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

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*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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Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

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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.

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

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

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## 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.

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# 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 m<sup>3</sup> air sample, without sample dilution, the working range is approximately 0,01 µg·m<sup>-3</sup> to 10 µg·m<sup>-3</sup>.

## 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 <sup>[1]</sup>]

##### 3.1.2

##### **breathing zone**

(general definition) space around the worker's face from where he or she takes his or her breath

##### 3.1.3

##### **breathing zone**

(technical definition) 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 <sup>[1]</sup>.

##### 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 <sup>[2]</sup>]

##### 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<sup>®</sup> (TLV) for a given substance in workplace air, as established by the American Conference of Government Industrial Hygienists (ACGIH) <sup>[3]</sup>.



**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 <sup>[1]</sup>]

**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

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**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 of 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:

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

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 <sup>[5]</sup>]

### 3.5.4 precision

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

[ISO 6879 <sup>[4]</sup>]

### 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 <sup>[6]</sup>]

### 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)<sup>[7]</sup> 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) <sup>[8]</sup>.

## 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.

Analytical results are obtained from plots of the measured absorbance as a function of concentration of hexavalent chromium. They can be used for assessment of occupational exposure to hexavalent chromium in air.

If desired, total hexavalent chromium can be measured directly using the sodium hydroxide/sodium carbonate extraction solution.

## 5 Reactions

Post-column derivatization involves reaction of hexavalent chromium with 1,5-diphenylcarbazide to produce trivalent chromium and diphenylcarbazone. These then combine to form a trivalent chromium-diphenylcarbazone complex containing the characteristic magenta chromagen ( $\lambda_{\text{max}} = 540 \text{ nm}$ ). However, the exact mechanism of this reaction is not fully understood.

## 6 Requirement

The measurement procedure shall comply with any relevant international, European or national standard which specifies performance requirements for procedures for measuring chemical agents in workplace air (e.g. EN 482 [5]).

## 7 Reagents

During the analysis, use only reagents of recognised analytical grade, and only water as specified in 7.1.

**7.1 Water**, complying with the requirements for ISO 3696 grade 2 water (electrical conductivity less than  $0,1 \text{ mS}\cdot\text{m}^{-1}$  and resistivity greater than  $0,01 \text{ M}\Omega\cdot\text{m}$  at  $25^\circ\text{C}$ ).

It is recommended that the water used be obtained from a water purification system that delivers ultrapure water having a resistivity greater than  $0,18 \text{ M}\Omega\cdot\text{m}$  (usually expressed by manufacturers of water purification systems as  $18 \text{ M}\Omega\cdot\text{cm}$ ).

**7.2 Sulfuric acid** ( $\text{H}_2\text{SO}_4$ ), concentrated,  $\rho \approx 1,84 \text{ g}\cdot\text{ml}^{-1}$ , mass fraction  $\approx 98 \%$ .

**WARNING** — Concentrated sulfuric acid is corrosive and causes burns. Avoid exposure by contact with the skin or eyes. Use suitable personal protective equipment (including suitable gloves, face shield or safety spectacles, etc.) when working with concentrated or dilute sulfuric acid. Fumes produced when concentrated sulfuric acid is heated are irritant. Therefore, carry out procedures with sulfuric acid in a fume hood. Exercise great caution when diluting sulfuric acid with water, since this process is very exothermic. Do not add water to sulfuric acid, since it reacts violently when mixed in this manner. Prepare sulfuric acid/water mixtures by adding sulfuric acid to water.

**7.3 Nitric acid** ( $\text{HNO}_3$ ), concentrated,  $\rho \approx 1,42 \text{ g}\cdot\text{ml}^{-1}$ , mass fraction  $\approx 70 \%$ .

**WARNING** — Concentrated nitric acid is corrosive and oxidizing, and nitric acid fumes are irritant. Avoid exposure by contact with the skin or eyes, or by inhalation of fumes. Use suitable personal protective equipment (including suitable gloves, face shield or safety spectacles, etc.) when working with the concentrated or dilute nitric acid, and carry out procedures with concentrated nitric acid in open vessels in a fume hood.

**7.4 Nitric acid**, diluted 1 to 10.

Carefully and slowly add 50 ml of concentrated nitric acid (7.3) to 450 ml of water (7.1) in a 1-litre polypropylene bottle (8.2.3.1). Add the acid in small aliquots. Between additions, swirl to mix and run cold tap water over the side of the bottle to cool the contents. Do not allow tap water to contaminate the contents of the