SLOVENSKI STANDARD
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Izpostavljenost na delovnem mestu - Postopki za merjenje kovin in polkovin v lebdeļ ih delcih - Zahteve in preskusne metode

Workplace exposure - Procedures for measuring metals and metalloids in airborne particles - Requirements and test methods

Exposition am Arbeitsplatz - Messung von Metallen und Metalloiden in luftgetragenen Partikeln - Anforderungen und Prüfverfahren

Exposition sur les lieux de travail - Procédures pour le mesurage des métaux et métalloïdes dans les particules en suspension dans l'air - Exigences et méthodes d'essai

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Workplace exposure - Procedures for measuring metals and metalloids in airborne particles - Requirements and test methods

Exposition sur les lieux de travail - Procédures pour le mesurage des métaux et métalloïdes dans les particules en suspension dans l'air - Exigences et méthodes d'essai

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Foreword

This document (EN 13890:2009) has been prepared by Technical Committee CEN/TC 137 "Assessment of workplace exposure to chemical and biological agents", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2010, and conflicting national standards shall be withdrawn at the latest by March 2010.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 13890:2002.

The major technical changes between this European Standard and the previous edition are as follows:

a) Adaptation of the framework for assessing the performance of procedures for measuring metals and metalloids against the general requirements for the performance of procedures for measuring chemical agents in workplace atmospheres as specified in EN 482;

b) Revision of the calculation model for the uncertainty of measurement to comply with EN 482 and ENV 13005.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

This European Standard provides a framework for assessing the performance of procedures for measuring metals and metalloids against the general requirements for the performance of procedures for measuring chemical agents in workplace atmospheres as specified in EN 482. It enables producers and users of procedures for measuring metals and metalloids in airborne particles to adopt a consistent approach to method validation.

Although this European Standard has been written for assessing the performance of procedures for measuring metals and metalloids, it can be used as the basis for assessing the performance of procedures for measuring other chemical agents that are present as or in airborne particles, e.g. sulphuric acid mist.

1 Scope

This European Standard specifies performance requirements and test methods for the evaluation of procedures for measuring metals and metalloids in airborne particles sampled onto a suitable collection substrate, e.g. a filter.

This European Standard specifies a method for estimating the uncertainties associated with random and systematic errors and combining them to calculate the expanded uncertainty of the measuring procedure as a whole, as prescribed in EN 482.

This European Standard is applicable to measuring procedures in which sampling and analysis is carried out in separate stages, but it does not specify performance requirements for collection, transport and storage of samples, since these are dealt with in EN 13205 and ISO 15767.

This European Standard is not applicable to procedures for measuring metals or metalloids present as inorganic gases or vapours, e.g. mercury, arsenic (see EN 838 and EN 1076), or to procedures for measuring metals and metalloids in compounds that could be present as a particle/vapour mixture, e.g. arsenic trioxide.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 481, *Workplace atmospheres — Size fraction definitions for measurement of airborne particles*

EN 482:2006, *Workplace atmospheres — General requirements for the performance of procedures for the measurement of chemical agents*

EN 1232:1997, *Workplace atmospheres — Pumps for personal sampling of chemical agents — Requirements and test methods*

EN 1540:1998, *Workplace atmospheres — Terminology*

EN 12919, *Workplace atmospheres — Pumps for the sampling of chemical agents with a volume flow rate of over 5 l/min — Requirements and test methods*

EN 13205[1)] *Workplace atmospheres — Assessment of performance of instruments for measurement of airborne particle concentrations*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 482:2006 and EN 1540:1998[2)] apply.

1) All references to EN 13205 in this European Standard refer to the ongoing revision of EN 13205:2001.

2) EN 1540:1998 is currently subject to revision. Until the revised EN is published the definitions given in EN 482:2006 take precedence.

4 Principle

For measuring procedures that involve sample dissolution, instrumental detection limits are determined by repeat analysis of the calibration blank solution. For all measuring procedures, method detection limits and quantification limits are determined by analysis of laboratory blanks. The determined quantification limits are then assessed against the performance requirements specified in 5.2.1.

Analytical recovery is determined by one of a number of different methods, depending upon the nature of the measuring procedure under evaluation. For measuring procedures for soluble compounds of metals and metalloids, analytical recovery is determined by analysis of spiked laboratory blanks (except for procedures that incorporate a design-based sample dissolution method (see A.1.1), for which it is taken to be 100 %). For measuring procedures for total metals and metalloids that involve sample dissolution, analytical recovery is determined by analysis of pure compounds, reference materials or reference air samples. For measuring procedures for total metals and metalloids that involve analysis of the sample on the collection substrate, analytical recovery is determined by analysis of reference air samples, by the analysis of workplace air samples that are characterised by subsequent analysis using a reference procedure or it is estimated from theory. The determined analytical recovery is then assessed against the performance requirements specified in 5.2.2.

Measurement uncertainty is estimated using a structured approach. Firstly, a cause and effect diagram is constructed to identify individual random and non-random uncertainty components of a measuring procedure. After simplification to resolve any duplication, the resulting diagram is used to identify components for which uncertainty estimates are required. Each of these uncertainty components is then estimated or calculated from experimental data, combined to obtain an estimate of the uncertainty of the measurement method as a whole and multiplied by an appropriate coverage factor to calculate the expanded uncertainty of the method, following the guidance in Annex B. In accordance with 5.2.3, the determined expanded uncertainty is then assessed against the general performance requirements specified in EN 482.

5 Requirements

5.1 Method description

5.1.1 Scope

The scope of the measuring procedure shall give at least information about the following:

— the metals and metalloids covered by the measuring procedure;

— the analytical technique(s) used in the measuring procedure;

— the range of concentrations of metals and metalloids in air for which the measuring procedure has been shown to meet the acceptance criteria for expanded uncertainty prescribed in EN 482, together with the associated range of sampled air volumes (e.g. 0,01 mg · m^{-3} to 0,5 mg · m^{-3} for sampled air volumes in the range 240 l to 960 l);

— any form of the metals and metalloids for which the sample preparation method described has been shown to be or is known to be ineffective; and

— any known interferences.

NOTE If there is no procedure for measuring a particular metal or metalloid that meets the requirements of this European Standard, a measuring procedure whose performance is nearest to the specified requirements should be used.

5.1.2 Method performance

For all metals and metalloids included in the scope of the method, the measuring procedure shall give comprehensive information about method performance, including the following:

— the detection and quantification limits of the measuring procedure;

— the analytical recovery for all test materials for which the sample preparation method has been shown to be effective;

— all random and non-random uncertainty components of the measuring procedure, together with their estimated or experimentally determined values, and the resulting expanded uncertainty; and

— full details of any known interferences, including suitable and sufficient information on how to minimise their effects, if applicable.

5.1.3 Safety information

The measuring procedure shall provide suitable and sufficient information on the safety hazards associated with the reagents and equipment used in the procedure.

5.1.4 Samplers

The measuring procedure shall:

— require the user to select samplers that are designed to collect an appropriate fraction of airborne particles, as defined in EN 481, according to the particle size fraction(s) that is applicable to the limit value for the metals and metalloids of interest (e.g. an inhalable sampler, a thoracic sampler or a respirable sampler);

— specify that the samplers shall comply with the provisions of EN 13205; and

— require, if appropriate, for procedures that do not involve sample dissolution, calibration of the analytical instrument used to be sampler specific, e.g. for X-ray fluorescence spectrometry (XRF).

5.1.5 Sampling pumps

The measuring procedure shall require the user to use sampling pumps that comply with the provisions of EN 1232 or EN 12919.

5.1.6 Other requirements

Where necessary, the measuring procedure shall give other requirements, e.g. for the collection substrate.

5.2 Performance requirements

5.2.1 Quantification limit

For each metal and metalloid included in the scope of the measuring procedure, determine the lower limit of the working range of the method that will be satisfactory for the intended measurement task. For example, if the measurement task is testing compliance with long-term limit values, use Equation (1) to calculate the least amount of the metal or metalloid that needs to be quantified when it is to be determined at a concentration of 0,1 times its limit value:

$$m_g = 0{,}1\ \rho_{LV}\ \cdot q_{v,a} \cdot t_{s,min} \qquad (1)$$

where

m_L is the lower limit of the required analytical range of the metal or metalloid, in micrograms;

ρ_{LV} is the limit value for the metal or metalloid, in milligrams per cubic metre;

$q_{v,a}$ is the design flow rate of the sampler to be used, in litres per minute; and

$t_{s,min}$ is the minimum sampling time that will be used, in minutes.

For procedures that involve sample dissolution, calculate the lower limit of the required working range for each metal and metalloid, in micrograms per millilitre, by dividing the lower limit of the required working range, in micrograms, by the volume of the test solution, in millilitres. When tested in accordance with 8.1.2.1, the determined quantification limits shall be lower than the resulting values.

For procedures that do not involve sample dissolution, when tested in accordance with 8.1.2.2, the determined quantification limits for each metal and metalloid shall be lower than the lower limit of the required working range in micrograms.

5.2.2 Analytical recovery

When tested in accordance with one of the procedures prescribed in 8.2, the mean analytical recovery shall be at least 90 % for all material types included within the scope of the measuring procedure and the coefficient of variation[3)] of the analytical recovery shall be less than 5 %.

5.2.3 Expanded uncertainty

The expanded uncertainty of the measuring procedure shall comply with the requirements specified in EN 482.

6 Reagents and materials

6.1 Reagents

During the analysis, use only reagents of analytical grade, and only water complying with the requirements for EN ISO 3696 grade 2 water (electrical conductivity less than 0,1 mS · m^{-1}, i.e. resistivity greater than 0,01 MΩ · m, at 25 °C).

It is recommended that the water used be obtained from a water purification system that delivers ultrapure water having a resistivity greater than 0,18 MΩ · m (usually expressed by manufacturers of water purification systems as 18 MΩ · cm water).

6.2 Standard solutions

Standard solutions with concentrations of the metals and metalloids of interest that are traceable to national and/or international standards. If commercial standard solutions are used observe the manufacturer's expiry date or recommended shelf life.

3) The predecessor term "relative standard deviation" is deprecated. See also ISO 3534-1:2006, 2.38, Note 2.

6.3 Test materials

For each metal or metalloid, a range of test materials that is representative of the substances of interest that could be present in the workplace atmosphere. The test materials can be pure compounds of known composition, certified reference materials or other well-characterised materials (e.g. materials characterised in an interlaboratory comparison). Follow the supplier's instructions when using certified reference materials.

NOTE 1 If there is a limit value for a specific compound, that compound should be included in the range of reference materials.

NOTE 2 For a method that is intended to have general applicability, the range of reference materials should include compounds and materials in industrial use and compounds and materials which could be generated by the work activity.

NOTE 3 It is important that the particle size of the reference materials be as close as possible to that of the particles analysed, since, compared to coarse bulk materials, inhalable particles are often much smaller and more readily soluble.

NOTE 4 Certified reference materials that have been characterised with respect to a particular sample dissolution method might not be suitable for use as a test material.

6.4 Reference air samples

Samples of dust on collection substrates (e.g. airborne particles collected on filters using a multiple simultaneous sample collection system), having a known or measured loading of the metals and metalloids of interest.

7 Apparatus

Usual laboratory apparatus and resources and:

— a system for applying a known volume of standard solution to collection substrates with a precision of better than 1 %;

— an analytical balance capable of weighing to at least 0,01 mg, calibrated with weights traceable to national standards, checked before use by means of a test weight;

— an instrument or instruments for analysing the metals and metalloids of interest.

8 Test methods

8.1 Detection limits and quantification limits

8.1.1 Instrumental detection limit

8.1.1.1 For measuring procedures that involve sample dissolution, analyse the calibration blank solution at least ten times under repeatability conditions.

If there is no measurable response from the analytical instrument, prepare a test solution with concentrations of the metals or metalloids of interest near their anticipated instrumental detection limits by diluting the standard solutions (6.2) by an appropriate factor. Analyse the test solution at least ten times under repeatability conditions.

NOTE For measuring procedures that involve analysis of the sample on the collection substrate, an instrumental detection limit is not a meaningful concept and, as such, cannot be determined.

8.1.1.2 Calculate the instrumental detection limit for each of the metals or metalloids of interest as three times the standard deviation.

NOTE An instrumental detection limit is of use in identifying changes in instrument performance, but it is not the same as a method detection limit. An instrumental detection limit is likely to be lower than a method detection limit because it only takes into account the variability between individual instrumental readings; determinations made on one solution do not take into consideration contributions to variability from the matrix or sample.

8.1.2 Method detection limits and quantification limits

8.1.2.1 For measuring procedures that involve sample dissolution, prepare at least ten test solutions from laboratory blanks, following the sample preparation method described in the measuring procedure, and analyse the test solutions for the metals or metalloids of interest under repeatability conditions.

If there is no measurable response from the analytical instrument, spike ten laboratory blanks with an appropriate volume of working standard solution containing appropriate known masses of the metals or metalloids of interest, such that the test solutions produced from them will have concentrations near their respective anticipated detection limits. Prepare test solutions from the spiked laboratory blanks, following the sample preparation method described in the measuring procedure, and analyse the test solutions for the metals or metalloids of interest under repeatability conditions.

Calculate the method detection limit and the quantification limit for each of the metals or metalloids of interest as three times and ten times the standard deviation, respectively (see reference [1]).

8.1.2.2 For measuring procedures that do not involve sample dissolution, analyse at least ten laboratory blanks under repeatability conditions.

Calculate the method detection limit and the quantification limit for each of the metals or metalloids of interest as three times and ten times the standard deviation, respectively.

8.1.2.3 Compare the quantification limits obtained with the requirements of 5.2.1.

8.2 Analytical recovery

8.2.1 General

Different test methods are applicable for the determination of analytical recovery, depending on the sample preparation method used. These are detailed separately in 8.2.2, 8.2.3 and 8.2.4. See Annex A for guidance.

8.2.2 Measuring procedures for soluble compounds of metals and metalloids

8.2.2.1 Measuring procedures that incorporate a design-based sample dissolution method

Unless there is a contra-indication (see A.1.2) take the analytical recovery to be 100 % for procedures for soluble compounds of metals and metalloids that incorporate a design-based sample dissolution method (see A.1.1).

8.2.2.2 Other measuring procedures

For measuring procedures that do not incorporate a design-based sample dissolution method or for which there could be a problem of chemical compatibility between the analyte and the substrate, prepare a minimum of six replicate test samples by spiking laboratory blanks with an appropriate volume of working standard solution containing a known mass of each of the metals or metalloids of interest. Then use the sample dissolution method described in the measuring procedure to prepare test solutions from the test samples and analyse the resulting solutions using the analytical method described in the measuring procedure.

Repeat the test on laboratory blanks spiked with other masses of each of the metals or metalloids of interest to determine the analytical recovery across the working range of the measuring procedure.

Calculate the mean analytical recovery and coefficient of variation for each of the tests performed and compare the results with the requirements of 5.2.2. If the requirements are not met, take corrective measures (e.g. use an alternative collection substrate), if possible, and repeat the analytical recovery test.

8.2.3 Measuring procedures for total metals and metalloids that involve sample dissolution

8.2.3.1 Determination of analytical recovery using pure compounds

Prepare a minimum of six test solutions from each of the selected pure compounds (6.3) using the sample preparation method described in the measuring procedure. Use a mass of the pure compound that can be weighed with an accuracy of at least 1 %. Analyse the test solutions as described in the measuring procedure.

NOTE 1 It is usually not necessary to include water-soluble compounds in the range of compounds tested.

NOTE 2 It is preferable to use the smallest mass of pure compound that can be easily weighed, to scale up the volume of reagents and to adjust the final test solution volume so that the experiment is as representative as possible of the analysis of workplace air samples.

8.2.3.2 Determination of analytical recovery using reference materials

Carry out the same test procedure prescribed for pure compounds in 8.2.3.1. Use a suitable mass of each of the selected reference materials (6.3), taking into consideration the concentration of the metal and metalloids of interest in the reference material and the supplier's instructions on the minimum amount of material that is required for an homogenous sample.

NOTE It is preferable to use the smallest mass of reference material that can be easily weighed, to scale up the volume of reagents and to adjust the final test solution volume so that the experiment is as representative as possible of the analysis of workplace air samples.

8.2.3.3 Determination of analytical recovery using reference air samples

Prepare and analyse test solutions from a minimum of six reference air samples (6.4) using the method described in the measuring procedure.

8.2.3.4 Comparison of results with the acceptance criteria

Calculate the mean analytical recovery and coefficient of variation for each of the tests performed and compare the results with the requirements of 5.2.2. If the requirements are not met for a test material, the analytical recovery test may be repeated using material with a smaller particle size and/or using a larger volume of reagents. If the requirements are still not met, the materials of a type similar to the test material concerned shall be excluded from the scope of the measuring procedure.

8.2.4 Measuring procedures that do not involve sample dissolution

8.2.4.1 Experimental determination of analytical recovery

8.2.4.1.1 Reference air samples

Analyse a minimum of six reference air samples (6.4) using the method described in the measuring procedure.