SLOVENSKI STANDARD
SIST EN 13890:2003

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

Workplace atmospheres - Procedures for measuring metals and metalloids in airborne paticles - Requirements and test methods

Arbeitsplatzatmosphäre - Verfahren zur quantitativen Bestimmung von Metallen und Metalloiden in Schwebstoffen - Anforderungen und Prüfverfahren

Atmosphères des lieux de travail - Procédures de mesurage des métaux et métalloides dans les particules en suspension dans l'air - Exigences et méthodes d'essai

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Workplace atmospheres - Procedures for measuring metals and metalloids in airborne paticles - Requirements and test methods

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

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This European Standard was approved by CEN on 9 September 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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Foreword

This document EN 13890:2002 has been prepared by Technical Committee CEN/TC 137 "Assessment of workplace exposure", 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of an identical text or by endorsement, at the latest by April 2003, and conflicting national standards shall be withdrawn at the latest by April 2003.

Annexes A, B, C and D are informative.

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

Introduction

EN 482 prescribes general requirements for the performance of procedures for measuring chemical agents in workplace atmospheres. These requirements include maximum values of overall uncertainty (a combination of precision and bias) achievable under prescribed laboratory conditions.

This European Standard provides a framework for assessing the performance of procedures for measuring metals and metalloids against the criteria 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.

1 Scope

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

This European Standard is not applicable to procedures for measuring metals or metalloids present in inorganic gases or vapours, e.g. mercury, arsine, etc (see EN 838 [1] and EN 1076 [2]), or to procedures for measuring metals and metalloids in compounds that could be present as a particle/vapour mixture, e.g. arsenic trioxide (see ENV 13936 [3]).

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.

This European Standard specifies a method for determining the bias and precision of the analytical method and combining this with the bias and precision of the sampling method to estimate the overall uncertainty of the measuring procedure as a whole.

If there is no procedure for measuring a particular metal or metalloid which meets the requirements of this European Standard, it is recommended to use a measuring procedure whose performance is nearest to the specified requirements.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

EN 482:1994*, Workplace atmospheres — General requirements for the performance of procedures for the measurement of chemical agents.*

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

EN 1540, *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 13205:2001, *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 European Standard, the following terms and definitions apply.

3.1 EN 1540 definitions

Averaging time, bias, chemical agent, limit value, measuring procedure, overall uncertainty, precision, specified measuring range, true value, validation:

Definitions for these terms are as in EN 1540.

3.2 Measurement terms

3.2.1
analysis
all operations carried out after sample preparation to determine the amount or concentration of the metals or metalloids of interest present in the sample

3.2.2
analytical method
all steps of the measuring procedure that describe the overall process of sample preparation and analysis

3.2.3
sampling method
all steps of the measuring procedure that describe the process of collecting an air sample

3.2.4
sample preparation
all operations carried out on a sample, after transportation and storage, to prepare it for analysis, including transformation of the sample into a measurable state, where necessary

3.2.5
sample dissolution
process of obtaining a solution containing the analytes of interest from a sample.

NOTE This can or cannot involve complete dissolution of the sample.

3.2.6
reference sample
sample having a known or measured content or loading of the metals and metalloids of interest, which is analysed to determine the analytical bias or the analytical precision of a measuring procedure

3.2.7
test solution
solution prepared by the process of sample dissolution and subjected to any further operations required to bring it into a state in which it is ready for analysis

3.3 Sampling terms

3.3.1
collection substrate
medium on which airborne particles are collected, e.g. a filter or a polyurethane foam

3.3.2
inhalable sampler
sampler which collects the inhalable fraction of airborne particles

3.3.3
particle/vapour mixture
aerosol consisting of airborne particles and vapour

[ENV 13936 [3]]

3.3.4
respirable sampler
sampler which collects the respirable fraction of airborne particles

3.3.5
sample
collection substrate and the airborne particles collected on it, including, if appropriate, airborne particles collected on the internal surfaces of the sampler

3.3.6
sampler
device for collecting airborne particles

3.4 Statistical terms

3.4.1
analytical bias
bias of the analytical method

3.4.2
analytical precision
imprecision arising from analytical variability

3.4.3
analytical recovery
ratio of the mass of analyte measured in a sample to the known mass of analyte in that sample, expressed as a percentage

3.4.4
detection limit
lowest concentration of an analyte that the analytical process can reliably detect

3.4.5
quantification limit
lowest concentration of an analyte that the analytical process can report with a specified degree of certainty

3.4.6
sampler bias
bias of the sampling method

3.4.7
sampling precision
combination of the imprecision of the aerosol sampling process and the imprecision arising from the flow rate variability

4 Principle

Sampler bias and sampling precision are estimated by examining sampler performance data reported in the scientific literature.

For measuring procedures that specify a method for the determination of soluble metals and metalloids, the analytical bias is, by definition, taken to be zero.

For measuring procedures that specify a method for the determination of total metals and metalloids that involves sample dissolution, the analytical bias is determined by assessing the effectiveness of the sample dissolution method described in the procedure. This is achieved by analysing reference samples, which can be pure compounds, bulk reference materials and/or samples of dust on collection substrates, and determining the analytical recovery of the metals and metalloids of interest. The analytical recovery is then carefully scrutinised to determine a typical upper limit for the analytical bias.

For measuring procedures that do not involve sample dissolution, e.g. X-ray fluorescence spectrometry, the analytical bias is determined by comparison with a reference method or by analysis of a reference material. Alternatively, in some instances, it can be estimated theoretically.

For measuring procedures that involve sample dissolution, the analytical precision is determined by analysing reference samples prepared by spiking collection substrates with standard solutions of the metals and metalloids of interest. The repeatability of measurements made on collection substrates spiked with various sample loadings (corresponding to different concentrations of metal or metalloid in air and air sample volumes) gives a measure of the precision of the analytical method for different averaging times across the measuring ranges specified in EN 482.

For measuring procedures that do not involve sample dissolution, the analytical precision is determined in a similar manner, by analysing reference samples prepared by loading collection substrates with known amounts of dust. Alternatively, in some instances, it can be estimated theoretically.

The determined analytical bias and analytical precision are combined with the estimated sampler bias and sampling precision to estimate the overall uncertainty of the measuring procedure as a whole. This is then assessed against the general performance requirements prescribed in EN 482.

5 Requirements

5.1 General requirements

5.1.1 Scope of the measuring procedure

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

— the metals and metalloids that are covered by the measuring procedure, and the analytical techniques used;

— the working range;

— metal and metalloid compounds and/or dusts that could be found in workplace air, for which the sample preparation method described is known to be inadequate;

— any known interferences.

5.1.2 Method performance

The measuring procedure shall give information about method performance, including the following:

— metal and metalloid compounds and/or dusts for which the sample preparation method described has been shown to be effective;

— the range of concentrations of metals and metalloids in air and sample volumes over which the measurement method has been shown to meet the acceptance criteria for overall uncertainty prescribed in EN 482;

— the detection limits and quantification limits of the analytical method for the metals and metalloids of interest;

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

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 exposure limits for the metals and metalloids of interest (for example, an inhalable sampler or a respirable sampler can be used);

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

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 Quantification limit

When tested in accordance with 9.1, the quantification limit shall be lower than the mass of metal or metalloid that would be sampled at 0,1 times the limit value for the minimum air sample volume specified in the measuring procedure.

5.1.7 Other detailed requirements

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

5.2 Analytical recovery

When tested in accordance with 9.3.2 the upper limit of the 95 % confidence interval of the mean analytical recovery shall be at least 90 % for all test materials.

5.3 Overall uncertainty

The overall uncertainty shall conform with the requirements specified in Table 1 of EN 482:1994.

6 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 and resistivity greater than 0,01 MΩ·m at 25 °C).

NOTE 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).