INTERNATIONAL STANDARD

ISO 12624

First edition
2011-01-15

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 — Détermination des faibles quantités d'ions chlorure, fluorure, silicate et sodium solubles dans l'eau et mesure du pH

Reference number
ISO 12624:2011(E)

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Published in Switzerland

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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 12624 was prepared by Technical Committee ISO/TC 163, *Thermal performance and energy use in the built environment*, Subcommittee SC 1, *Test and measurement methods*.

ISO 12624 includes the original EN 13468 prepared by Technical Committee CEN/TC 88 *Thermal insulating materials and products*. However,

- Subclause 5.4, “conditioning of test specimen”,
- Subclause 6.1, “test conditions”, and
- Clause 9, “test report”

have been modified to reflect conditions for tropical countries.

This International Standard is one of a series of standards which specify test methods for determining dimensions and properties of thermal insulating materials and products. The original EN 13468 supports a series of product standards for thermal insulating materials and products which derive from the Council Directive of 21 December 1988 on the approximation of laws, regulations and administrative provisions of the Member States relating to constructive products (Directive 89/106/EEC) through the consideration of the essential requirements.

This International Standard is one of a series of existing European Standards on test methods for products used to insulate building equipment and industrial installations which is comprised of the following group of International Standards:

ISO standard	Title	Respective EN standard
ISO 12623	*Thermal insulating products for building equipment and industrial installations — Determination of short-term water absorption by partial immersion of preformed pipe insulation*	EN 13472
ISO 12624	*Thermal insulation products — Determination of trace quantities of water soluble chloride, fluoride, silicate, sodium ions and pH*	EN 13468
ISO 12628	*Thermal insulating products for building equipment and industrial installations — Determination of dimensions, squareness and linearity of preformed pipe insulation*	EN 13467
ISO 12629	*Thermal insulating products for building equipment and industrial installations — Determination of water vapour transmission properties of preformed pipe insulation*	EN 13469

A further series of existing European Standards on test methods was adopted by ISO. This "package" of standards comprises the following group of interrelated standards:

ISO standard	Title	Respective EN standard
ISO 29465	*Thermal insulating products for building applications — Determination of length and width*	EN 822
ISO 29466	*Thermal insulating products for building applications — Determination of thickness*	EN 823
ISO 29467	*Thermal insulating products for building applications — Determination of squareness*	EN 824
ISO 29468	*Thermal insulating products for building applications — Determination of flatness*	EN 825
ISO 29469	*Thermal insulating products for building applications — Determination of compression behaviour*	EN 826
ISO 29470	*Thermal insulating products for building applications — Determination of the apparent density*	EN 1602
ISO 29471	*Thermal insulating products for building applications — Determination of dimensional stability under constant normal laboratory conditions (23 degrees C/50 % relative humidity)*	EN 1603
ISO 29472	*Thermal insulating products for building applications — Determination of dimensional stability under specified temperature and humidity conditions*	EN 1604
ISO 29764	*Thermal insulating products for building applications — Determination of deformation under specified compressive load and temperature conditions*	EN 1605
ISO 29765	*Thermal insulating products for building applications — Determination of tensile strength perpendicular to faces*	EN 1607
ISO 29766	*Thermal insulating products for building applications — Determination of tensile strength parallel to faces*	EN 1608
ISO 29767	*Thermal insulating products for building applications — Determination of short-term water absorption by partial immersion*	EN 1609

ISO standard	Title	Respective EN standard
ISO 29768	*Thermal insulating products for building applications — Determination of linear dimensions of test specimens*	EN 12085
ISO 29769	*Thermal insulating products for building applications — Determination of behaviour under point load*	EN 12430
ISO 29770	*Thermal insulating products for building applications — Determination of thickness for floating-floor insulating products*	EN 12431
ISO 29771	*Thermal insulating materials for building applications — Determination of organic content*	EN 13820
ISO 29803	*Thermal insulation products for building applications — Determination of the resistance to impact of external thermal insulation composite systems (ETICS)*	EN 13497
ISO 29804	*Thermal insulation products for building applications — Determination of the tensile bond strength of the adhesive and of the base coat to the thermal insulation material*	EN 13494
ISO 29805	*Thermal insulation products for building applications — Determination of the mechanical properties of glass fibre meshes*	EN 13496

The Application of Agreement on technical cooperation between ISO and CEN (Vienna Agreement), Modes 1, 2, 4 and 5, was not approved by CEN/TC 88 and the necessity not seen by its stakeholders.

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 International Standard 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. This International Standard is applicable to thermal insulating products.

NOTE The determination of these parameters may 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.

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 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 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.

4 Apparatus

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.

4.1 Preparation of aqueous extract

4.1.1 1 000 ml borosilicate glass flask.

4.1.2 Water condenser.

4.1.3 Heater, complying with the requirements of 7.2.

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

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

4.1.6 Disposable gloves.

4.1.7 Plastic bag or container.

4.1.8 Balance, permitting reading to ±0,01 g.

4.2 Analyses

4.2.1 Ion chromatography equipment (IC). See Annex C for an example of suitable equipment.

4.2.2 Titration equipment with automatic end point.

4.2.3 Atomic Absorption Spectrophotometer (AAS).

4.2.4 Atomic Emission Spectrometer with Inductively Coupled Plasma (ICP-AES).

4.2.5 Spectrophotometer.

4.2.6 pH meter/millivolt meter.

4.2.7 Fluoride ion selective electrode.

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

4.3 Materials

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

4.3.2 0,01 N sodium chloride (NaCl).

4.3.3 0,01 N silver nitrate ($AgNO_3$).

5 Test specimens

5.1 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.

5.2 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.

5.3 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 European technical specification, the number of the test specimens may be agreed between parties.

5.4 Conditioning of test specimens

The test specimens shall be stored for at least 6 h and not more than 24 h at (23 ± 5) °C. In case of dispute, they shall be stored at (23 ± 2) °C for the time stated in the relevant product standard.

In tropical countries, different conditioning and testing conditions can be relevant. In this case, the conditions shall be (27 ± 5) °C and (65 ± 5) % RH (relative humidity), and shall be stated clearly in the test report.

6 Procedure

6.1 Test conditions

The test shall be carried out at (23 ± 5) °C.

In tropical countries, different conditioning and testing conditions can be relevant. In this case, the conditions shall be (27 ± 2) °C and (65 ± 5) % RH.

6.2 Test procedure

6.2.1 Preparation of the leaching solution

Take (20 ± 0,1) g or (7,5 ± 0,05) g of the test specimen (m), as specified in the relevant product standard and place it in the wide-mouth glass flask. Add (400 ± 5) 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 informative Annex D).

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
$\geqslant$ 100	(100 ± 1)	0,5
$\geqslant$ 90 to < 100	(90 ± 1)	1,0
$\geqslant$ 80 to < 90	(80 ± 1)	2,0
$\geqslant$ 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.

6.2.2 Chloride determination

6.2.2.1 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 6.2.2.2 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.

6.2.2.2 Ion chromatographic (IC) determination

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

5 ml of the filtered solution is injected 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.

6.2.2.3 $AgNO_3$ 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_t) from the tested product and the blank.

Where necessary, in order to remove organic contaminants, 1 ml to 2 ml HNO_3 (65 % HNO_3, 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 be added, too, to precipitate sulphide. 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.

6.2.3 Fluoride determination

6.2.3.1 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.

6.2.3.2 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.

5 ml of the filtered solution is injected 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.

6.2.3.3 Fluoride selective electrode determination

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

6.2.3.4 Spectrophotometric method

The spectrophotometric determination of fluoride shall be carried out in accordance with Annex A.

6.2.4 Silicate determination

6.2.4.1 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.

6.2.4.2 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.

6.2.4.3 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.

6.2.4.4 Silicate determination according to ISO 10136-1

Measure in accordance with ISO 10136-1.

6.2.5 Sodium determination

6.2.5.1 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.

6.2.5.2 Sodium determination according to ISO 10136-2

Measure in accordance with ISO 10136-2.

6.2.5.3 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.

6.2.6 pH determination by use of pH meter

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

7 Calculation and expression of results

The results are expressed as the mean value of the individual measurements.

7.1 Chloride (Cl^-)

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

7.1.1 Ion-chromatography

Calculate the chloride content, w (Cl^-), in mg/kg Cl^- for the product using the equation:

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

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.

7.1.2 Silver nitrate titration

Calculate the chloride content, w (Cl^-), in mg/kg Cl^- for the product using the equation:

$$w(\mathrm{Cl}^-) = \frac{\left[\mathrm{ml\ AgNO_3(test\ specimen)\ ml\ AgNO_3(blank)}\right] \times V \times F}{V_\mathrm{t} \times m \times 1000} \tag{2}$$

where

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).

7.2 Fluoride (F^-)

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

Calculate the fluoride content, w (F^-), in mg/kg F^- for the product using the equation:

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

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, ½ 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.

7.3 Silicate (SiO_3^{--})

The results are expressed to the nearest mg/kg.

Calculate the silicate content, w (SiO_3^{--}), in mg/kg SiO_3^{--} for the product using the equation:

$$w(SiO_3^{--}) = \frac{(c_1 - c_2)V}{m} \tag{4}$$

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.

7.4 Sodium (Na^+)

The results are expressed to the nearest mg/kg.

Calculate the sodium content, w (Na^+), in mg/kg Na^+ for the product using the equation:

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

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.

7.5 pH

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

8 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

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.

9 Test report

The test report shall include the following information:

a) reference to this International Standard;

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 5 and 6, if applicable;
4) date of testing;
5) conditioning and testing conditions in tropical countries, if applicable;
6) general information relating to the test including reference to the methods used and leaching temperature and time;
7) events which may 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-sulfophenylazo)-1,8-dihydroxy-3,6-napthalene disulfonate in distilled water and dilute to 500 ml.

A.3.2 Zirconium(IV) solution. Dissolve 0,133 g zirconyl chloride-octahydrate, $ZrOCl_2 \times 8H_2O$, 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 exactly 10,0 ml of the mixed (zirconium-SPADNS) reagent solution. Mix well after addition of this reagent. Read absorption, after exactly 10 minutes, 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.