

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

where

OM = oil mist concentration (mg.m⁻³)

M₂ = sample filter weight before solvent extraction (mg)

M₃ = sample filter weight after solvent extraction (mg)

B₂ = average blank filter weight change after extraction (mg)

37 Calculate the limit of detection by multiplying the standard deviation of the weight changes in the blank filter by three. Where the blank-corrected weight change of a sample filter weight changes is less than the limit of detection, record the oil mist concentration as less than the limit of detection.

Appendix: Additional information

Detection limit

1 Under the stated sampling and analytical conditions, the limit of detection for samples taken over an 8-hour sampling period at 2 l.min⁻¹ is typically around 0.1 mg.m⁻³.

Overall uncertainty

2 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 meet the specifications of BS EN 482.³

Interferences

3 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.

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

References

1 *General methods for sampling and gravimetric analysis of respirable, thoracic and inhalable aerosols* MDHS14/4 HSE 2014
www.hse.gov.uk/pubns/mdhs/index.htm

2 BS EN 13137:2013 *Workplace atmospheres. Pumps for personal sampling of chemical and biological agents. Requirements and test methods* British Standards Institution

3 BS EN 482:2012 *Workplace exposure. General requirements for the performance of procedures for the measurement of chemical agents* British Standards Institution

You should use the 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.

British Standards can be obtained in PDF or hard copy formats from BSI:
<http://shop.bsigroup.com> or by contacting BSI Customer Services for hard copies only Tel: 020 8996 9001 email: cservices@bsigroup.com.

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