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Workplace air — Chemical agent present as a mixture of airborne particles and vapour — Requirements for evaluation of measuring procedures using samplers

Air des lieux de travail — Agent chimique présent sous forme de mélange de particules en suspension dans l'air et de vapeur — Exigences d'évaluation des procédures de mesure utilisant des dispositifs de prélèvement

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Foreword

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This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 137, *Assessment of workplace exposure to chemical and biological agents,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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.

Introduction

This document provides a framework for assessing the performance of procedures for measuring a chemical agent present as a mixture of airborne particles and vapour against the general requirements for the performance of procedures for measuring chemical agents in workplace atmospheres as specified in ISO 20581.

This document enables manufacturers, users of samplers, developers and users of procedures for measuring a chemical agent present as a mixture of airborne particles and vapour to adopt a consistent approach to method validation.

This document is based on EN 13936.

Workplace air — Chemical agent present as a mixture of airborne particles and vapour — Requirements for evaluation of measuring procedures using samplers

1 Scope

This document specifies requirements for the evaluation of measuring procedures using samplers for the determination of a chemical agent present in the workplace atmosphere as a mixture of airborne particles and vapour.

The procedures given in this document provide results only for the sum of airborne particles and vapour. The concentration is calculated in terms of mass per unit volume.

NOTE The physical behaviour of a mixture of airborne particles and vapour is described in Annex A. Examples of substances which can be present in multiple phases are toluene diisocyanate, diethanolamine, ethyleneglycol and tributylphosphate.

This document can also be applied to complex mixtures, such as metal working fluids or bitumen fumes.

This document is applicable to samplers and measuring procedures using these samplers in which sampling and analysis are carried out in separate stages.

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

ISO 13137, *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*

ISO 21832, *Workplace air — Metals and metalloids in airborne particles — Requirements for evaluation of measuring procedures*

ISO 22065:2020, *Workplace air — Gases and vapours — Requirements for evaluation of measuring procedures using pumped samplers*

EN 13205-1, *Workplace exposure — Assessment of sampler performance for measurement of airborne particle concentrations — Part 1: General requirements*

3 Terms and definitions

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

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

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

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

3.1
mixed-phase sampler
sampler or sampling train that is used to collect airborne particles and vapours onto one or more collection substrates

[SOURCE: ISO 18158:2016, 2.2.2.1.7, modified — the given cross-references have been removed.]

3.2
joint extraction mode
procedure that simultaneously extracts and analyses all collection substrates contained in the *mixed-phase sampler* (3.1), resulting in a unique quantification of the analyte for each air sample

3.3
separate extraction mode
procedure that separately extracts and analyses the collection substrates contained in the *mixed-phase sampler* (3.1), resulting in multiple quantifications for each air sample that are summed to give the final result

4 Symbols and abbreviated terms

NOTE For symbols used for calculation of combined standard uncertainty see C.2.

U.S. EPA	is the United States environmental protection agency
k	is the coverage factor
K_v	is the coefficient of variation
LV	is the limit value
m_min	is the minimum mass which shall be quantified
m_p	is the mass determined on the collection substrate for airborne particles, in milligrams;
m_v	is the mass determined on the collection substrate for vapour, in the same unit as for m_p.
N	is the number of extractions realized to analyse all collection substrates, control sections excluded
Q	is the recommended air flow rate of the mixed-phase sampler
R_an	is the analytical recovery
RH	is the relative humidity
SVOC	is a semi-volatile organic compound
t_min	is the minimum sampling time
u_c	is the combined standard uncertainty
U	is the expanded uncertainty
$\gamma_\mathrm{d,p}$	is the distribution coefficient for airborne particles, in percentage;
$\gamma_\mathrm{d,v}$	is the distribution coefficient for vapour, in percentage;
ρ_LV	is the limit value considered
x	is the fraction of LV

5 Sampler types

Samplers are classified based on differences in the collection substrate because of differences in the analytical procedures.

Where the vapour phase is collected on a sorbent bed, the mixed-phase sampler is classified as type A sampler.

Where the vapour phase is collected on an impregnated filter, the mixed-phase sampler is classified as type B sampler.

NOTE Other systems, for example denuder and filter or impinger and filter, can be used alternatively for specific chemical agents. See Annex B.

6 Requirements

6.1 General

The measuring procedure used shall comply with the requirements of ISO 20581 and those clauses of ISO 13137, ISO 21832, ISO 22065 and EN 13205-1 which apply.

When the use of a sampler for measurement of a particular mixture of airborne particles and vapour is claimed, the sampler shall meet the requirements specified in 6.2. Measuring procedures shall meet the requirements specified in 6.3.

Known or suspected interferences as well as the results of any tests performed to evaluate interferences, including suitable and sufficient information to minimize their effects shall be presented in the method description as required by 6.3.4.

6.2 Sampler requirements

6.2.1 General

The sampler shall comply with the general requirements given in EN 13205-1 and with the performance requirements for pumped samplers prescribed in ISO 22065:2020, 6.2.2 to 6.2.6.

6.2.2 Flow resistance and stability of the air flow

The back pressure of the mixed-phase sampler shall not exceed the maximum values specified for the pump performance test in ISO 13137, unless the combination of mixed-phase sampler and pump has been tested and shown to be able to sample for the required sampling period.

The air flow through the sampling train associated with the pump shall be measured over the entire sampling period and not deviate more than 5 % as specified in ISO 13137.

Pumps used with size-selective mixed-phase samplers shall also meet the requirements of the pump pulsation test as specified in ISO 13137.

6.2.3 Connecting parts

The volume of any connecting parts between collection substrates within the mixed-phase sampler shall be kept to a minimum and any connection shall be made of an inert material that

— does not retain the chemical agent of interest,

— does not react with the chemical agent of interest,

— does not emit chemical agents that can interfere with the one of interest, and

— is resistant to solvents, if applicable.

6.2.4 Pumps

Pumps used in the measuring procedure shall conform with ISO 13137.

6.3 Measuring procedure requirements

6.3.1 Sampling procedure requirements

6.3.1.1 General

Measuring procedures shall specify the use of a mixed-phase sampler designed to collect the inhalable fraction of airborne particles, as defined in ISO 7708, and vapour.

The requirements specified in ISO 22065:2020, 6.3.1, shall apply according to the types of collection substrates that are used in the mixed-phase sampler.

NOTE Due to the particularity of mixed-phase samplers, some requirements are adapted from ISO 22065 as given in 6.3.1.2 and 6.3.1.3.

6.3.1.2 Air flow rate

For type A samplers, the air flow rate constrained by the particle-size selector of the sampler should not exceed the maximal air flow rate of the sorbent tube. If not, the air flow should be split to achieve this requirement.

For type B samplers, the maximum air flow rate to ensure complete sampling according to ISO 22065:2020, 6.3.1.3.1, shall comply with the air flow rate required by the particle-size selector used.

6.3.1.3 Storage condition after sampling

When tested in accordance with the procedure prescribed in 8.2.2, the mean analytical recovery after storage shall not differ by more than 10 % from the value before storage.

6.3.2 Analytical procedure requirements

6.3.2.1 General

The requirements given in ISO 22065:2020, 6.3.2, shall apply according to the types of collection substrates that are used in the mixed-phase sampler.

NOTE Due to the particularity of mixed-phase samplers, some requirements are adapted from ISO 22065 as stated in 6.3.2.2 to 6.3.2.5.

6.3.2.2 Extraction of the collection substrates

The extraction procedure shall ensure that all phases are extracted and presented for analysis of total mass of the analyte(s) of interest.

When collection substrates are extracted and analysed separately, the masses determined on each collection substrate shall not be interpreted as an accurate separation of a particle fraction or vapour fraction as these fractions were not stabilized during the sampling period and thus, transfer can occur between collection substrates.

NOTE A preponderance of analyte on the portion of the sampler intended for either particulate or vapour collection can give valuable guidance regarding the environment and the control measures, including respiratory protection measures, which can be needed for implementation. Samplers which consist of a filter and adsorbent are not able to give an accurate assessment of partition, but samplers have been and are being designed to provide more accurate information.

6.3.2.3 Analytical limit of quantification

The analytical limit of quantification shall be lower than or equal to m_{min}. The minimum mass of analyte m_{min} that would be collected for the minimum air sample volume specified in the measuring procedure at the following concentrations is calculated by Formula (1):

$$m_{\text{min}} = \frac{(x \cdot \rho_{\text{LV}})}{N} \cdot Q \cdot t_{\text{min}} \qquad (1)$$

where

m_{min} is the minimum mass which shall be quantified;

x is the fraction of LV considered as follows:

- $x = 0{,}1$ for substances with long-term limit value, and
- $x = 0{,}5$ for substances with short-term limit value;

ρ_{LV} is the limit value considered;

N is the number of extractions realized to analyse all collection substrates, control sections excluded;

Q is the recommended air flow rate of the mixed-phase sampler;

t_{min} is the minimum sampling time.

6.3.2.4 Analytical recovery

For extraction when tested in accordance with ISO 22065:2020, 8.3.2.2.3, the analytical recovery R_{an} shall be ≥75 % with $K_{\text{v}} \leq 10$ % at each loading.

The values given for analytical recovery are targets; lower values may be used provided equivalent precision is achieved.

6.3.2.5 Blank value

In order to obtain acceptable values for the limit of quantification of the method, the blank values of the collection substrates should be as low as technically possible.

When tested in accordance with ISO 22065:2020, 8.3.2.3, the total of the blank values shall be less than one-tenth of the mass calculated by Formula (1).

NOTE Higher blank values can be allowed provided the requirement of 6.3.2.3 is met.

Where it is known that a blank value is significant and varies between batches of samplers, it shall be checked for each batch.

6.3.3 Expanded uncertainty

When tested in accordance with ISO 22065:2020, 8.3, the expanded uncertainty of the measuring procedure as a whole, including the measurement of airborne particles and vapour, shall comply with the requirements of ISO 20581. For the uncertainty budget of the airborne particles, the numbers given for inhalable samplers in ISO 21832:2018, C.3.4, can be used.

When fractions are analysed separately, the expanded uncertainty can be calculated according to Clause C.3.

6.3.4 Method description

ISO 22065:2020, 6.3.4, shall apply.

7 General test conditions

7.1 Reagents

Use only reagents of recognized analytical grade.

7.2 Apparatus

Test equipment as stated in ISO 22065:2020, 7.2.2 to 7.2.7, shall be used except for 7.2.3.

NOTE 1 A dynamic system for generating, pre-mixing and delivering a known concentration of a test atmosphere that contains known concentrations of vapour and particles of a semi-volatile compound is technically difficult to obtain compared to a test atmosphere containing only vapour as prescribed in ISO 22065. The apparatus described in ISO 22065:2020, 7.2.3, can be used to generate the test atmospheres of pure air in the climatic conditions required by the tests.

NOTE 2 Test atmospheres generated at high concentrations tend to bias towards the aerosol phase compared to lower concentrations that can be observed in the workplace; therefore, it is important that the concentrations in the test atmosphere are relevant to the sampling situations at workplaces.

8 Test methods

8.1 Spiking method

8.1.1 General

The spiking method allows the deposit of the analyte on the collection substrates under controlled conditions. Only if no standard atmosphere chamber for mixtures of vapour and aerosols is available the spiking method described in 8.1.2 shall be used.

Tests prescribed in 8.2 need the analyte to be spiked on the collection substrates. Due to the semi-volatile characteristic of the analyte, this spiking shall be made as required by 8.1.2 to 8.1.4.

8.1.2 Deposit of the analyte on the first collection substrate

8.1.2.1 Type A samplers

The first collection substrate of a type A sampler is a filter. A deposit of the analyte in the form of a spot with micropipette or syringe shall be avoided due to the dramatic reduction of the evaporation surface in comparison with the one presented by the micrometric airborne particles collected on the surface of the filter during the sampling period. If so, the transfer of the analyte from the filter to the sorbent bed will not be realistic.