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Workplace air — Analysis of airborne water immiscible mineral oil droplets and vapor with Fourier — Transform infrared spectroscopy

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A list of organizations represented on this committee can be obtained on request to its committee manager.

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Workplace air — Analysis of airborne water immiscible mineral oil droplets and vapor with Fourier — Transform infrared spectroscopy

Air des lieux de travail — Analyse par transformée de Fourier des vapeurs et des gouttelettes d'huile minérale non miscibles à l'eau en suspension dans l'air — Spectroscopie infrarouge à transformée de Fourier

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

Mineral oils are by-products from the distillation of crude oil. The mineral oils produced in this way are used in a wide range of modern applications. Their composition varies according to their application. Mineral oils contain saturated hydrocarbons, cyclic saturated hydrocarbons, aromatic compounds, and unsaturated hydrocarbons.

They are used as fuels, for example in the form of petroleum, diesel and kerosene for internal combustion engines, as heating oil, and as lubricants in the metal processing and application industries. Mineral oils are also found in cosmetics and medical applications. This wide range of applications gives rise to exposure for human beings and the natural environment to mineral oils in the form of vapours and droplets. Since mineral oils and their constituents are harmful to health, it is important for quantification of the exposure to be possible and where necessary for corrective measures to be taken.

The method described in this document is intended to support swift, cost-effective and straightforward quantification of the concentration of mineral oil in a large number of samples. This method has been validated in accordance with the provisions of EN 13936 and ISO 20581 and has an expanded measurement uncertainty in the region of 20 % to 30 % within the measurement range stated.

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Workplace air — Analysis of airborne water immiscible mineral oil droplets and vapor with Fourier — Transform infrared spectroscopy

1 Scope

The method described in this document quantifies the absolute exposure to mineral oil vapours and droplets, within a concentration range from 0,5 mg/m^3 to 125 mg/m^3, in the inhalable fraction of the workplace air.

This document contains comprehensive information and instructions on the equipment and chemicals to be used.

This method is applicable for water soluble oils and metal working fluids.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 7708, *Air quality — Particle size fraction definitions for health-related sampling*

ISO 13137:2013, *Workplace atmospheres — Pumps for personal sampling of chemical and biological agents — Requirements and test methods*

ISO 18158, *Workplace air — Terminology*

ISO 20581, *Workplace air — General requirements for the performance of procedures for the measurement of chemical agents*

EN 13936, *Workplace exposure — Procedures for measuring a chemical agent present as a mixture of airborne particles and vapour — Requirements and test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

A defined airflow is drawn over the sampler with the aid of a pump. The droplets are deposited on the glass fibre filter, the vapours on the polymer resin. By extraction of the glass fibre filter and the polymer resin, the adsorbed mineral oils are desorbed and can be analysed by means of Fourier trasnform infrared spectrocopy (FTIR) and evaluated.

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The Gesamtstaub-Gas-Probenahme (GGP) sampling system enables both droplets and vapours to be collected but any other personal sampler capable of collecting the inhalable droplets and vapours simultenously can be used. A glass fibre filter is used to collect the droplets. A downstream sampling tube containing polymer resin is used to collect the vapours and more volatile components in the mineral oil. The GGP system cannot be used for distinction between exposure to gases and droplets, since droplets collected during sampling can vaporize from the filter and collect on the polymer resin.

Following extraction, the samples are analysed by Fourier transform infrared spectroscopy (FTIR) in the wavenumber range from 3 000 cm^{-1} to 2 800 cm^{-1}. This method is not specific for mineral oil, since it detects all substances with C-H bonds collected on the sampler.

The principle of the method described here for the quantification of mineral oil is based upon activation of the C-H bonds of the hydrocarbons in the mineral oil. Hydrocarbons absorb light in the wavenumber range from 3 000 cm^{-1} to 2 800 cm^{-1}. This enables the concentration of mineral oil at the site of sampling to be determined by external calibration against a defined mineral oil at known concentrations.

The method described in this document is based upon the "MAK Collection for Occupational Health and Safety 2016, Vol 1, No 1", "Cooling lubricants and other complex hydrocarbon mixtures, immiscible with water – droplets and vapours"[2].

5 Apparatus and reagents

5.1 Collecting materials for analysis and other chemicals

5.1.1 Glass fibre filter.

The droplets shall be collected by a binder-free 85/90 glass fibre filter with a diameter of 37 mm.

Glass fibre filters generally have no blank value. This shall however be verified beforehand.

5.1.2 Polymer resin.

Poly[styrene-divinylbenzene], a mesoporous polymer resin in the particle size range from 0,5 mm to 0,9 mm shall be used for collection of the vapour.

Polymer resins are well suited as a collection phase for mineral oils. This has been demonstrated with reference to Amberlite® XAD-2[1]. XAD-2 requires cleaning prior to use in case it exhibits a blank value.

Any other polymer resin equivalent to XAD-2 can be accepted if it is validated according to Annex A.

5.1.3 Tetrachloroethene.

Tetrachloroethene, also know as perchloroethylene, in the purest grade (≥99,0 %) is used to extract the samples and to prepare the calibration standards, and to clean the polymer resin if necessary.

Any other solvent can be used if it is validated according to Annex A.

5.1.4 Mineral oil, for calibration purposes.

The mineral oil used for evaluation of the air samples shall be that used at the workplace.

Fresh unused mineral oil is usually preferred for practical reasons.

If there are several mineral oil products used, it is recommended to use the one with the lower mineral oil content. This leads to a conservative result.

1) Amberlite® XAD-2 is the trade name of a product supplied by Sigma Aldrich. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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5.1.5 Liquid paraffin standard.

In case it not be possible to obtain a site-specific oil sample or if this sample is not soluble in tetrachloroethene, a mineral oil standard for spectroscopy, namely liquid paraffin, CAS number 8012-95-1, should be used for evaluation.

The different response factors against the mineral oils of the workplaces which were used for this validation are listed in Table A.13 and Table A.14.

5.2 Sampler equipment

The following equipment is required for sampling.

The performance of the samplers used shall match the criteria for inhalable fraction as specified in ISO 7708.

5.2.1 Sampling pump, conforming with the requirements of ISO 13137.

5.2.2 Personal sampler, shall be used for the simultaneous collection of inhalable droplets and vapours. Any personal sampler capable of collecting the inhalable droplets and vapours simultaneously may be used; for the validation experiments written in this document, the GGP sampling system, with matching cone for the specified flow rate (3,5 l/min) was used[4].

The GGP sampler is a modification of the GSP sampler (see CEN/TR 15230[11]), which match the criteria of ISO 7708. Sampling efficiency of this modification was determined by George C. Dragan et al. (2015)[9].

5.2.2.1 Various lengths of **tubes** (e.g. outer diameter: 9 mm or 10 mm; inner diameter: 6 mm), for connecting the personal sampler to the pump.

5.2.2.2 Matching **filter capsule** for the 37 mm glass fibre filter, for insertion into the personal sampler.

5.2.2.3 Plastic cartridge manufactured from polyvinylidene fluoride (PVDF).

Matching plastic tube (outer diameter: 20 mm; inner diameter: 16 mm, length: 70 mm) for the polymer resin for insertion into the personal sampling system, with high-grade steel sieve stopper and plastic sealing caps.

5.2.2.4 Silicone adapter, which shall fit onto the sampler head to connect the flow meter for measuring/setting the air flow.

5.2.3 Flow meter, conforming with the requirements for test instruments in ISO 13137:2013, 6.2.

5.2.4 Glass metering aid, for filling the plastic cartridge with 8 ml polymer resin.

5.2.5 A 500 ml glass **Erlenmeyer flask** for the tilting dispenser head.

5.3 Analytical equipment

5.3.1 Dispenser, for organic solvents, with a volume of 1 ml to 10 ml, shall be used for extraction of the samples and preparation of the calibration.

5.3.2 Piston pipettes with tips for organic solvents, with a volume range of 30 µl to 300 µl and of 0,5 ml to 5,0 ml are required for preparation of the calibration.

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