

9a [g]YbYdfYa] b\ j]fcj '!Fc bUa YhcXUXc`c Yj Ub'U<7`!' "XY.'5 bU]nU
UWgcdWg_\ 'fUhrd]b]b]nfU i b`_cbWbhfUW^d`]bcj

Stationary source emissions - Manual method of determination of HCl - Part 3:
Absorption solutions analysis and calculation

Emissionen aus stationären Quellen - Manuelle Methode zur Bestimmung von HCl - Teil
3: Analyse der Absorptionslösungen und Berechnung der Ergebnisse

Emissions de sources fixes - Méthode manuelle de dosage du HCl - Partie 3: Analyse
des solutions d'absorption et calculs

[SIST EN 1911-3:1999](https://standards.iteh.ai/catalog/standards/sist/93be32e7-6c74-4686-ac1d-5562edad3a00/sist-en-1911-3-1999)

[https://standards.iteh.ai/catalog/standards/sist/93be32e7-6c74-4686-ac1d-](https://standards.iteh.ai/catalog/standards/sist/93be32e7-6c74-4686-ac1d-5562edad3a00/sist-en-1911-3-1999)

[5562edad3a00/sist-en-1911-3-1999](https://standards.iteh.ai/catalog/standards/sist/93be32e7-6c74-4686-ac1d-5562edad3a00/sist-en-1911-3-1999)

Ta slovenski standard je istoveten z: EN 1911-3:1998

ICS:

13.040.40 Stationary source emissions

SIST EN 1911-3:1999

en

iTeh STANDARD PREVIEW **(standards.iteh.ai)**

SIST EN 1911-3:1999

<https://standards.iteh.ai/catalog/standards/sist/93be32e7-6c74-4686-ac1d-5562edad3a00/sist-en-1911-3-1999>

ICS 13.040.40

Descriptors: air pollution, air, quality, gas analysis, emission, gaseous effluent, determination of content, hydrochloric acid, absorption, computation

English version

Stationary source emissions - Manual method of determination of HCl - Part 3: Absorption solutions analysis and calculation

Emissions de sources fixes - Méthode manuelle de dosage
du HCl - Partie 3: Analyse des solutions d'absorption et
calculs

Emissionen aus stationären Quellen - Manuelle Methode
zur Bestimmung von HCl - Teil 3: Analyse der
Absorptionslösungen und Berechnung der Ergebnisse

This European Standard was approved by CEN on 23 March 1998.

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 Central Secretariat 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 Central Secretariat 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, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

 **SIST EN 1911-3:1998**

<https://standards.iteh.ai/catalog/standards/sist/93be32c7-6c74-4686-ac1d-5562edad3a00/sist-en-1911-3-1998>

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Content	Page
Foreword	3
1 Scope	4
2 Normative references	4
3 Analysis	4
4 Expression and validation of results	10
5 Performance characteristics of the whole measurement method	11
6 Test report	12
Annex A (informative) Comparison between mercuric thiocyanate spectrophotometry and ion exchange chromatography method (methods B and C)	14
Annex B (informative) Effect of volatile chlorides	15
Annex C (informative) Internal uncertainty of the method	16

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN 1911-3:1999

<https://standards.iteh.ai/catalog/standards/sist/93be32e7-6c74-4686-ac1d-5562edad3a00/sist-en-1911-3-1999>



Foreword

This European Standard has been prepared by Technical Committee CEN/TC 264 "Air quality", 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 month of October 1998, and conflicting national standards shall be withdrawn at the latest by month of October 1998.

The determination of gaseous hydrogen chloride emissions¹⁾ from stationary sources by a manual method is divided in three parts described in the following standards :

- | | |
|-----------|---|
| EN 1911-1 | Stationary source emissions - Manual method of determination of HCl - Part 1 : Sampling of gases |
| EN 1911-2 | Stationary source emissions - Manual method of determination of HCl - Part 2 : Gaseous compounds absorption |
| EN 1911-3 | Stationary source emissions - Manual method of determination of HCl - Part 3 : Absorption solutions analysis and calculations |

This European standard is an integral part of complete measurement procedure and the use of the two other parts is necessary for determination of hydrogen chloride.

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, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN 1911-3:1999
<https://standards.iteh.ai/catalog/standards/sist/93be32e7-6c74-4686-ac1d-5562edad3a00/sist-en-1911-3-1999>

¹⁾ In this standard no distinction is made between hydrogen chloride and hydrochloric acid.

1 Scope

This European standard specifies methods for analysis of chlorides resulting from hydrogen chloride absorption, according to EN 1911-2, in waste gases having been sampled and filtered according to EN 1911-1.

All compounds which are volatile at the filtration temperature and produce chloride ions upon dissolution during sampling are measured by this method, which gives therefore the volatile inorganic chlorides content of gases expressed as HCl. In most cases, this corresponds to the hydrogen chloride content ; an indicative distinction between hydrogen chloride and volatile chlorides may be achieved, using additional checks.

This standard specifies also validation criteria for the whole HCl measurement method described in EN 1911-1, EN 1911-2 and EN 1911-3, and indicates the performance characteristics of this measurement method.

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.

EN 1911-1	Stationary source emissions - Manual method of determination of HCl - Part 1 : Sampling of gases
EN 1911-2:1998	Stationary source emissions - Manual method of determination of HCl - Part 2 : Gaseous compounds absorption.
EN ISO 3696:1995	Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)
EN ISO 10304-1:1995	Water quality - Determination of dissolved fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulfate ions, using liquid chromatography of ions - Part 1 : Method for water with low contamination (ISO 10304-1:1992)

3 Analysis

3.1 Introduction

After sampling according to EN 1911- 1 and EN 1911-2, the solutions shall be analysed by one of the following methods :

- silver titration : potentiometric method (Method A) ;
- mercuric-thiocyanate spectrophotometry (Method B) ;
- ion-exchange chromatography (Method C).

The method to be used depends on the anticipated range of chloride concentration to be measured, therefore on HCl concentration in sampled gases, on sampled gas volumes, and on the final solution volumes (absorption solution and rinses).

Because the detection limit of method A is approximately $0,5 \text{ mg}\cdot\text{l}^{-1}$ to $1 \text{ mg}\cdot\text{l}^{-1}$, this method should not be used for the measurement of chloride concentrations less than $2 \text{ mg}\cdot\text{l}^{-1}$, which corresponds usually to HCl concentrations in gases less than $1 \text{ mg}\cdot\text{m}^{-3}$ (sampled volume $0,2 \text{ m}^3$, solution volume approximately 100 ml).

Methods B and C, which have a detection limit approximately $0,05 \text{ mg}\cdot\text{l}^{-1}$ to $0,1 \text{ mg}\cdot\text{l}^{-1}$, may be used in any case, if necessary after dilution of the solution to be analysed ; their results may be considered as equivalent (see annex B).

3.2 Reagents and samples to be analysed

3.2.1 Reagents for analysis

All reagents shall be of analytical grade.

The reagents common to the 3 methods are :

- chloride-free water of at least grade 2 purity according to EN ISO 3696:1995. (Conductivity less than $100 \mu\text{S}\cdot\text{m}^{-1}$) ;
- stock solution of sodium chloride . Dissolve $1,603 \text{ g}$ of sodium chloride previously dried 2 h at $110 \text{ }^{\circ}\text{C}$ in 1 l of water. 1 ml of solution corresponds to 1 mg HCl ;
- reference solution of sodium chloride freshly prepared by taking 10 ml of stock solution and diluting to $1\,000 \text{ ml}$. This solution then corresponds to a concentration in HCl of $0,01 \text{ mg}\cdot\text{ml}^{-1}$.

Other reagents for particular analytical methods are specified in 3.3, 3.4 and 3.5.

3.2.2 Samples to be analysed

3.2.2.1 Absorption solutions (S_e)

According to 6.3.6 of EN 1911-2:1998, the washing bottle(s) content and the rinses are poured into a flask after sampling. They are made up to a known volume (250 ml for example).

To determine absorption efficiency, the second washing bottle content can be analysed separately.

SIST EN 1911-3:1998
<https://standards.iteh.ai/catalog/standards/sist/93be32e7-6c74-4686-ac1d-5562edad3a00/sist-en-1911-3-1998>

3.2.2.2 Samples of equipment blank

These samples (see 7.2 of EN 1911-2:1998) are to make up to a known volume, as for the tests in 3.2.2.1.

3.3 Silver titration : potentiometric method

3.3.1 Apparatus

A potentiometric determination system including a silver electrode and a reference electrode, or a combined silver electrode. These electrodes shall release no chloride during the determination.

3.3.2 Reagents and solutions

3.3.2.1 Silver nitrate, reference solution $0,1 \text{ mol}\cdot\text{l}^{-1}$ shall be freshly prepared e.g. from commercially available ampoules, and shall be kept in a brown bottle.

3.3.2.2 Silver nitrate, reference solution, $0,02 \text{ mol}\cdot\text{l}^{-1}$ freshly prepared by dilution to 1/5 of solution (see 3.3.2.1) and kept in a brown bottle.

3.3.2.3 Nitric acid solution, approximately $1 \text{ mol}\cdot\text{l}^{-1}$ prepared by diluting 70 ml of HNO_3 ($\rho = 1,42 \text{ g}\cdot\text{cm}^{-3}$) to 1 000 ml with water.

3.3.3 Procedure

Pipette an aliquot of solution to be analysed into a titration flask and add 5 ml of nitric acid (see 3.3.2.3).

Lower the electrodes into the liquid.

If necessary, add sufficient chloride free water (see 3.2.1) to allow the electrode to be covered.

Stir the medium with a magnetic bar.

According to the type of potentiometric determination system used, use the appropriate procedure as follows :

- record the whole potential titration curve and determine the volume at the inflexion point (see 3.3.4.1) ;
- using potentiometry with a predetermined final potential, ie. determine, the final potential prior to the measurements, under measurement conditions, on a known quantity of chloride ;
- continuously add identical volumes of titrate and determine the volume resulting in maximum potential increment.

Titrate the sample with silver nitrate solution described in 3.3.2.1 (see 3.3.2.2 in the case where using $0,1 \text{ mol}\cdot\text{l}^{-1}$ solution results in the use of small volume which can not be determined accurately).

Repeat the procedure with chloride free water, in order to take into account the reagent blank value.

3.3.4 Interferences

Any ions reacting with silver ions (such as Br^- , I^- , CN^- , S^{2-} , SO_3^{2-} , SCN^-) may be interferent.

3.3.4.1 The recording titration curve method (see 3.3.3) allows the presence of interferents to be established, if any, when the curve shows more than one inflexion point.

When bromides and iodides have the same order of concentration as chlorides, recorded titration provide separate measuring spots and separate estimation. The measurements is carried out in the following order : iodide or bromide then chloride.

3.3.4.2 Interference by sulfides and sulfites can be eliminated by adding a few drops of concentrated hydrogen peroxide solution (30 %).

3.3.4.3 Interference from cyanides can be eliminated by adding formaldehyde. Where thiocyanates are present as a result of the presence of both sulfides and cyanides, those ions can be eliminated by oxidation under heat using hydrogen peroxide solution at pH 10 in an ammoniacal medium. After oxidation, the aliquot solution shall be acidified by adding HNO_3 before titration.

3.3.5 Calculations

The HCl quantity in the absorption solution is calculated using the following formula :

$$Q_{\text{HCl}} = \frac{a}{b} \cdot (V - V_0) \cdot C_{\text{Ag}} \cdot 36,5 \quad (1)$$

where :

Q_{HCl} is the quantity of HCl collected, in milligrams ;

a is the volume of absorption solution S_e (see 3.1.2.1), in millilitres ;

b is the aliquot portion of S_e (see 3.1.2.1) used for analysis, in millilitres ;

V is the volume of the Ag NO_3 solution used for dosing the solution S_e , (see 3.1.2.1) in millilitres ; <https://standards.iteh.ai/catalog/standards/sist/93be32e7-6c74-4686-ac1d-5562edad3a00/sist-en-1911-3-1999>

V_0 is the volume of the Ag NO_3 solution used for taking into account the reagent blank value in millilitres ;

C_{Ag} is the concentration of silver nitrate solution, in mole per litre ;

36,5 is the molar weight of HCl.