
**Thermal insulating products for
building equipment and industrial
installations — Determination of trace
quantities of water-soluble chloride,
fluoride, silicate, sodium ions and pH**

*Produits isolants thermiques pour les équipements de bâtiments et
les installations industrielles — Détermination des faibles quantités
d'ions chlorure, fluorure, silicate et sodium solubles dans l'eau et
mesure du pH*



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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 documents 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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 163, *Thermal performance and energy use in the built environment*, Subcommittee SC 1, *Test and measurement methods*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 88, *Thermal insulating materials and products*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 12624:2011), which has been technically revised.

The main changes are as follows:

- EN 13468:2001 and ISO 12624:2011 have been merged into one document;
- [Clause 3](#), Terms and definitions, has been added and the numbering of the following clauses has been changed accordingly;
- editorial revisions.

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.

Thermal insulating products for building equipment and industrial installations — Determination of trace quantities of water-soluble chloride, fluoride, silicate, sodium ions and pH

1 Scope

This document specifies the equipment and procedures for determining trace quantities of the water-soluble chloride, fluoride, silicate and sodium ions in an aqueous extract of the product. It also describes a procedure for the determination of the pH of the aqueous extract. It is applicable to thermal insulating products.

NOTE The determination of these parameters can be relevant for thermal insulating products intended for application to stainless austenitic steel surfaces. The presence of chloride, fluoride, silicate and sodium ions under certain conditions can influence the risk of stress corrosion cracking. See [Annex B](#) for further information on general use of this document.

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 10136-1, *Glass and glassware — Analysis of extract solutions — Part 1: Determination of silicon dioxide by molecular absorption spectrometry*

ISO 10136-2, *Glass and glassware — Analysis of extract solutions — Part 2: Determination of sodium oxide and potassium oxide by flame spectrometric methods*

3 Terms and definitions

No terms and definitions are listed in this document.

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

Test specimens of the insulating product are boiled or heated in deionized water to leach out soluble ions. Tests to determine water-soluble chloride, fluoride, silicate and sodium ions are performed on aliquots of the filtered aqueous extract. A pH value is determined on one of the aliquots.

5 Apparatus

5.1 General information

All equipment and working instruments used for this purpose shall be free of soluble chloride, fluoride, silicate, sodium ions and grease. Chloride-free solvents shall be used to clean equipment and working

instruments (avoid chlorinated solvents). During the performance of the analytic procedure, extraneous sources of these ions shall be reduced or eliminated.

5.2 Preparation of aqueous extracts

5.2.1 1 000 ml borosilicate glass flask.

5.2.2 Water condenser.

5.2.3 Heater, conforming to the requirements of [8.2](#).

5.2.4 Prewashed filter, diameter of pores 0,45 µm.

5.2.5 Cork borer, having a diameter which enables taking the specified mass for the test specimen.

5.2.6 Disposable gloves.

5.2.7 Plastic bag or container.

5.2.8 Balance, allowing a reading to $\pm 0,01$ g.

5.3 Analyses

5.3.1 Ion chromatography equipment (IC).

5.3.2 Titration equipment with automatic end point.

5.3.3 Atomic absorption spectrophotometer (AAS).

5.3.4 Atomic emission spectrometer with inductively coupled plasma (ICP-AES).

5.3.5 Spectrophotometer.

5.3.6 pH meter/millivolt meter.

5.3.7 Fluoride ion selective electrode.

NOTE Any device providing the same result with at least the same accuracy can be used.

5.4 Materials

5.4.1 Deionized water, conductivity less than 0,5 µS/cm.

5.4.2 0,01 N sodium chloride (NaCl).

5.4.3 0,01 N silver nitrate (AgNO₃).

6 Test specimens

6.1 General

To ensure that the results are representative of the product, care shall be taken that the test specimens are not subjected to any outside contamination.

6.2 Dimensions of test specimens

Take out each test specimen by using a cork borer at positions evenly distributed over the total surface of a full-size product. To avoid contamination, chloride-free gloves shall be worn and the test specimen shall be placed in a closed polyethylene bag. Depending on the product family, the weight of each test specimen shall be at least 7,5 g or 20 g.

6.3 Number of test specimens

The number of test specimens shall be as specified in the relevant product standard. If the number is not specified, then at least three test specimens (three weights of 7,5 g or 20 g) shall be used.

NOTE In the absence of a product standard or any other technical specification, the number of test specimens can be agreed between parties.

6.4 Conditioning of test specimens

The test specimens shall be stored for at least 6 h and not more than 24 h at $(23 \pm 5) ^\circ\text{C}$. In case of dispute, they shall be stored at $(23 \pm 2) ^\circ\text{C}$ for the time stated in the relevant product standard.

In tropical climates, different conditioning and testing conditions can be relevant. In this case, the conditions shall be $(27 \pm 5) ^\circ\text{C}$ and $(65 \pm 5) \%$ relative humidity (RH) and shall be stated clearly in the test report.

7 Procedure

7.1 Test conditions

The test shall be carried out at $(23 \pm 5) ^\circ\text{C}$.

In tropical climates, different conditioning and testing conditions can be relevant. In this case, the conditions shall be $(27 \pm 2) ^\circ\text{C}$ and $(65 \pm 5) \% \text{ RH}$.

7.2 Test procedure

7.2.1 Preparation of the leaching solution

Take $(20 \pm 0,1) \text{ g}$ or $(7,5 \pm 0,05) \text{ g}$ of the test specimen (m), as specified in the relevant product standard and place it in the wide-mouth glass flask. Add $(400 \pm 5) \text{ ml}$ deionized water (V). The flask shall be closed with a ground-in stopper and shaken until the test specimen is completely wetted and submerged. If the test specimen floats, it should be held under water (see [Annex C](#)).

Fix the water condenser to the flask and electrically heat for 5 min to 10 min until the leaching solution has reached the test temperature. The leaching process shall be continued, under reflux conditions, for the specified time.

Depending on the maximum service temperature specified for the product, the test temperature and leaching time shall be chosen as specified in [Table 1](#).

Table 1 — Test temperature and leaching time

Maximum service temperature °C	Test temperature °C	Leaching time h
≥ 100	(100 ± 1)	0,5
≥ 90 to < 100	(90 ± 1)	1,0
≥ 80 to < 90	(80 ± 1)	2,0
≥ 70 to < 80	(70 ± 1)	4,0
< 70	(60 ± 1)	8,0

Remove the flask with the condenser from the heat and cool for 10 min to 15 min until the temperature reaches room temperature, e.g. in a water bath. Filter the leaching solution.

Make a “blank” test, using the same procedure as above, but without a test specimen of insulating product.

The filtered solutions will subsequently be used for the analyses.

NOTE Other test methods than described below can be used if the same result with at least the same accuracy is obtained.

7.2.2 Chloride determination

7.2.2.1 General

One of the following methods shall be used on a fresh aliquot of the filtered solution. In case of dispute, the method described in 7.2.2.2 is the reference method. The repeatability of the methods is calculated as the standard deviation on 10 determinations taken from the same leaching solution. The standard deviation shall be smaller than 0,5 mg/kg for concentrations smaller than 8,3 mg/kg and smaller than 6 % of the mean of the 10 determinations for concentrations larger than or equal to 8,3 mg/kg.

7.2.2.2 Ion chromatographic (IC) determination

The ion chromatographic determination of chloride-ions shall be carried out in accordance with the equipment manufacturer's operating instructions.

Inject 5 ml of the filtered solution into the test loop through a disposable, chloride-free filter. The volume of the test loop of the IC apparatus is chosen (normally 50 µl).

NOTE Depending on the kind of binding or waterproofing agent, mineral wool products can release organic contaminants during the boiling process, which can cause peaks that are close to the chloride peaks in the ion chromatogramme.

A clear separation of test peaks shall be obtained during the IC determination.

7.2.2.3 AgNO₃ titration

The determination of chloride ions by polarization titration shall be according to the alternating current voltage method with amperometric indication.

Precautions shall be taken to avoid interference from other ions, e.g. fluoride.

The following procedure shall be carried out on aliquots of the filtered solution (minimum 200 ml, V_f) from the tested product and the blank.

Where necessary, in order to remove organic contaminants, 1 ml to 2 ml HNO₃ (65 % w/w HNO₃, diluted 1:1) and 0,5 g of fine activated carbon shall be added to the solution before filtering through a prewashed filter.

For cellular glass, 50 mg zinc acetate shall also be added to precipitate sulfide. The solution shall then be stirred for a few minutes (magnetic stirrer) before filtering.

Take a measured amount of the (filtered) solution (V_t in millilitres) and add 2,00 ml of 0,01 N NaCl solution (to improve the end-point observation). Titrate with 0,01 N AgNO_3 solution using the titration equipment. The end point is given by a sudden change in the meter reading.

In advance of carrying out the titration, the silver electrode of the titration equipment shall be cleaned and prepared according to the equipment manufacturer's operating instructions.

7.2.3 Fluoride determination

7.2.3.1 General

The repeatability of the methods is calculated as the standard deviation on 10 determinations taken from the same leaching solution. The standard deviation shall be smaller than 0,5 mg/kg for concentrations smaller than 8,3 mg/kg and smaller than 6 % of the mean of the 10 determinations for concentrations larger than or equal to 8,3 mg/kg.

7.2.3.2 Ion chromatographic (IC) determination

The ion chromatographic (IC) determination of fluoride-ions shall be carried out in accordance with the equipment manufacturer's operating instructions.

Inject 5 ml of the filtered solution into the test loop through a disposable, fluoride-free filter. The volume of the test loop of the IC apparatus is chosen (normally 50 μl).

NOTE Depending on the kind of binding or waterproofing agent, mineral wool products can release organic contaminants during the boiling process, which can cause peaks that are close to the fluoride peaks in the ion chromatogramme.

A clear separation of test peaks shall be obtained during the IC determination.

7.2.3.3 Fluoride selective electrode determination

The selective electrode determination of fluoride shall be carried out in accordance with equipment manufacturer's operating instructions.

7.2.3.4 Spectrophotometric method

The spectrophotometric determination of fluoride shall be carried out in accordance with [Annex A](#).

7.2.4 Silicate determination

7.2.4.1 General

One of the following methods shall be carried out on a fresh aliquot of the filtered solution. The repeatability of the methods is calculated as the standard deviation on 10 determinations taken from the same leaching solution. The standard deviation shall be smaller than 30 mg/kg for concentrations smaller than 430 mg/kg and smaller than 7 % of the mean of the 10 determinations for concentrations larger than or equal to 430 mg/kg.

7.2.4.2 Silicate determination by use of atomic absorption spectrophotometry (AAS)

The AAS determination of silicate-ions shall be carried out in accordance with the equipment manufacturer's operating instructions.

7.2.4.3 Silicate determination by use of atomic emission spectrometer with inductively coupled plasma (ICP-AES)

The ICP-AES determination of silicate-ions shall be carried out in accordance with the equipment manufacturer's operating instruction.

7.2.4.4 Silicate determination according to ISO 10136-1

Measure in accordance with ISO 10136-1.

7.2.5 Sodium determination

7.2.5.1 General

One of the following methods shall be carried out on a fresh aliquot for the filtered solution. The repeatability of the methods is calculated as the standard deviation on 10 determinations taken from the same leaching solution. The standard deviation shall be smaller than 2,0 mg/kg for concentrations smaller than 40 mg/kg and smaller than 5 % of the mean of the 10 determinations for concentrations larger than or equal to 40 mg/kg.

7.2.5.2 Sodium determination according to ISO 10136-2

Measure in accordance with ISO 10136-2.

7.2.5.3 Sodium determination by use of atomic emission spectrometer with inductively coupled plasma (ICP-AES)

The ICP-AES determination of sodium-ions is carried out in accordance with the equipment manufacturer's operating instructions.

7.2.6 pH determination by use of pH meter

The pH shall be measured within 24 h after the preparation of the leaching solution.

8 Calculation and expression of results

8.1 General

The results shall be expressed as the mean value of the individual measurements.

8.2 Chloride (Cl^-)

The results shall be expressed to the nearest 0,1 mg/kg.

8.2.1 Ion chromatography

Calculate the chloride content, $w(\text{Cl}^-)$, in mg/kg Cl^- for the product using [Formula \(1\)](#):

$$w(\text{Cl}^-) = \frac{(c_1 - c_2)V}{m} \quad (1)$$

where

c_1 (Cl^-) is the concentration of chloride-ions in the filtered solution, in milligrammes per litre;

c_2 (Cl^-) is the concentration of chloride-ions in the blank test, in milligrammes per litre;

V is the volume of water used for leaching, in litres;

m is the mass of the test specimen, in kilogrammes.

8.2.2 Silver nitrate titration

Calculate the chloride content, $w(\text{Cl}^-)$, in mg/kg Cl^- for the product using [Formula \(2\)](#):

$$w(\text{Cl}^-) = \frac{(V_1 - V_2) \times V \times F}{V_t \times m \times 1\,000} \quad (2)$$

where

V_1 is the AgNO_3 solution added in titrating, in millilitres;

V_2 is the AgNO_3 solution added in titrating during blank test, in millilitres;

V_t is the volume for titration, in litres;

V is the volume of water used for leaching, in litres;

m is the mass of the test specimen, in kilogrammes;

F is the stoichiometric factor given by the reaction (equals 355 with a 0,01 N AgNO_3 solution).

8.3 Fluoride (F^-)

The results shall be expressed to the nearest 0,1 mg/kg.

Calculate the fluoride content, $w(\text{F}^-)$, in mg/kg F^- for the product using [Formula \(3\)](#):

$$w(\text{F}^-) = \frac{(c_1 - c_2)V}{m} \quad (3)$$

where

c_1 (F^-) is the concentration of fluoride-ions in the filtered solution, in milligrammes per litre;

c_2 (F^-) is the concentration of fluoride-ions in the blank test, in milligrammes per litre;

V is the volume of water used for leaching, in litres;

m is the mass of the test specimen, in kilogrammes.

8.4 Silicate (SiO_3^{--})

The results shall be expressed to the nearest mg/kg.

Calculate the silicate content, $w(\text{SiO}_3^{--})$, in mg/kg SiO_3^{--} for the product using [Formula \(4\)](#):

$$w(\text{SiO}_3^{--}) = \frac{(c_1 - c_2)V}{m} \quad (4)$$

where

c_1 (SiO_3^{--}) is the concentration of silicate-ions in the filtered solution, in milligrammes per litre;

c_2 (SiO_3^{--}) is the concentration of silicate-ions in the blank test, in milligrammes per litre;

V is the volume of water used for leaching, in litres;

m is the mass of the test specimen, in kilogrammes.

8.5 Sodium (Na^+)

The results shall be expressed to the nearest mg/kg.

Calculate the sodium content, $w(\text{Na}^+)$, in mg/kg Na^+ for the product using [Formula \(5\)](#):

$$w(\text{Na}^+) = \frac{(c_1 - c_2)V}{m} \quad (5)$$

where

c_1 (Na^+) is the concentration of sodium-ions in the filtered solution, in milligrammes per litre;

c_2 (Na^+) is the concentration of sodium-ions in the blank test, in milligrammes per litre;

V is the volume of water used for leaching, in litres;

m is the mass of the test specimen, in kilogrammes.

8.6 pH

Express pH to the nearest 0,1 part of a unit.

9 Accuracy of measurement

Interlaboratory tests performed by five laboratories on eight different products (mineral wool and cellular glass) have given the 95 % confidence interval values shown in [Table 2](#).

Table 2 — 95 % confidence interval values

Component	95 % confidence interval value	Range of trace quantity mg/kg
Cl^-	mean ± 10 %	0 to 170
F^-	mean ± 7 %	0 to 30
SiO_3^{--}	mean ± 10 %	500 to 3 600
Na^+	mean ± 5 %	40 to 1 800

Trueness test on reference solutions performed by the five laboratories (only one concentration for each component) are shown in [Table 3](#).

Table 3 — Trueness test

Component	True value mg/kg	Measured mean value mg/kg	Bias %
Cl^-	2,0	2,02	1,0
F^-	1,0	1,01	1,0
SiO_3^{--}	135,0	135,4	0,3
Na^+	75,0	75,1	0,1

The bias is insignificant for all components.

10 Test report

The test report shall include the following information:

- a) reference to this document, i.e. ISO 12624:2022;
 - b) product identification:
 - 1) product name, factory, manufacturer or supplier;
 - 2) production code number;
 - 3) type of product;
 - 4) packaging;
 - 5) the form in which the product arrived at the laboratory;
 - 6) other information as appropriate, e.g. nominal dimensions, nominal density;
 - c) test procedure:
 - 1) pre-test history and sampling, e.g. who sampled and where;
 - 2) conditioning;
 - 3) deviation from [Clauses 6](#) and [7](#), if applicable;
 - 4) date of testing;
 - 5) conditioning and testing conditions in tropical climates, if applicable;
 - 6) general information relating to the test including reference to the methods used and leaching temperature and time;
 - 7) events which can have affected the results.
- Information about the apparatus and identity of the technician should be available in the laboratory but it need not be recorded in the report.
- d) results;
 - e) all individual values and the mean value for each of the measured components.

Annex A (normative)

Spectrophotometric determination of fluoride with zirconium SPADNS

A.1 Principle

Fluoride reacts with the zirconium-dye-lake, dissociating a portion of it into a complex anion (ZrF_6^{-}). As the amount of fluoride is increased, the colour produced becomes progressively lighter. Absorption versus fluoride concentration gives a straight line of inverse slope in the fluoride concentration range of 0,00 mg/kg to 1,4 mg/kg.

A.2 Apparatus

Spectrophotometer (set to 570 nm).

A.3 Reagents

A.3.1 SPADNS solution. Dissolve 0,958 g sodium 2(p-sulphophenylazo)-1,8 dihydroxy-3,6 naphthalene disulfonate in distilled water and dilute to 500 ml.

A.3.2 Zirconium(IV) solution. Dissolve 0,133 g zirconyl chloride octahydrate, $\text{ZrOCl}_2 \times 8\text{H}_2\text{O}$, in about 25 ml distilled water. Add 350 ml concentrated HCl and dilute to 500 ml with distilled water.

A.3.3 Mixed reagent solution. Mix equal volumes of the SPADNS and zirconium(IV) solution.

A.3.4 Reference solutions. Add 10,0 ml of the SPADNS solution to 100 ml distilled water. Dilute 10,0 ml concentrated HCl with 10,0 ml distilled water and add to the diluted SPADNS solution.

A.4 Calibration

Make a calibration graph, absorption (at 570 nm) versus F^- -concentration, using 0,05 mg/kg, 0,1 mg/kg, 0,2 mg/kg, 0,5 mg/kg, 0,7 mg/kg, 1,0 mg/kg, 1,5 mg/kg, 2,0 mg/kg, based on a stock solution of 0,221 g/l NaF (= 100 mg/kg). This standard graph shall be prepared each time that a fresh batch (zirconium-SPADNS) mixed reagent solution has been prepared (has been known to remain stable for at least two years).

A.5 Procedure

Take a 50,0 ml aliquot of the sample solution and add 10,0 ml of the mixed (zirconium-SPADNS) reagent solution. Mix well after addition of this reagent. Read absorption, after 10 min, against reference solution and determine F^- -concentration by reference to the calibration graph.

A.6 Interferences

Colour and turbidity shall be removed, or compensation made to minimize their interference.