BRITISH STANDARD

BS EN 13468:2001

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

The European Standard EN 13468:2001 has the status of a British Standard

ICS 91.120.10; 91.100.60

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National foreword

This British Standard is the official English language version of EN 13468:2001. This British Standard, together with BS EN 13467:2001, BS EN 13469:2001, BS EN 13470:2001, BS EN 13471:2001 and BS EN 13472:2001, partially supersedes BS 2972:1989.

The UK participation in its preparation was entrusted by Technical Committee RHE/9, Thermal insulating materials, to Subcommittee RHE/9/4, Nomenclature and specifications, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled “International Standards Correspondence Index”, or by using the “Find” facility of the BSI Standards Electronic Catalogue.

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This British Standard, having been prepared under the direction of the Engineering Sector Policy and Strategy Committee, was published under the authority of the Standards Policy and Strategy Committee on 09 November 2001

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 13468

September 2001

ICS 91.100.60

English version

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 l'équipement du bâtiment et les installations industrielles - Détermination des faibles quantités d'ions chlorure, fluorure, silicate et sodium solubles dans l'eau et mesure du pH

Wärmedämmstoffe für die Haustechnik und für betriebstechnische Anlagen - Bestimmung des Gehalts von wasserlöslichen Chlorid-, Fluorid-, Silikat- und Natrium-Ionen und des pH-Wertes

This European Standard was approved by CEN on 18 August 2001.

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 Management Centre 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 Management Centre has the same status as the official versions.

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Ref. No. EN 13468:2001 E

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 88 "Thermal insulating materials and products", 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 March 2002, and conflicting national standards shall be withdrawn at the latest by March 2002.

This European Standard is one of a series of standards which specify test methods for determining dimensions and properties of thermal insulating materials and products. It 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 construction products (Directive 89/106/EEC) through the consideration of the essential requirements.

This standard contains the following normative annex:

Annex A - Spectrophotometric fluoride determination of Fluoride with zirconium-SPADNS

and three informative annexes:

Annex B - General information related to the use of this standard

Annex C - Example of ion chromatography equipment

Annex D - Example of borosilicate glass equipment for preparation of leaching solutions for thermal insulating products that float

This European Standard has been prepared for products used to insulate building equipment and industrial installations, but it may also be applied to products used in other areas.

No existing European Standard is superseded.

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

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.

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1 Scope

This 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. The 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 may influence the risk of stress corrosion cracking. See informative annex B for further information.

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. The latest edition of this undated reference applies (including amendments).

ISO 10136-1, *Glass and Glassware - Analyses of extract solutions – Part 1: Determination of silicon dioxide by molecular absorption spectrometry.*

ISO 10136-2, *Glass and Glassware - Analyses of extract solutions – Part 2: Determination of sodium oxide and potassium oxide by flame spectrometric methods.*

3 Terms and definitions

This European Standard contains no terms and definitions.

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

All equipment and working instruments used for this purpose must be free from 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.

5.1 Preparation of aqueous extract

5.1.1 1000 ml borosilicate glass flask;

5.1.2 Water condenser;

5.1.3 Heater, complying with the requirements of 7.2;

5.1.4 Prewashed filter, diameter of pores 0,45 µm;

5.1.5 Cork borer having a diameter which enables to take the specified mass for the test specimen;

5.1.6 Disposable gloves;

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5.1.7 Plastic bag or container;

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

5.2 Analyses

5.2.1 Ion Chromatography equipment (IC). See informative annex C for example of suitable equipment;

5.2.2 Titration equipment with automatic end point;

5.2.3 Atomic Absorption Spectrophotometer (AAS);

5.2.4 Atomic Emission Spectrometer with Inductively Coupled Plasma (ICP-AES);

5.2.5 Spectrophotometer;

5.2.6 pH meter/millivolt meter;

5.2.7 Fluoride ion selective electrode.

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

5.3 Materials

5.3.1 deionized water, conductivity less than 0,5 µS/cm;

5.3.2 0,01 N sodium cloride (NaCl);

5.3.3 0,01 N silver nitrate ($AgNO_3$);

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 must be worn and the test specimen is 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 (3 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.

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 ± 5) °C. In case of dispute they shall be stored at (23 ± 2) °C for the time stated in the relevant product standard.

7 Procedure

7.1 Test conditions

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

7.2 Test procedure

7.2.1 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 (example, 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
≥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 decribed below may 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 decribed 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.

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7.2.2.2 Ion-chromatographic determination (IC)

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

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

NOTE Depending on the kind of binding and/or waterproofing agent, mineral wool products may release organic contaminants during the boiling process which may 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 $AgNO_3$ 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 to remove organic contaminents 1ml 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 instruction.

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 determination (IC)

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

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

NOTE Depending on the kind of binding and/or waterproofing agent, mineral wool products may release organic contaminants during the boiling process which may 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.

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7.2.3.3 Fluoride selective electrode determination

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

7.2.3.4 Spectrophotometric method

The spectrophotometric determination of fluoride shall be carried out in accordance with the normative 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 instruction.

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 instruction.

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7.2.6 pH determination by use of pH meter

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

8 Calculation and expression of results

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

8.1 Chloride (Cl^-)

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

8.1.1 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.

8.1.2 Silver nitrate titration

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

$$w\,(\mathrm{Cl}^-) = \frac{[\mathrm{ml\ AgNO_3(test\ specimen)} - \mathrm{ml\ AgNO_3(blank)}] \times V \times F}{V_t \times m \times 1\,000} \tag{2}$$

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).

8.2 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, ½in milligrammes per litre;

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

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

8.3 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:

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

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 litress;

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

8.4 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:

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

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.

8.5 pH

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

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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

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 European 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) if any deviation from clauses 6 and 7;
4) date of testing;
5) general information relating to the test including reference to the methods used and leaching temperature and time;
6) events which may have affected the results;

NOTE 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

all individual values and the mean value for each of the measured components.

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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, $ZrOCl_2 \cdot 8H_2O$, 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 must be prepared each time 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 minimise their interference.

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Annex B
(informative)

General information related to the use of this standard

B.1 This standard has been developed to determine some parameters which may have an influence on stress corrosion cracking on austenitic stainless steel surfaces.

B.2 This standard gives test procedures to evaluate the trace quantities of some ions and the pH value of an aqueous extract of the product. Some of these ions may not be relevant to all types of insulating products. Some ions other than those determined by this standard may also be important.

B.3 When referring to this standard a limiting value of chloride may be used to show whether a particular thermal insulating product is suitable for application to austenitic stainless steel surfaces (if appropriate the limiting value may be given in the relevant product standard).

Alternatively the role of inhibitor ions (e.g. silicate and sodium) can be taken into account to balance a higher amount of chloride ions. Results obtained by use of this standard can not generally be combined with requirements established in current national standards because of different test specimen preparation.

B.4 A low chloride content in the thermal insulating product is no assurance for no stress corrosion cracking of austenitic stainless steel under insulation as a lot of other factors are also relevant.

Annex C
(informative)

Example of ion chromatography equipment

The following are examples of ion chromatograph equipment which are suitable for the measurement of trace quantities of water soluble chloride and fluoride.

EXAMPLE 1

- Dionex DX 100
- Column IonPac NG 1 Guard
- Column IonPac AS 4 A

EXAMPLE 2

- Dionex DX 100
- Column IonPac AS 10

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Annex D
(informative)

Example of borosilicate glass equipment to prepare leaching solutions for thermal insulating products that float

The main components are:

- Beaker, NW100, with flat flange with groove (for joint), wall equipped with two rows of 3 indentations each, situated at 120°, the two rows staggered for 30 °;
- O-ring, silicone rubber joint;
- lid, flat flange, with ground centre neck for reflux condenser and parallel side neck with GL screw thread for mercury contact thermometer or Pt100 thermometer;
- quick release clamp in stainless steel;
- perforated glass plate with stem (for easy manipulation) and 3 incisions at 120 °, fitting the indentations in the beaker wall (to prevent test specimen from floating)

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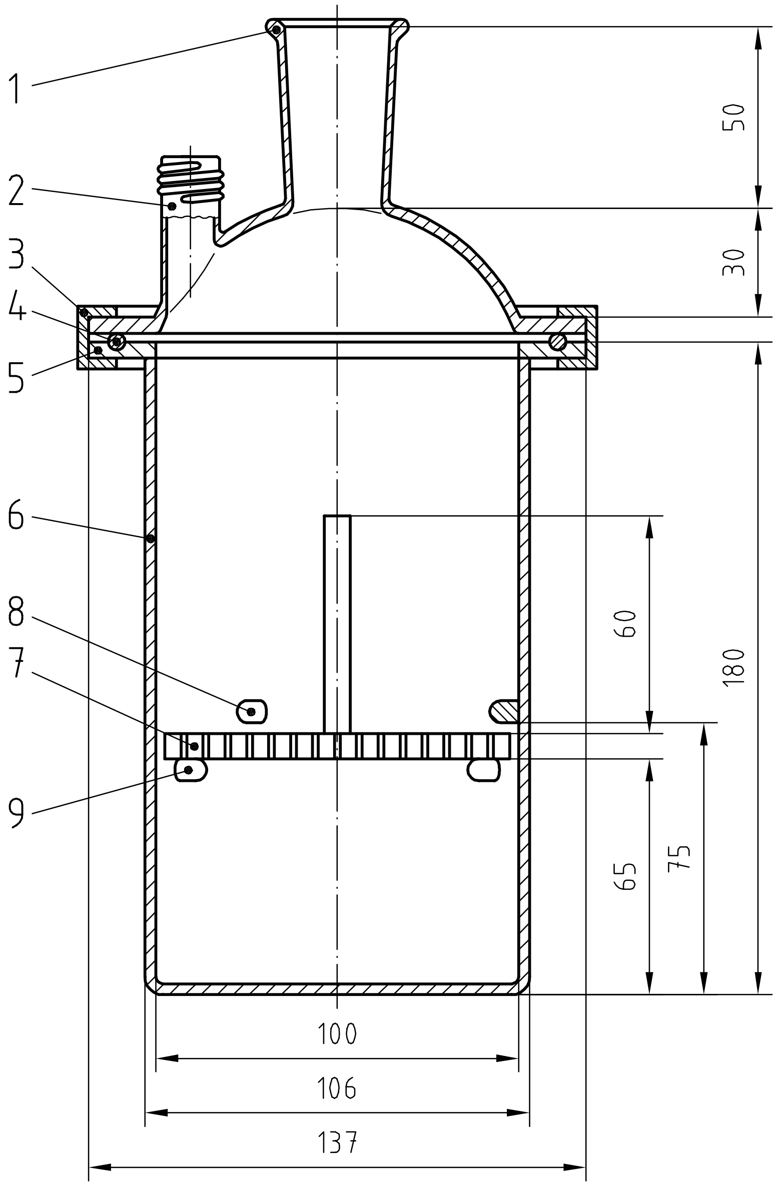
Dimensions in millimetres



Key

1 NS 29.2/32 Ground centre neck for reflux condenser
2 GL 18/8 screw thread parallel side neck for thermometer (mercury contact thermometer or Pt100 thermometer)
3 Stainless steel quick release clamp
4 O-ring (silicone rubber)
5 Flat flange
6 Beaker NW 100 with flat flange
7 Perforated glass plate, 7 mm thick Ø95 mm with 60 mm long stem
8 3 indentations at 120°
9 3 indentations at 120° (staggered 30°) to keep glass plate at ~ 65 mm from bottom

NOTE Glass plate has 3 incisions that fit the indentations at 120°

Figure D.1 - Example of borosilicate glass equipment to prepare leaching solutions for thermal insulating products that float

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