



SLOVENSKI STANDARD
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Tests for chemical properties of aggregates - Part 1: Chemical analysis

Prüfverfahren für chemische Eigenschaften von Gesteinskörnungen - Teil 1: Chemische Analyse

Essais pour déterminer les propriétés chimiques des granulats - Partie 1: Analyse chimique

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71.040.40	Kemijska analiza	Chemical analysis
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English version

Tests for chemical properties of aggregates - Part 1: Chemical analysis

Essais pour déterminer les propriétés chimiques des granulats - Partie 1: Analyse chimique

Prüfverfahren für chemische Eigenschaften von Gesteinskörnungen - Teil 1: Chemische Analyse

This European Standard was approved by CEN on 15 February 1998.

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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.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 154 "Aggregates", the secretariat of which is held by BSI.

This standard forms part of a series of tests for chemical properties of aggregates. Test methods for other properties of aggregates will be covered by the following European Standards:

EN 932	Tests for general properties of aggregates
EN 933	Tests for geometrical properties of aggregates
EN 1097	Tests for mechanical and physical properties of aggregates
EN 1367	Tests for thermal and weathering properties of aggregates

The other parts of EN 1744 will be:

Part 2	Determination of resistance to alkali reaction
Part 3	Water leaching test
Part 4	Determination of water susceptibility of fillers for bituminous mixtures

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of an identical text or by endorsement, at the latest by September 1998, and conflicting national standards shall be withdrawn at the latest by December 1999.

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 European Standard specifies procedures for the chemical analysis of aggregates. It specifies the reference procedures and, in certain cases, an alternative method which can be considered as giving equivalent results.

If other methods are used it is necessary to show that they give results equivalent to those given by the reference methods.

NOTE: In cases of dispute, only the reference procedures should be used.

Unless otherwise stated, the test methods specified in this European Standard may be used for factory production control, for audit tests or for type tests.

2 Normative references

This European Standard incorporates by dated or by 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 196-1:1993	Methods of testing cement Part 1: Determination of strength
EN 196-2:1987	Methods of testing cement Part 2: Chemical analysis of cement
EN 196-3:1987	Methods of testing cement Part 3: Determination of setting time and soundness
ENV 197-1:1992	Cement - composition, specifications and conformity criteria. Part 1: Common cements
EN 932-1:1996	Tests for general properties of aggregates Part 1: Methods for sampling
prEN 932-2	Tests for general properties of aggregates Part 2: Methods for reducing laboratory samples
prEN 932-5	Tests for general properties of aggregates Part 5: Common equipment and calibration
EN 933-2:1995	Tests for geometrical properties of aggregates Part 2: Determination of particle size distribution - Test sieves, nominal size of apertures
prEN 1015-4	Methods of test for mortar for masonry Part 4: Determination of consistence of fresh mortar (by plunger penetration)

prEN 1015-9	Methods of test for mortar for masonry Part 9: Determination of service life of fresh mortars
prEN 1015-11	Methods of test for mortar for masonry Part 11: Determination of flexural and compressive strength of hardened mortar
prEN 1097-6	Tests for mechanical and physical properties of aggregates Part 6: Determination of particle density and water absorption
ISO 384:1978	Laboratory glassware - Principles of design and construction of volumetric glassware.
ISO 648:1977	Laboratory glassware - One-mark pipettes
ISO 650:1977	Relative density 60/60 degrees F hydrometers for general purposes
ISO 1042:1983	Laboratory glassware - One-mark volumetric flasks
ISO 4788:1980	Laboratory glassware - Graduated measuring cylinders

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3 Definitions

For the purposes of this standard, the following definitions apply:

- 3.1 **test portion:** The sample used as a whole in a single test.
- 3.2 **test specimen:** When a test method requires more than one determination of a property, the test specimen is the sample used in a single determination.
- 3.3 **laboratory sample:** A reduced sample derived from a bulk sample for laboratory testing.
- 3.4 **constant mass:** Successive weighings after drying at least 1 h apart not differing by more than 0,1 %.

NOTE: In many cases constant mass can be achieved after a test portion has been dried for a pre-determined period in a specified oven at (110 ± 5) °C. Test laboratories can determine the time required to achieve constant mass for specific types and sizes of sample dependent upon the drying capacity of the oven used.

- 3.5 **batch:** A production quantity, a delivery quantity, a partial delivery quantity (railway wagon-load, lorry-load, ship's cargo) or a stockpile produced at one time under conditions that are presumed uniform.

NOTE: With a continuous process the quantity produced during an agreed period is treated as a batch.

- 3.6 **finer:** The particle size fraction of an aggregate which passes the 0,063 mm sieve.

4 Reagents

Unless otherwise stated, use only analytical grade reagents and demineralised water, or water of equivalent purity.

NOTE 1: Unless otherwise stated "%" means "% by mass".

NOTE 2: Where no tolerances are given for reagent volumes or masses, the values quoted are approximate. In such cases volumes delivered from measuring cylinders and indicated masses using the ordinary balances specified in 5.2.4 and 5.2.5 are considered sufficiently accurate for the purposes of this European Standard.

NOTE 3: Unless otherwise stated reagent solutions may be assumed to have long term stability.

NOTE 4: All chemicals should be treated as potential poisons with toxic properties and appropriate precautions taken before their use. Always take time to assess possible hazards before starting any procedures and constant attention should be maintained.

4.1 General requirements for densities

The following concentrated liquid reagents required by this standard shall have the following densities in grams per cubic centimetre at 20 °C:

Hydrochloric acid	: 1,18 to 1,19
Nitric acid	: 1,40 to 1,42
Sulfuric acid	: 1,84
Ammonium hydroxide	: 0,88 to 0,91

The degree of dilution shall be indicated as a volumetric sum.

NOTE 1: For example in 4.11.4, "hydrochloric acid (1+1)" means that 1 volume of concentrated hydrochloric acid is to be mixed with 1 volume of water.

NOTE 2: Ready for use solutions are allowed as an alternative.

4.2 Reagents for determination of water-soluble chloride salts using the Volhard method (clause 7).

4.2.1 Silver nitrate (AgNO_3) solution, 0,100 mol/l, prepared by drying about 20 g of silver nitrate for at least 1 h at a temperature of (110 ± 5) °C, allowing to cool in a desiccator and then weighing $(16,987 \pm 0,001)$ g of the dried silver nitrate, dissolving in water and diluting to 1 l in a volumetric flask (5.3.6). Store the solution in the amber-coloured glass reagent bottle (5.2.14) and protect from prolonged exposure to sunlight.

4.2.2 Thiocyanate (KSCN or NH_4SCN) solution, approximately 0,1 mol/l, prepared by dissolving 9,7 g of potassium thiocyanate or 7,6 g ammonium thiocyanate in water and diluting to 1 l in a volumetric flask.

Pipette 25 ml of silver nitrate solution (4.2.1) into a flask (5.3.5) and add 5 ml of nitric acid (4.2.3) and 1 ml of ammonium iron (III) sulfate indicator solution (4.2.5).

Add the thiocyanate solution from a burette (5.2.13) until the first permanent colour change occurs, that is from white opalescence to pale brown. Note the volume of thiocyanate solution added.

Calculate the concentration of the thiocyanate solution c_T , (in moles per litre), from the following equation:

$$c_T = 2,5/V_1$$

where:

V_1 is the volume of thiocyanate added (in millilitres).

Standardize the solution at weekly intervals or before use if the tests are carried out infrequently.

4.2.3 Nitric acid (HNO₃), approximately 6 mol/l, prepared by adding 100 ml of nitric acid (4.1) to 150 ml water, boiling the diluted acid in a fume cupboard (5.2.17) until it is colourless and allow to cool to room temperature.

4.2.4 Chloride free technical grade 3,5,5,-trimethylhexan-1-ol.

4.2.5 Ammonium iron (III) sulfate NH₄Fe(SO₄)₂·12H₂O indicator solution, prepared by adding 60 g of water to 50 g ammonium iron (III) sulfate, warming to dissolve, and adding 10 ml nitric acid (4.2.3).

Allow the solution to cool to room temperature and store in a glass bottle (5.2.15).

4.3 Reagents for potentiometric determination of water-soluble chloride salts (clause 8)

4.3.1 Silver nitrate (AgNO₃) solution, 0,01 mol/l prepared using the same procedure as specified in 4.2.1, but dissolving 1,699 g of dried silver nitrate in a 1 l volumetric flask (5.3.6).

4.3.2 Sodium chloride (NaCl) solution, 0,02 mol/l, prepared by drying about 2 g of sodium chloride at a temperature of (110 ± 5) °C for 1 h to 2 h, allowing to cool and then weighing (1,169 ± 0,001) g of the dried sodium chloride, dissolving in water and diluting to 1 l in a volumetric flask (5.3.6).

4.4 Reagent for factory production control determination of water-soluble chloride salts using the Mohr method (clause 9)

Potassium chromate (K₂CrO₄) solution, prepared by dissolving 10 g of potassium chromate in 100 ml water.

4.5 Reagents for determination of water-soluble sulfates (clause 10)

4.5.1 Hydrochloric acid (HCl) solution, made by adding 200 ml concentrated hydrochloric acid (relative density 1,18) to 800 ml water.

4.5.2 Barium chloride (BaCl₂) solution, made by dissolving 100 g of barium chloride (BaCl₂·2H₂O) in 1 l of water, and filtered through a medium grade filter paper before use.

4.6 Reagents for determination of total sulfur content (clause 11)

4.6.1 Bromine.

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4.6.2 Indicator methyl red (dissolve 20 mg methyl red powder in 50 ml of ethanol, then add 50 ml of water).

4.7 Reagents for determination of sulfide content (clause 13)

4.7.1 Lead acetate solution (dissolve approximately 0,2 g of lead acetate $\{Pb(CH_3COO)_2 \cdot 3H_2O\}$ in water and make up to 100 ml).

4.7.2 Ammoniacal zinc sulfate solution (dissolve 50 g of zinc sulfate $(ZnSO_4 \cdot 7H_2O)$ in 150 ml water and add 350 ml of concentrated ammonium hydroxide (NH_4OH)). Leave to stand for at least 24 h and filter through a medium grade filter paper.

4.7.3 Tin (II) chloride $(SnCl_2 \cdot 2H_2O)$.

4.7.4 Metallic chromium Cr powder form (**CAUTION : CARCINOGENIC**).

4.7.5 Standard potassium iodate solution containing 0,0167 mol/l; dissolve successively in freshly boiled and cooled water in a 1 l volumetric flask, $(3,6 \pm 0,1)$ g to the nearest 0,1 mg (**6.3**) of potassium iodate KIO_3 , dried at (110 ± 5) °C, two pellets (about 0,4 g) of sodium hydroxide $(NaOH)$ and 25 g of potassium iodide (KI) .

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Make up to the mark with freshly boiled and cooled water.

NOTE 1 : A trace of sodium hydroxide stabilises this solution for a considerable time; the solution should be discarded when it becomes discoloured.

The factor F of this solution is calculated from the following equation:

$$F = \frac{m_1}{3,5668}$$

where:

m_1 is the mass of the portion of potassium iodate.

NOTE 2 : If the sulfide content is low (less than 0,1 %), solutions ten times less concentrated should be used. They are prepared by pipetting 100 ml of the solutions (**4.7.5** and **4.7.6**) into 1 l volumetric flasks and making up to the mark with water.

4.7.6 Sodium thiosulfate solution approximately 0,1 mol/l.

Dissolve 24,82 g of sodium thiosulfate $(Na_2S_2O_3 \cdot 5H_2O)$ in water and make up to 1 l. Before each test series, determine the factor f of this solution as specified as follows.

Standardize the thiosulfate solution in one of the following ways.

a) Standardization carried out preferably in relation to the standard potassium iodate solution (**4.7.5**).

For this standardization, pipette 20 ml of the standard potassium iodate solution into a 500 ml conical flask and dilute with approximately 150 ml of water. Acidify with 25 ml of hydrochloric

acid (1 + 1) and titrate with the approximately 0,1 mol/l sodium thiosulfate solution to a pale yellow colour.

Then add 2 ml of the starch solution (4.7.7) and continue the titration until the colour changes from blue to colourless.

The factor f of this solution is calculated from the following equation:

$$f = \frac{20 \times 0,01667 \times 214,01 \times F}{3,5668 \times V_2} = 20 \frac{F}{V_2}$$

where:

F is the factor of the standard potassium iodate solution (4.7.5) expressed in moles per litre;

V_2 is the volume of the approximately 0,1 mol/l sodium thiosulfate solution used for the titration;

3,5668 g/l of potassium iodate corresponds to a solution with exactly 0,01667 mol/l of potassium iodate;

214,01 is the molecular mass of KIO_3 .

b) Standardization carried out in relation to a known quantity of potassium iodate.

For this standardization, place in a 500 ml conical flask (70 ± 5) mg of potassium iodate and dissolve in approximately 150 ml of water.

Add about 1 g of potassium iodide, acidify with 25 ml of hydrochloric acid (1 + 1) and titrate with the approximately 0,1 mol/l sodium thiosulfate solution until a pale yellow colour is obtained. Then add 2 ml of the starch solution (4.7.7) and titrate until the colour changes from blue to colourless.

The factor f of this solution is calculated from the following equation:

$$f = \frac{1\,000 \times m_2}{3,5668 \times V_3} = 280,3634 \frac{m_2}{V_3}$$

where:

m_2 is the mass of the portion of potassium iodate in grams;

V_3 is the volume of the approximately 0,1 mol/l sodium thiosulfate solution used for the titration;

3,5668 g/l of potassium iodate corresponds to a solution with exactly 0,01667 mol/l of potassium iodate.

4.7.7 Starch solution (to 1 g of starch (water soluble), add 1 g of potassium iodide KI, dissolve in water and make up to 100 ml).

4.8 Reagents for determination of lightweight contaminators (see 14.2)

4.8.1 Zinc chloride solution, obtained by dissolving 7 kg of $ZnCl_2$ in 3 l of water in order to obtain a saturated solution of density of $(1,98 \pm 0,02) \text{ g/cm}^3$ at $(20 \pm 3) \text{ }^\circ\text{C}$. The relative density of the solution, after cooling to room temperature, shall be checked using a suitable hydrometer (5.8.3).

NOTE: Zinc chloride solution is moderately irritating to skin and mucous membranes.

4.8.2 Sodium polytungstate solution (as an alternative to 4.8.1), prepared by dissolving $3Na_2WO_4 \cdot 9W_2O_3 \cdot H_2O$ crystals in water until the density of well stirred solution containing no undissolved crystals is $(1,98 \pm 0,02) \text{ g/cm}^3$ at $(20 \pm 3) \text{ }^\circ\text{C}$.

4.9 Reagents for determination of humus content (see 15.1)

4.9.1 Sodium hydroxide solution, a 3 % NaOH solution, obtained by dissolving 30 g of sodium hydroxide pellets in water, cooling to room temperature and diluting to 1 l in a volumetric flask.

4.9.2 Standard colour solution, prepared by dissolving 45,0 g $FeCl_3 \cdot 6H_2O$ and 5,50 g $CoCl_2 \cdot 6H_2O$ in 279,5 g of water with 1 ml concentrated HCl. This solution is stored in a glass bottle and is stable for at least 2 weeks.

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4.10 Reagents for determination of fulvo acid content (see 15.2)

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4.10.1 Hydrochloric acid (1 + 23).

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4.10.2 Tin (II) chloride solution. Dissolve 22,5 g $SnCl_2 \cdot 2H_2O$ in 1 l of hydrochloric acid (4.10.1). The quality of this solution will be retained for 2 weeks.

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4.11 Reagents for complexometric determination of free lime (see 18.1)

4.11.1 Ethanediol (Ethylene glycol), fresh, anhydrous.

4.11.2 Propan-2-ol (Isopropanol), anhydrous.

4.11.3 Filter paper pulp, in anhydrous ethanediol.

4.11.4 Hydrochloric acid diluted (1+1).

4.11.5 Triethanolamine.

4.11.6 m-Nitrophenol (0,1 g in 100 ml H_2O).

4.11.7 Sodium hydroxide solution 2 mol/l, obtained by dissolving 80 g of sodium hydroxide pellets in water, cooling to room temperature and diluting to 1 l in a volumetric flask.

4.11.8 Indicator, grind together with pestle and mortar 1 g of murexide (ammonium purpurate) and 100 g NaCl.

4.11.9 EDTA solution 1/112 mol/l, (3,3 ± 0,1) g of ethylenediaminetetra - acetic acid disodium salt, dried to constant mass at 80 °C, dissolved in water and made up to 1 l. This solution needs to be standardized against a solution of known calcium content (**4.11.10**).

4.11.10 Standard calcium solution (1 ml = 1 mg of calcium oxide). Dissolve (1,785 ± 0,001) g of pure calcium carbonate (**4.11.11**) dried at (110 ± 5) °C in a slight excess of (1 + 4) hydrochloric acid. Boil the solution to expel carbon dioxide, cover and cool to room temperature and dilute to 1 l with water in a volumetric flask (**5.3.6**).

NOTE: Commercially standardized solutions are available, e.g. (1,000 ± 0,002) g CaO/l.

4.11.11 Calcium carbonate (CaCO₃) precipitated grade, volumetric standard.

4.11.12 Soda-lime, granulated.

4.12 Reagent for conductimetric determination of free lime (see 18.2)

4.12.1 Ethanediol (**4.11.1**).

4.13 Reagents for acidimetric determination of free lime (see 18.3)

4.13.1 Ethyl acetoacetate, anhydrous grade.

4.13.2 2-methyl propan-1-ol, (isobutyl alcohol) anhydrous grade.

4.13.3 Thymol blue indicator (thymolsulfonephthalein).

4.13.4 Hydrochloric acid, (4.1).

4.13.5 Solvent solution, 450 ml of ethyl acetoacetate in 3 l of 2-methylpropan-1-ol.

4.13.6 Indicator (0,1 g of thymol blue indicator powder dissolved in 100 ml of 2-methylpropan-1-ol).

4.13.7 Hydrochloric acid solution approximately 0,2 mol/l.

To prepare this solution, make up 17 ml of hydrochloric acid (4.1) to 1 l with 2-methylpropan-1-ol.

To standardize this solution, weigh (100 ± 0,1) mg of calcium carbonate (**4.11.11**) in a crucible (**5.6.2**) and calcine for 1 h at 1000 °C. Extract the free lime and titrate in accordance with **18.3.3**.

Determine the *k* factor from the following equation:

$$k = \frac{56,08}{100,09} \times \frac{100}{V_4}$$

where:

V_4 is the volume of hydrochloric acid added (in millilitres);

k represents the number of milligrams of free CaO per millilitre of standardized hydrochloric acid solution.

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4.13.8 Sodium hydroxide on support granulated about 0,8 mm to 1,6 mm for elementary analysis.

4.14 Reagent for the determination of the expansion of steel slag (see 19.3)

4.14.1 Silicone oil.

4.14.2 Hydrochloric acid diluted (1 + 5).

5 Apparatus

5.1 General requirements

All apparatus shall comply with the general requirements of prEN 932-5.

Unless otherwise stated, all volumetric glassware shall be of class B accuracy as defined in ISO 384. Volumetric glassware of class A accuracy shall be used for audit tests and for type tests.

NOTE: Where no tolerances are specified for dimensions, the values quoted are approximate.

5.2 Apparatus for general purposes

5.2.1 Well ventilated oven, capable of being controlled to maintain a constant temperature in the range of 40 °C to 150 °C with a precision of ± 5 °C, equipped with a tray of non-corrodible material.

5.2.2 Electric muffle furnace capable of being controlled to maintain a constant temperature in the range of 800 °C to 1100 °C with a precision of ± 25 °C.

5.2.3 Crushing and grinding equipment to reduce aggregates to sizes so that they pass through sieves suitable for particular tests while producing a minimum of fines.

5.2.4 Balance, capable of weighing up to 10 kg, readable to the nearest 1 g.

5.2.5 Balance, capable of weighing up to 1 kg, readable to the nearest 0,01 g.

5.2.6 Analytical balance, capable of weighing up to 100 g, readable to the nearest 0,1 mg.

5.2.7 Hot plate with magnetic stirrer.

5.2.8 pH meter, readable to 0,1 pH units.

5.2.9 Beakers, conical flasks, funnels and filter paper.

5.2.10 Pipettes, 25 ml, 50 ml and 100 ml, complying with the requirements of ISO 648.

5.2.11 Graduated measuring cylinders, capacity 10 ml, 250 ml and 500 ml, complying with the requirements of ISO 4788.

5.2.12 Wash bottles, containing demineralised water.

5.2.13 Two burettes, 50 ml size, graduated to 0,1 ml.

5.2.14 Amber-coloured glass reagent bottles.

5.2.15 Plain glass reagent bottles.

5.2.16 Desiccators.

5.2.17 Fume cupboard.

5.3 Additional apparatus required for determination of water-soluble chloride salts following Volhard (see clause 7)

5.3.1 Test sieve, 16 mm square hole perforated plate complying with the requirements of EN 933-2.

5.3.2 Two glass, plastic or metal bottles, wide-mouthed, with well fitting stoppers.

NOTE: The bottles should be approximately 5 l capacity when testing coarse aggregates or lightweight aggregates or 2 l when testing fine aggregates.

5.3.3 Mechanical shaker or roller, to take the extraction bottles (5.3.2).

5.3.4 Two filter funnels, of approximately 100 mm diameter with medium and fine grade filter papers of a diameter appropriate to the size of the funnel.

5.3.5 Stoppered conical flasks, 100 ml and 250 ml capacity.

5.3.6 Two volumetric flasks, capacity of 1 l, complying with the requirements of ISO 1042.

5.4 Additional apparatus required for potentiometric determination of water-soluble chloride salts (see clause 8)

5.4.1 A potentiometric titrator suitable for the determination of chloride ion concentration with an electrode system consisting of:

- a) measuring electrode - either a silver electrode (preferably chloridised) or a chloride ion - selective electrode.
- b) reference electrode - either mercurous sulfate or a double junction silver/silver chloride with chloride free electrolyte in the outer chamber.

5.5 Additional apparatus required for factory production control determination of water soluble chloride salts following Mohr (see clause 9)

5.5.1 Two wide-mouthed plastic bottles of 1 l, with stoppers.

5.6 Additional apparatus required for determination of water-soluble sulfates (see clause 10)

5.6.1 Sintered silica filtering crucibles, porosity grade 4, approximately 35 mm in diameter and 40 mm in height.

5.6.2 Ignition crucibles, as alternative to 5.6.1, approximately 35 mm in diameter and 40 mm in height and capable of maintaining a constant mass when heated to 1100 °C.