Workplace air — Determination of hexavalent chromium in airborne particulate matter — Method by ion chromatography and spectrophotometric measurement using diphenyl carbazide

Air des lieux de travail — Détermination du chrome hexavalent dans les particules en suspension dans l'air — Méthode par chromatographie ionique et détection spectrophotométrique avec diphényl carbazide
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 16740 was prepared by Technical Committee ISO/TC 146, Air quality, Subcommittee SC 2, Workplace atmospheres.
Introduction

The health of workers in many industries is at risk through exposure by inhalation of hexavalent chromium compounds. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure, and this is generally achieved by making workplace air measurements. This International Standard has been published in order to make available a method for making valid exposure measurements for hexavalent chromium compounds used in industry. It will be of benefit to agencies concerned with health and safety at work; industrial hygienists and other public health professionals; analytical laboratories; industrial users of hexavalent chromium compounds and their workers.

It has been assumed in the drafting of this International Standard that the execution of its provisions, and the interpretation of the results obtained, is entrusted to appropriately qualified and experienced people.
Workplace air — Determination of hexavalent chromium in airborne particulate matter — Method by ion chromatography and spectrophotometric measurement using diphenyl carbazide

1 Scope

This International Standard specifies a method for the determination of the time-weighted average mass concentration of hexavalent chromium in workplace air.

Separate sample preparation methods are specified for the extraction of soluble and insoluble hexavalent chromium. The method for insoluble hexavalent chromium can also be used to prepare samples for determination of total hexavalent chromium, if desired.

This International Standard is applicable to the personal sampling of the inhalable fraction of airborne particles, as defined in ISO 7708, and to static (area) sampling. The analytical method is applicable to the determination of masses of 0,01 µg to 10 µg of hexavalent chromium per sample, without dilution.

The concentration range of hexavalent chromium in air for which the measuring procedure is applicable is determined by the sampling method selected by the user (see 10.1). For a 1 m³ air sample, without sample dilution, the working range is approximately 0,01 µg m⁻³ to 10 µg m⁻³.

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.

ISO 648, Laboratory glassware — One-mark pipettes
ISO 1042, Laboratory glassware — One-mark volumetric flasks
ISO 3585, Borosilicate glass 3.3 — Properties
ISO 3696, Water for analytical laboratory use — Specification and test methods
ISO 7708:1995, Air quality — Particle size fraction definitions for health-related sampling
ISO 8655-1, Piston-operated volumetric apparatus — Part 1: Terminology, general requirements and user recommendations
ISO 8655-2, Piston-operated volumetric apparatus — Part 2: Piston pipettes
ISO 8655-5, Piston-operated volumetric apparatus — Part 5: Dispensers
ISO 8655-6, Piston-operated volumetric apparatus — Part 6: Gravimetric methods for the determination of measurement error
EN 13205, Workplace atmospheres — Assessment of performance of instruments for measurement of airborne particle concentrations
3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 General definitions

3.1.1 chemical agent
any chemical element or compound, on its own or admixed, as it occurs in the natural state or as produced, used or released, including release as waste by any work activity, whether or not produced intentionally and whether or not placed on the market

[EN 1540 [1]]

3.1.2 breathing zone
(space around the worker's face from where he or she takes his or her breath

3.1.3 breathing zone
(hemisphere (generally accepted to be 0.3 m in radius) extending in front of the human face, centred on the midpoint of a line joining the ears; the base of the hemisphere is a plane through this line, the top of the head and the larynx

NOTE 1 This definition is not applicable when respiratory protective equipment is used.

NOTE 2 Adapted from EN 1540 [1].

3.1.4 exposure
(by inhalation) situation in which a chemical agent is present in air which is inhaled by a person

3.1.5 measuring procedure
procedure for sampling and analysing one or more chemical agents in the air and including storage and transportation of the sample

3.1.6 operating time
period during which a sampling pump can be operated at specified flow rate and back pressure without recharging or replacing the battery

[EN 1232 [2]]

3.1.7 time-weighted average concentration
TWA concentration
concentration of a chemical agent in the atmosphere, averaged over the reference period

NOTE A more detailed discussion of TWA concentrations is available in Reference [3].

3.1.8 limit value
reference figure for concentration of a chemical agent in air

NOTE An example is the Threshold Limit Value® (TLV) for a given substance in workplace air, as established by the American Conference of Government Industrial Hygienists (ACGIH) [3].
3.1.9
reference period
specified period of time stated for the limit value of a specific chemical agent

NOTE Examples of limit values for different reference periods are short-term and long-term exposure limits, such as those established by the ACGIH [3].

3.1.10
workplace
defined area(s) in which the work activities are carried out

[EN 1540 [1]]

3.2 Particle size fraction definitions

3.2.1
inhalable convention
target specification for sampling instruments when the inhalable fraction is of interest

[ISO 7708]

3.2.2
inhalable fraction
mass fraction of total airborne particles which is inhaled through the nose and mouth

NOTE The inhalable fraction depends on the speed and direction of air movement, on breathing rate and other factors.

[ISO 7708]

3.2.3
respirable convention
target specification for sampling instruments when the respirable fraction is of interest

[ISO 7708]

3.2.4
respirable fraction
mass fraction of inhaled particles penetrating to the unciliated airways

[ISO 7708]

3.2.5
total airborne particles
all particles surrounded by air in a given volume of air

NOTE Because all measuring instruments are size-selective to some extent, it is often impossible to measure the concentration of total airborne particles.

[ISO 7708]

3.3 Sampling definitions

3.3.1
personal sampler
device attached to a person that samples air in the breathing zone

[EN 1540 [1]]
3.3.2 personal sampling
process of sampling carried out using a personal sampler

[EN 1540 [1]]

3.3.3 sampler
device for collecting airborne particles

NOTE Instruments used to collect airborne particles are frequently referred to by a number of other terms, e.g. sampling heads, filter holders, filter cassettes, etc.

3.3.4 static sampling
area sampling
process of air sampling carried out in a particular location

3.3.5 static sampler
area sampler
device, not attached to a person, used in static sampling

3.4 Analytical definitions

3.4.1 blank solution
solution prepared by taking a reagent blank, laboratory blank or field blank through the same procedure used for sample dissolution

3.4.2 calibration blank solution
calibration solution prepared without the addition of any working standard solution

NOTE The concentration of the analyte(s) of interest in the calibration blank solution is taken to be zero.

3.4.3 calibration solution
solution, prepared by dilution of the working standard solution, containing the analyte(s) of interest at a concentration that is suitable for use in calibration of the analytical instrument

3.4.4 field blank
filter that is taken through the same handling procedure as a sample, except that it is not used for sampling, but is loaded into a sampler, transported to the sampling site and then returned to the laboratory for analysis

3.4.5 laboratory blank
unused filter, taken from the same batch used for sampling, that does not leave the laboratory

3.4.6 linear dynamic range
range of concentrations over which the calibration curve for an analyte is linear

NOTE The linear dynamic range extends from the detection limit to the onset of calibration curvature.
3.4.7 reagent blank
all reagents used in sample dissolution, in the same quantities used for preparation of laboratory blank, field blank and sample solutions

3.4.8 sample dissolution
process of obtaining a solution containing all analytes on interest from a sample, which might or might not involve complete dissolution of the sample

3.4.9 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, if necessary

3.4.10 sample solution
solution prepared from a sample by the process of sample dissolution

NOTE A sample solution might need to be subjected to further operations, e.g. dilution, in order to produce a test solution that is ready for analysis.

3.4.11 test solution
blank solution or sample solution that has been subjected to all operations required to bring it into a state in which it is ready for analysis, e.g. dilution

NOTE The blank test solution is the blank solution and the sample test solution is the sample solution, if these solutions are not subjected to any further operations before analysis.

3.4.12 working standard solution
solution, prepared by dilution of the stock standard solution, that contains the analyte(s) of interest at a concentration(s) that is better suited to preparation of calibration solutions than the concentration(s) of the analyte(s) in the stock standard solution(s)

3.5 Statistical terms

3.5.1 analytical recovery
ratio of the mass of analyte measured in a sample when it is analysed to the known mass of analyte in that sample, expressed as a percentage

3.5.2 bias
consistent deviation of the results of a measurement process from the true value of the air quality characteristic itself

[ISO 6879 [4]]

3.5.3 overall uncertainty
(of a measuring procedure or of an instrument) quantity used to characterise as a whole the uncertainty of a result given by an apparatus or measuring procedure

NOTE Uncertainty is expressed, as a percentage, by a combination of bias and precision, usually according to the formula:

$$\frac{|x - x_{ref}| + 2s}{x_{ref}} \times 100$$
where

\[ \bar{x} \] is the mean value of results of a number \( (n) \) of repeated measurements;

\[ x_{\text{ref}} \] is the true or accepted reference value of concentration;

\[ s \] is the standard deviation of the measurements.

[EN 482 [5]]

3.5.4 precision

closeness of agreement of results obtained by applying the method several times under prescribed conditions

[ISO 6879 [4]]

3.5.5 true value

〈of a quantity〉 value which characterises a quantity perfectly defined in the conditions which exist when that quantity is considered

NOTE The true value of a quantity is a theoretical concept and, in general, cannot be known exactly.

[ISO 3534-1 [6]]

3.5.6 uncertainty of measurement

parameter associated with the result of a measurement that characterises the dispersion of the values that could reasonably be attributed to the measurand

NOTE 1 The parameter might be, for example, a standard deviation (or a given multiple of it), or the width of a confidence interval.

NOTE 2 Uncertainty of measurement comprises, in general, many components. Some of these components can be evaluated from the statistical distribution of the results of series of measurements and can be characterised by standard deviations. The other components, which also can be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information. The Guide to the Expression of Uncertainty in Measurement (GUM)[7] refers to these different cases as Type A and Type B evaluations of uncertainty, respectively.

NOTE 3 Adapted from the International Vocabulary of Basic and General Terms in Metrology (VIM) [8].

4 Principle

A known volume of air is drawn through a filter to collect airborne hexavalent chromium, using a sampler designed to collect the inhalable fraction of airborne particles (see 8.1.1).

The filter and collected sample are then treated to dissolve hexavalent chromium. Separate sample preparation methods are described for extraction of soluble and insoluble hexavalent chromium compounds, and either or both of these methods are used, as required, to prepare the sample for analysis. Soluble hexavalent chromium compounds are extracted with water or with ammonium sulfate/ammonium hydroxide solution, without heating. Insoluble hexavalent chromium compounds are extracted with sodium hydroxide \((\rho = 20 \text{ g}\cdot\text{l}^{-1})\)/sodium carbonate solution \((\rho = 30 \text{ g}\cdot\text{l}^{-1})\), either by heating on a hotplate or by ultrasonic extraction.

Aliquots of the sample solutions are analysed by ion chromatography in order to separate the extracted hexavalent chromium from trivalent chromium and other metal cations. Hexavalent chromium is measured spectrophotometrically at 540 nm after post-column derivatization with 1,5-diphenylcarbazide in acid solution.