



FINAL DRAFT

International Standard

ISO/FDIS 6323-1

Workplace air — Determination of arsenic and arsenic compounds by electrothermal atomic absorption spectrometry —

Part 1: Arsenic and arsenic compounds, except arsine by ET-AAS

Air des lieux de travail — Détermination de l'arsenic et des composés d'arsenic par spectrométrie d'absorption atomique avec atomisation électrothermique —

Partie 1: Arsenic et composés d'arsenic, à l'exception de l'arsine par ET-AAS

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

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Introduction

Arsenic and arsenic compounds are toxic and are recognized as human carcinogens. In particular arsenic and arsenic compounds are a hazard to the health of workers in many industries through exposure by inhalation. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure. The collection of samples of air during a work activity and then measuring the amount of particular arsenic and arsenic compounds are often done to assess an individual's exposure, the effectiveness of workplace controls or respiratory protection measures. The air sampling can be done as stationary or personal air sampling. Electrothermal atomic absorption spectrometry (ET-AAS) analysis of particular arsenic and arsenic compounds in a sample of respirable or inhalable dust collected on a collection substrate (membrane filter) is employed in many countries to measure and estimate exposure to arsenic and arsenic compounds. ET-AAS is able to quantify arsenic and arsenic compounds.

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Workplace air — Determination of arsenic and arsenic compounds by electrothermal atomic absorption spectrometry —

Part 1:

Arsenic and arsenic compounds, except arsine by ET-AAS

1 Scope

This document specifies a method for the determination of the mass concentration of particulate arsenic and arsenic compounds in workplace air sampled on a filter (e. g. 37 mm cellulose nitrate filter), digested with acid or an acid mixture and analysed quantitatively by using electrothermal atomic absorption spectrometry (ET-AAS). The method is not suitable for determination of arsenic in the form of metal arsenides, which decompose in the presence of water or acid, or for arsenic trioxide vapour.

Many different types of sampling apparatus are used to collect respirable or inhalable dust, according to the occupational hygiene convention. This document is designed to accommodate the variety of samplers and collection substrates available to analysts. This document is intended to be used in conjunction with ISO 21832 which promotes best practices for these analyses.

The method is applicable to the determination of masses of approximately 0,2 µg to 2 µg of arsenic per sample, for analysis of test solutions prepared using sample solution aliquots in the recommended range (see [10.1.3](#) and [10.1.4.1](#)). The concentration range for arsenic in air, for which this procedure is applicable, is determined in part by the sampling procedure selected by the user.

The method is applicable to personal and stationary air sampling.

A number of transition metals can interfere with the determination of arsenic by electrothermal atomic absorption spectrometry (see [11.3](#)).

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 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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*

DIN 12353, *Laboratory ware made from fused quartz and fused silica; boiling flasks made from fused quartz; round bottom flasks, flat bottom flasks and conical flasks*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158 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/>

4 Principle

A filter (6.2.1; see Table 1) is mounted in a sampler (6.1.1) designed to collect the respirable or inhalable fraction of airborne particles. The sampling can be performed with personal or stationary fixed samplers. Before sampling is performed, the filter batch used shall be verified with regard to its metal content and consequently the suitability of the minimum requirements for the performance of measuring methods.

Currently limit values in different countries exist for arsenic and its compounds either as “total dust”, defined by the performance of a sampler, or in the inhalable size selective fraction. A suitable sampling device for the applicable particle fraction shall be used considering the existing limit value/particle fraction.

The fraction separated on the filter is analysed for arsenic after acid digestion using ET-AAS. As digestion media, nitric acid or a mixture of nitric and hydrochloric acid can be used. The sample solution is allowed to cool and diluted to a given volume with ultrapure water (7.3.1), depending on the digestion type used. A test solution is prepared by transferring an aliquot of the sample solution to a volumetric flask and dilution to volume with ultrapure water.

The atomic absorption spectrometer is equipped with an arsenic hollow cathode lamp or electrodeless charge lamp and heated electrically.

Absorbance measurements are made at 193,7 nm or 197,2 nm, using a graphite furnace with platform and a matrix modifier (7.4.1.2). For background compensation, Zeeman-Mode is used. Deuterium background compensation can also be used as an option. Results obtained by the analytical-curve technique or the analyte addition technique.

5 Requirement

The measuring procedure shall conform to ISO 20581 or ISO 21832, which specify performance requirements for procedures for measuring chemical agents in workplace air.

6 Apparatus and equipment

6.1 Sampling equipment

6.1.1 Sampler

The performance of the sampler used shall match the criteria for respirable or inhalable dust as specified in ISO 7708. Samplers that use 37 mm diameter filters (6.2.1) as the collection substrate are required. A plastic filter capsule for filters with a diameter of 37 mm are necessary. A suitable supporting grid can be necessary.

Each sampler should be labelled with a unique number, in order to identify samplers that start to underperform after long-term use.

Samplers shall meet the manufacturer's requirements for calibration.

NOTE For person-related or stationary sampling, filters with diameters of e.g. 70 mm up to 150 mm, can also be used with specific sampling systems, with appropriate adjustments to the digestion conditions.

NOTE In some countries, there can be exceptions due to national requirements.

6.1.2 Filter capsule

Matching plastic filter capsule with covers for the 37 mm filter ([6.2.1](#)), for insertion into the sampler.

6.1.3 Sampling pumps

Sampling pumps shall conform to the requirements of ISO 13137.

If the sampling pump is used outside the range of conditions specified in ISO 13137, appropriate actions should be taken to ensure that the performance requirements are met.

6.1.4 Portable flowmeter

The flowmeter shall conform to the requirements of ISO 13137.

The flowmeter shall be capable of measuring the appropriate flow rate (see [9.3.1](#) and [9.4](#)) to within $\pm 5\%$, and calibrated against a primary standard, i.e. a flowmeter of which the accuracy is traceable to national standards. If appropriate, the atmospheric temperature and pressure at which the flowmeter was calibrated should be recorded.

6.1.5 Silicone adapter

The silicone adapter shall fit into the sampler head to connect the flowmeter ([6.1.4](#)) for measuring/setting the air flow.

6.1.6 Ancillary equipment

The following ancillary equipment shall be used:

- a) flexible tubing, to connect the sampler to the sampling pump ([6.1.3](#));
- b) belts or harnesses to which the sampling pumps can conveniently be fixed for personal air sampling; a tripod is required for person-related or stationary sampling;
- c) a means to transport the samples from the workplace to the laboratory, which minimises the possibility of accidental transfers of collected dust to or from the collection substrate (filter); transportation requires caps or covers for the samplers (filter capsule);
- d) a thermometer (readable to 1 °C) and a barometer (readable to 0,1 kPa), to measure atmospheric temperature and pressure for flow rate correction, when the temperature and pressure at the time of use differ from the conditions under which the flowmeter ([6.1.4](#)) was calibrated.

6.2 Collection media

6.2.1 Filters

Filters shall be of a diameter suitable for use in the selected sampler ([6.1.1](#)) and have a capture efficiency for respirable or inhalable particles of not less than 99 %. It is important for the analyst to know the composition of the collection substrate used to collect the sample since it has a direct bearing on the analytical approach used. The collection substrates generally used for the sampling of arsenic and arsenic compounds, and their characteristics, are listed in [Table 1](#).

Table 1 — Dust collection substrates

Sampling medium (pore size)	Comments
Cellulose nitrate/cellulose acetate membrane filters (8 µm)	Membrane filters are very suitable for subsequent analysis of metals in dust, as they exhibit very low blank values and can be readily digested by acids.
Mixed esters of cellulose (MCE) (0,8 µm)	MCE filters are comprised mixtures of cellulose acetate and cellulose nitrate. They are low in metal background and completely dissolvable with acids.
Quartz glass fibre filters	They are suitable for dust sampling due to their good retention capacity. As a result of their exceedingly low and relatively constant blank values, they are substantially more suitable than glass fibre filters for the screening of dusts for metallic components.
Glass fibre filters	The high and often fluctuating blank values of this filter material can adversely affect the analysis of metals in dust.
Fluoropolymer filters (PTFE)	They exhibit high chemical and thermal durability and are not dissolved by common digestion agents. Their blank value concentrations are low, however, this filter material must be tested before use, as it can contain small amounts of metals. Furthermore, the high flow resistance of this filter material must be taken into consideration.
Other filter materials Polycarbonate or polyvinyl chloride filters (PVC)	Other filter materials can also be used such as polycarbonate or polyvinyl chloride (PVC) filters. Polycarbonate filters, in particular, have a very good resistance to chemicals.
Polyurethane foams	Polyurethane foams (PU foams) can be manufactured with various pore sizes and are suitable for dividing collections of particles into fractions. Depending on the manufacturing process, PU foams can contain many interfering impurities due to auxiliary components (e.g. organic tin compounds), pre-cleaning of the material is crucial to minimise blank values. The limited solubility of the material during digestion can also lead to interference.
Disposable inhalable sampler (DIS) with filter or foam and filter	DIS is a single-use sampler for the inhalable fraction and includes an MCE filter bonded to a cellulose capsule. If the capsule is fitted with a foam pre-selector, the analysis of the capsule corresponds to the respirable fraction. The inhalable fraction is the sum of the pre-selector foam and the capsule bonded with MCE filter. Effects of wall deposits can be eliminated with this sampler type.

Cellulose membrane filters are rigid and easy to handle when weighing and loading the sampler. Glass fibre filters, quartz glass fibre filters, PVC and especially polycarbonate filters are flexible and require careful handling.

NOTE Cellulose membrane filters completely dissolve when digested with nitric and hydrochloric acid, or only nitric acid. Glass or quartz fibre filters, PVC or polycarbonate filters do not completely dissolve in digestion media hydrochloric and or nitric acid. It can be necessary to filtrate the digestion solution before analysis.

6.2.2 Recommendations for filters

An important property of an analytical filter (6.2.1) is that it should contain no or only low concentrations of the metals to be analysed. A constantly stable concentration of metal content of the filter is also easy to handle. Filter materials listed in Table 1 generally do not contain compounds that interfere with the measurement of arsenic and arsenic compounds. Impurities can be introduced during the filter manufacturing process and blank values can increase depending on filter material. Therefore, batches of filters should be regularly tested to detect potential interferences and background levels.

It is advisable to use filters that exhibit no blank values or blank values that are as low as possible and constant. Experience has shown that blank value concentrations are batch-dependent, therefore a test

certificate giving information on relevant components of the filters should be available (e.g. from the manufacturer or supplier). The test certificate should contain information on the level of the metal content and their bandwidth. Only filters from a single batch should be used in the course of a measurement series. Cellulose nitrate membrane filters exhibit the least variability and lowest background levels and thus are useful in situations where low limits of detection are required.

6.2.3 Back pressure of filters

Some filters (6.2.1) have a high back pressure and thereby negatively influence the sampling, due to a higher load on the pump. High values for back pressure can compromise the sampling time, when the sampling of a complete 8 h shift is desired.

6.2.4 Weighing of filters

Weighing can be performed to determine the inhalable or respirable fraction and should be performed following ISO 15767^[1] (see 9.2.4). Filters (6.2.1) shall not be weighed in cassettes as large weight variations have been reported.^[2] Reference shall be made to the instructions of the collection substrate manufacturer.

6.3 Equipment for the determination of dust concentration

If the determination of the inhalable or respirable fraction should be performed, a microbalance capable of weighing $\pm 1 \mu\text{g}$ or better over the range 0 g to 5 g is required. An electrostatic eliminator is needed when weighing collection substrates. Weighing should be performed according to ISO 15767^[1].

6.4 Equipment for sample digestion

6.4.1 General

Equipment and aids for the digestion of metals and metal compounds in dust samples are listed in 6.4.2 to 6.4.4. All equipment and materials used should be as metal-free as possible. Inert materials should be used for digestion. Contamination with metals by equipment or materials used shall be avoided. If necessary, the equipment should be suitable cleaned before use.

6.4.2 Equipment for all types of sample digestion

6.4.2.1 **Ceramic tweezer**, for the transfer of the filters (6.2.1) into the digestion vessels.

6.4.2.2 **Measuring cylinder of perfluoralkoxy-alkane (PFA) copolymer** with volumes of 50 ml, 100 ml and 500 ml.

6.4.2.3 **Quartz glass bottle with a polytetrafluorethylene (PTFE) dispenser** for the transfer of the digestion acid or acid mixture into the digestion vessels.

6.4.2.4 **Bottle of PFA with PTFE dispenser**, for adding ultrapure water (7.3.1) to the digested samples.

6.4.3 Laboratory equipment for open vessel hot block digestion

6.4.3.1 **Heating block** made of metal or graphite with time/temperature control.

6.4.3.2 **Graduated digestion vessels**, preferably made of quartz glass or of comparable quality in accordance with the requirements of DIN 12353 for reaction vessels.

Vessels made of borosilicate glass should not be used due to possible interference from boron.