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Methods of test for masonry units - Part 5: Determination of the active soluble salts content of clay masonry units

Prüfverfahren für Mauersteine - Teil 5: Bestimmung des Gehalts an aktiven löslichen Salzen von Mauerziegeln

Méthodes d'essai des éléments de maçonnerie - Partie 5: Détermination de la teneur en sels solubles actifs des éléments de maçonnerie en terre cuite

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EUROPEAN STANDARD

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (EN 772-5:2016) has been prepared by Technical Committee CEN/TC 125 “Masonry”, the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by September 2016, and conflicting national standards shall be withdrawn at the latest by December 2017.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 772-5:2001.

The crushing procedure in 7.2 has been amended so that the sample, after reducing to particles of not greater than approximately 1 mm in size, is dried in a ventilated oven to constant mass prior to further grinding and sieving.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

EN 772-5:2016 (E)**1 Scope**

This European Standard specifies a method for determining the active soluble salts content of clay masonry units.

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.

EN 771-1, *Specification for masonry units — Part 1: Clay masonry units*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

3 Principle

The method adopted is based on water extraction from a crushed representative sample of the clay masonry units, and determines the amounts of soluble magnesium, sodium and potassium ions, released under the test conditions, which may be correlated with the potentially damaging effect of salts of those ions on cementitious mortars in certain circumstances, or even on the units themselves. These salts are known as «active» soluble salts in EN 771-1.

4 Symbols

M_{Mg}	is the number of milligrams of Mg equivalent to 1 ml of EDTA
x, y	is the volume of EDTA titrated, in millilitres (ml)
C_1	is the lower reference sample concentration, in percentage (%)
C_2	is the higher reference sample, concentration, in percentage (%)
C_x	is the sample concentration, in percentage (%)
E_1	is the measured signal for the lower reference sample concentration C_1
E_2	is the measured signal for the higher reference sample, concentration C_2
E_x	is the measured signal for sample
d	is the dilution factor

5 Materials**5.1 For all methods**

Distilled or deionized water for extraction of active soluble salts from the sample, and for preparation of analytical test solutions.

Hydrochloric acid (relative density 1,18).

All chemicals shall be of analytical reagent grade.

5.2 Instrumental method Atomic Absorption Spectroscopy (AAS) and flame photometry

Hydrated Lanthanum chloride $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$, 210 g/l Solution

Caesium Chloride CsCl, 63 g/l Solution

5.3 Instrumental method Atomic Absorption Spectroscopy (AAS) and flame photometry or inductively coupled plasma spectrometry (ICP)

High purity standard solutions

5.4 EDTA method (alternative)

Magnesium metal

Ethylenediamine tetra acetic acid (EDTA) Indicators: Calcein
 Methyl thymol blue complexone

Potassium hydroxide

Ammonia solution (relative density 0,88)

Potassium nitrate

6 Apparatus

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6.1 **Test sieves**, complying with the requirements of ISO 3310-1, ISO 3310-2
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6.2 **Mortar** or other suitable grinding equipment capable of producing particles to pass a 150 μm test sieve.
 [SIST EN 772-5:2016](https://standards.itih.ai/catalog/standards/sist/778e0b7b-3881-47ce-aa80-1e77f672053/sist-en-772-5-2016)

6.3 **Polyethylene bottle or Conical flask**, typically with a capacity 500 ml
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6.4 **Weighing instrument**, readable to the nearest 0,1 mg

6.5 **Burette**, typically 10 ml

6.6 **Pipette**, typically 10 ml, 50 ml

6.7 **Graduated flask**, typically 1 l

6.8 **Volumetric flask**, typically 100 ml

6.9 **One dimensional horizontal shaking equipment**, capable of oscillating at $(120 \pm 5) \text{ min}^{-1}$ having a horizontal movement of 20 mm, or a rotary shaker capable of revolving at 30 ± 3 revolutions per minute.

6.10 **Polyethylene bottle**, typically with a capacity of between 1 l and 1,5 l.

6.11 **Ventilated oven**, capable of maintaining a temperature of $(105 \pm 5) ^\circ\text{C}$

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7 Preparation of sample

7.1 Sampling

The method of sampling shall be in accordance with EN 771-1. The minimum number of specimens for the determination of the active soluble salts content shall be six and shall comprise whole units but a larger number may be specified in the product specification, in which case that larger number shall be used. The specimens shall be sampled in such a manner that they have not been exposed to water that would lead to leaching of soluble salts from the units.

From the bulk sample of units a representative sample of 50 g to 250 g of material, ground to pass a 150 μm test sieve (6.1), is prepared, using the crushing method described in 7.2.

7.2 Crushing

Crush each unit to give lumps not greater than 10 mm. Obtain from each crushed specimen a representative subsample of at least 50 g, either by coning and quartering or using a suitable mechanical sampling device. Mix this material and crush it to give particles not greater than approximately 1 mm. Reduce the prepared sample to 50 g to 250 g using the same splitting method.

Dry the crushed sample to constant mass in a ventilated oven at a temperature of $(105 \pm 5) ^\circ\text{C}$. Before weighing, the sample shall be cooled to room temperature in a desiccator.

Constant mass is achieved, when the loss in mass between two successive determinations conducted at an interval of 24 h during the drying, process does not exceed 0,2 % of the total mass.

Finally grind the complete sample such that 95 % will pass a 150 μm test sieve (6.1) complying with ISO 3310-1 or ISO 3310-2, using a mortar (6.2) or other suitable grinding equipment.

8 Extraction procedure

Weigh $20 \text{ g} \pm 0,05 \text{ g}$ of the sample and transfer it to a 500 ml polyethylene bottle, or a 500 ml conical flask (6.3) when using horizontal shaking equipment (6.9).

Add 200 ml of distilled or deionised water at room temperature, close the bottle with a screw-on polyethylene top and shake the bottle for $60 \text{ min} \pm 2 \text{ min}$, using a one dimensional horizontal shaking equipment at $(120 \pm 5) \text{ min}^{-1}$ with a horizontal movement of 20 mm, or a rotary shaker (6.9) revolving at $30 \text{ min}^{-1} \pm 3 \text{ min}^{-1}$.

Within $15 \text{ min} \pm 1 \text{ min}$ of completing the extraction filter the suspended sample using an ashless blue ribbon filter paper or equivalent and collect the filtrate in a clean dry flask (6.10). Do not wash the residue on the filter. Alternatively use a centrifuge. It is essential that the filtrate shall be clear.

9 Determinations of cations by instrumental techniques

9.1 General

Determine the metal ion content using an established method, e.g. inductively coupled plasma spectrometry, atomic absorption spectroscopy, or flame photometry. Alternatively determine and calculate the magnesium content following the procedure described in Clause 10.

9.2 Atomic absorption spectroscopy method (AAS) and flame photometry

9.2.1 Sample preparation

Pipette (6.6) a 50 ml aliquot of the soluble salts extract into a 100 ml volumetric flask (6.8) add 4 ml of hydrochloric acid (1 + 1 volume) and 4 ml of lanthanum chloride. If Na and K are to be measured by AAS, add 4 ml of caesium chloride. Fill to the mark with water and mix.

9.2.2 Calibration: preparation of the reference series

Either:

- a) use dilutions of commercially available standard solutions, magnesium 1 000 μ /g, potassium 1 000 μ /g, sodium 1 000 μ /g or,
- b) use a multi-element solution for example:
 - 1) weigh the following substances up to nearest 0,1 mg;
 - 2) 1 000 mg magnesium metal;
 - 3) 1 767,3 mg potassium carbonate $K_2 CO_3$ dried to $105\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$;
 - 4) 2 305,1 mg sodium carbonate $Na_2 CO_3$ dried to $105\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$;
 - 5) add 50 ml of hydrochloric acid (1+1 volume) wait till the reaction is completed and warm up carefully on a heating plate until complete dissolution; complete to the mark in a 1 000 ml graduated flask (6.7).

All chemicals shall be of analytical reagent grade.

Then prepare a series of reference solutions as suggested in Table 1.

Table 1 — Suggested reference solution series

Solution	Volume of standard solutions or multi-element solution each other diluted 1/10	Add Hydrochloric acid (1 + 1 volume) and Lanthanum chloride and Caesium chloride ^a	Fill to 100 ml with water and mix	Concentration in Mg, K, Na% of the mass of the original sample
0	0 ml	4 ml of each		0
1	1 ml			0,002
2	2 ml			0,004
3	5 ml			0,010
4	10 ml			0,020
5	15 ml			0,030

NOTE If the concentration is higher than 0,03 % pipette (6.6) a low aliquot of the solution salts extract and multiply by dilution factor d (e.g. aliquot 25 ml, dilution factor d = 2).

^a Only in case where Na, K are measured by AAS.

9.2.3 Spectroscopic lines and parameters for AAS and flame photometry analysis

The data to be used for the analysis may be as given in Table 2.

Table 2 — Suggested spectroscopic lines and parameters for AAS and flame photometry analysis

Type analysis	Element	nm	Type of flame
AAS	Mg	285,2	air/acetylene
flame emission or AAS	K	766,5	air/acetylene
flame emission or AAS	Na	589,0	air/acetylene