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## Workplace atmospheres — Determination of inorganic acids by ion chromatography —

Part 2:

### Volatile acids, except hydrofluoric acid (hydrochloric acid, hydrobromic acid and nitric acid)

*Air des lieux de travail — Détermination des acides inorganiques par  
chromatographie ionique —*

*Partie 2: Acides volatils, sauf acide fluorhydrique (acide  
chlorhydrique, acide bromhydrique et acide nitrique)*

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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.

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](http://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*.

This second edition cancels and replaces the first edition (ISO 21438-2:2009), which has been technically revised.

The main changes are as follows:

- terms, definitions and references have been updated;
- information on sample preparation and analytical methodology has been updated.

A list of all parts in the ISO 21438 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

The health of workers in many industries is at risk through exposure by inhalation of volatile inorganic acids. 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 document has been published in order to make available a method for making valid exposure measurements for volatile inorganic acids in use in the industry, such as hydrochloric acid, hydrobromic acid and nitric acid, but excluding hydrofluoric acid. This document is intended to be of benefit to:

- agencies concerned with health and safety at work;
- industrial hygienists and other public health professionals;
- analytical laboratories;
- industrial users of hydrochloric acid, hydrobromic acid and nitric acid and their workers, etc.

The execution of the provisions and the interpretation of the results obtained with the use of this document are entrusted to appropriately qualified and experienced people.

The procedure is intended to differentiate between the acids and their corresponding salts. If both are present in the air, particulate salts are trapped on a pre-filter. Co-sampled particulate matter either trapped on the pre-filter or deposited on the walls of the sampler, or both, can be analysed, if desired.

Acids can react with co-sampled particulate matter on the pre-filter, causing interference with the measurement of the acid concentration.

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# Workplace atmospheres — Determination of inorganic acids by ion chromatography —

## Part 2:

## Volatile acids, except hydrofluoric acid (hydrochloric acid, hydrobromic acid and nitric acid)

### 1 Scope

This document specifies a method for the determination of the time-weighted average mass concentration of hydrogen chloride (HCl) gas and hydrochloric acid mist, hydrogen bromide (HBr) vapour and hydrobromic acid mist and nitric acid (HNO<sub>3</sub>) vapour and mist in workplace air by collection on an alkali-impregnated quartz fibre filter and analysis by ion chromatography.

For mist sampling, this method is applicable to the personal sampling of the inhalable fraction of airborne particles as defined in ISO 7708 and to static (area) sampling.

### 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 3585, *Borosilicate glass 3.3 — Properties*

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

ISO 7708, *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: Pipettes*

ISO 8655-6, *Piston-operated volumetric apparatus — Part 6: Gravimetric reference measurement procedure for the determination of volume*

EN 13205, *Workplace exposure - Assessment of sampler performance for measurement of airborne particle concentrations*

ISO 18158, *Workplace air — Terminology*

ISO 20581, *Workplace air — General requirements for the performance of procedures for the measurement of chemical agents*

ISO 21832:2018, *Workplace air — Metals and metalloids in airborne particles — Requirements for evaluation of measuring procedures*

### 3 Terms and definitions

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

**3.1**  
**blank solution**  
solution prepared by taking a reagent blank, laboratory blank or field blank through the same procedure used for *sample dissolution* (3.5)

**3.2**  
**calibration blank solution**  
*calibration solution* (3.3) prepared without the addition of any *working standard solution* (3.9)

Note 1 to entry: The concentration of chloride, bromide and nitrate in the calibration blank solution is taken to be zero.

**3.3**  
**calibration solution**  
solution prepared by dilution of the *working standard solution* (3.9), containing chloride, bromide and nitrate at concentrations that are suitable for use in calibration of the analytical instrument

**3.4**  
**extraction solution**  
solvent or solution used to solubilise the analyte(s) of interest

**3.5**  
**sample dissolution**  
process of obtaining a solution containing chloride, bromide and nitrate from a sample, which does or does not involve complete dissolution of the sample

**3.6**  
**sample solution**  
solution prepared from a sample by the process of *sample dissolution* (3.5)

Note 1 to entry: A sample solution can require further operations, for example, dilution, in order to produce a *test solution* (3.8) that is ready for analysis.

**3.7**  
**stock standard solution**  
solution, used for preparation of the *calibration solutions* (3.3), containing chloride, bromide and/or nitric at a certified concentration that is traceable to national standards

**3.8**  
**test solution**  
*blank solution* (3.1) or *sample solution* (3.6) that has been subjected to all operations required to bring it into a state in which it is ready for analysis, e.g. dilution, filtered

Note 1 to entry: 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.

**3.9**  
**working standard solution**  
solution, prepared by dilution of the *stock standard solution(s)* (3.7), that contains chloride, bromide and nitrate at concentrations that are better suited to the preparation of *calibration solutions* (3.3) than the concentrations of chloride, bromide and nitric in the stock standard solution(s)



## 4 Principle

A known volume of air is drawn through a pre-filter and an alkali-impregnated quartz fibre sampling filter mounted in an inhalable sampler to collect HCl, HBr and HNO<sub>3</sub>. The acids are collected on the sampling filter, while particulate salts of the acids are trapped on the pre-filter.

The acids collected on the sampling filter are extracted with water or eluent (see [10.2.1](#)), without heating, to solubilize the analytes of interest.

Aliquots of the sample solution are subjected to ion chromatography in order to separate the extracted chloride, nitrate or bromide from other anions. Following this separation, the anions are measured using a conductivity or UV/visible detector.

Analytical results are obtained by plotting the measured conductivity as a function of concentration. They can be used for assessment of occupational exposure to HCl, HBr and HNO<sub>3</sub> in air.

## 5 Requirement

The measuring procedure shall comply with the performance requirements for procedures for measuring chemical agents in workplace air in ISO 20581.

## 6 Reagents

Use only reagents of recognized analytical grade and only water as specified in [6.1](#). It is advisable to check the blank values of all chemicals before use.

NOTE Chlorides and nitrates are found ubiquitously in the environment, and the presence of chloride and nitrate in reagents can lead to high blank values. It is therefore advisable to check the blank values of all chemicals before use.

**6.1 Water**, from a purification system that delivers ultrapure water of grade 1, in accordance with the requirements of ISO 3696.

NOTE State-of-art water purification systems deliver water of grade 1 with higher quality than specified in ISO 3696 (e.g. resistivity greater than 18 MΩ•cm).

**6.2 Sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>)**, anhydrous, mass fraction greater than 99,9 %.

**6.2.1 Sodium carbonate solution**, 1 mol/l, for impregnation of 37 mm diameter quartz fibre filters.

Dissolve 10,6 g of Na<sub>2</sub>CO<sub>3</sub> ([6.2](#)) in water. Quantitatively transfer the solution into a 100 ml one-mark volumetric flask, dilute to the mark with water, stopper and mix thoroughly.

**6.3 Sodium bicarbonate (NaHCO<sub>3</sub>)**, anhydrous, mass fraction greater than 99,9 %.

**6.4 Sodium carbonate/sodium bicarbonate extraction and eluent stock solution.**

Create a stock solution for the extraction and eluent solution. The stock solution shall be appropriate to the used application.

**6.4.1 Sodium carbonate/sodium bicarbonate extraction and eluent solution.**

The concentration of the extraction and eluent solution are typically matched, but this can be modified according to the laboratory procedure.

NOTE An alkaline solution (potassium hydroxide or sodium hydroxide) can be used in place of the buffer solution according to modifications to the method, but the resulting solution will require adjustment before analysis.

## 6.5 Chloride, bromide and nitrate standard solutions.

### 6.5.1 Chloride, bromide and nitrate stock solutions.

Use a commercial standard solution with a certificate of its concentration, e.g. 1 000 mg/l, traceable to national standards. Observe the manufacturer's expiry date or recommended shelf-life. If necessary, dilute the standard solution (e.g. from 1 000 mg/l to 100 mg/l) to prepare the stock solution.

### 6.5.2 Chloride, bromide and nitrate calibration solutions.

Pipette appropriate volumes of the stock solution to create the calibration. The calibration range shall be in accordance with the calculated working range in ISO 21832:2018, 5.2.1, and the limit of quantification (LOQ) in ISO 21832:2018, 8.1.2. A multi-point (the number of points to be consistent with the quality system of the laboratory) calibration shall be constructed within the linear range of the analytical method.

## 6.6 Chloride, bromide and nitrate check standard.

Use a certified reference material (CRM) as described in [10.5.3](#) with a certificate of its concentration, for example, 200 mg/l for chloride, 400 mg/l for bromide and 400 mg/l for nitrate, traceable to national standards. Observe the manufacturer's expiry date or recommended shelf-life.

### 6.6.1 Chloride, bromide and nitrate check-standard working solution.

The check-standard working solution should be in the middle of the calibration range. Pipette a proper volume of check-standard ([6.6](#)) to create a check-standard working solution with the eluent ([6.4](#)). Prepare this solution fresh for every calibration. If necessary, create a check-standard working solution for chloride, bromide and nitrate separately.

## 7 Apparatus

### 7.1 Sampling equipment

**7.1.1 Samplers**, designed to collect the inhalable fraction of airborne particles, complying with EN 13205, suitable for mounting a pre-filter and sampling filter separated by a spacer, manufactured from a material that does not react with acids.

NOTE 1 If samplers have an internal filter cassette, this too has to be manufactured from a material that does not react with acids.

NOTE 2 Materials which do not react with acids, from which samplers and internal filter cassettes can be manufactured, include polytetrafluoroethylene (PTFE) and other fluorinated polymers, polyvinyl chloride (PVC), polyethylene, polypropylene and polycarbonate.

NOTE 3 CEN/TR 15230<sup>[9]</sup> gives examples of inhalable samplers with the potential to meet the requirements of EN 13205 that were available on the market up to 2004, including a list of published reports on their performance.

**7.1.2 Filters**, of a diameter suitable for use with the samplers ([7.1.1](#)).

Hydrogen chloride (HCl) gas and hydrochloric acid mist, hydrogen bromide (HBr) vapour and hydrobromic acid mist and nitric acid (HNO<sub>3</sub>) vapour and mist are strong acids. Therefore, correct selection of the filter used for sample collection is of paramount importance. Also, some filters, for example, glass fibre filters, can contain metals, for example, barium, that react with acids to produce insoluble salts.

Chloride, bromide and nitrate are found ubiquitously in the environment and the presence of chloride, bromide and nitrate in filter materials can lead to high blank values. It is therefore essential to check the blank values of each batch of filters used.

**7.1.2.1 Pre-filters**, of a diameter suitable for use with the samplers (7.1.1), with a collection efficiency  $\geq 99\%$  for particles with a  $0,3\ \mu\text{m}$  diffusion diameter and manufactured from a material that does not react with HCl, HBr or  $\text{HNO}_3$ , for use as pre-filters to remove interfering particulate salts and also is compatible with the sample preparation and analysis method.

Refer to [Clause B.1](#) for guidance on suitable materials from which pre-filters can be manufactured.

**7.1.2.2 Sampling filters**, such as quartz fibre, impregnated with sodium carbonate solution, for use as sampling filters for volatile inorganic acids; e.g. 25 mm diameter filters impregnated with  $200\ \mu\text{l}$  of  $2,5\ \text{mol/l}$  sodium carbonate solution or 37 mm diameter filters impregnated with  $500\ \mu\text{l}$  of  $1\ \text{mol/l}$  sodium carbonate solution (6.2.1) (see References [10] and [11]).

Refer to [Clause B.2](#) for guidance on materials from which sampling filters can be manufactured.

**7.1.3 Spacers**, of a diameter suitable for use with the samplers (7.1.1), for separating the pre-filters (7.1.2.1) and sampling filters (7.1.2.2), manufactured from an inert material that does not react with the acids and on which the acids are not adsorbed, e.g. polypropylene sleeves or PTFE-coated screens.

**7.1.4 Sampling pumps**, according to ISO 13137<sup>[1]</sup> should be used.

If the sampling pump is used outside the range of conditions specified in ISO 13137<sup>[1]</sup> appropriate action should be taken to ensure that the performance requirements are met. For instance, at sub-zero temperatures it can be necessary to keep the pump warm.

**7.1.5 Flow meter**, portable, with an accuracy that is sufficient to enable the volumetric flow rate to be measured to within  $\pm 5\%$ .

The calibration of the flow meter shall be checked against a primary standard, i.e. a flow meter whose accuracy is traceable to national standards. If appropriate (see 9.1.3), record the atmospheric temperature and pressure at which the calibration of the flow meter was checked.

It is advisable that the flow meter used be capable of measuring the volumetric flow rate to within  $\pm 2\%$  or better.

#### 7.1.6 Ancillary equipment.

- Flexible tubing, for example, silicone of an outer diameter of 10 mm and an inner diameter of 6 mm for making a leakproof connection from the samplers to the sampling pumps without compressing as a result of the pressure difference between ambient atmosphere and the in-line vacuum at the used flow rate of  $2\ \text{l min}^{-1}$ .
- Belts or harnesses, which the sampling pumps can conveniently fix for personal sampling.
- Tweezers manufactured from or tipped with PTFE, for handling filters.
- Filter transport cassettes, or similar, if required (see 9.5.1), in which to transport samples to the laboratory.
- Thermometer, of range  $0\ \text{°C}$  to  $50\ \text{°C}$ , graduated in divisions of  $1\ \text{°C}$  or less, for measurement of atmospheric temperature. For applications at temperatures below freezing, the range of the thermometer shall extend to the appropriate desired range.
- Barometer, suitable for measurement of atmospheric pressure.

## 7.2 Laboratory apparatus

**CAUTION** — Chlorides, nitrates and, to a minor degree, bromides are found ubiquitously in the environment, and the presence of chlorides, in particular, can lead to elevated blanks. Check