

Masonry units — Methods of test

Part 3:

Determination of the active soluble salts content of clay masonry units

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TECHNICAL COMMITTEE REPRESENTATION

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Coast Clay Works Ltd
Consumer Information Network
University of Nairobi
Kenya Industrial Research & Development Institute
Architectural Association of Kenya
M&O Consulting Engineers
Kenya Association of Manufacturers,
Kenya National Federation of Jua Kali Association
Lake Basin Development Authority
Kenya Bureau of Standards — Secretariat

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Masonry units — Methods of test

Part 3:

Determination of the active soluble salts content of clay masonry units

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Foreword

This Kenya Standard was prepared by the Clay and Clay Products Technical Committee under the guidance of the Standards Projects Committee and in accordance with the procedures of the Kenya Bureau of Standards.

During the development of this standard, reference was made to the following documents:

BS EN 772-5:2016 Methods of test for masonry units - Part 1: Determination of the active soluble salts content of clay masonry units.

Acknowledgement is hereby made for the assistance received from these sources.

Masonry units — Methods of test

Part 3:

Determination of the active soluble salts content of clay masonry units

1. Scope

This Kenyan 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.

DKS 2801 -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 DKS 2801 -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

6.1. Test sieves, complying with the requirements of ISO 3310-1, ISO 3310-2

6.2. Mortar or other suitable grinding equipment capable of producing particles to pass a 150 μm test sieve.

6.3. Polyethylene bottle or Conical flask, typically with a capacity 500 ml

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 $(105 \pm 5) ^\circ\text{C}$

7. Preparation of sample

7.1. Sampling

The method of sampling shall be in accordance with DKS 2801-1. 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 1000 µg/ml, potassium 1000 µg/ml, sodium 1000 µg/ml 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_2CO_3 dried to $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$;
- 4) 2 305,1 mg sodium carbonate Na_2CO_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 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).				
40.				
^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

9.3. Inductively coupled plasma spectrometry method (ICP)

9.3.1. General

The sample preparation (see 9.2.1) and the preparation of reference series (see 9.2.2) shall be as that for the atomic absorption spectroscopy method, but without any addition of lanthanum chloride or caesium chloride.

9.3.2. Spectroscopic lines for ICP analysis

The data to be used for the analysis can be as given in Table 3.

Table 3 - Suggested spectroscopic lines for ICP analysis

Element	nm
Mg	279,553
	285,213
K	766,491
	769,900
Na	588,995
	589,592

9.4. Calculation of the results

Construct calibration on the basis of measurements taking into account the directions given by the instrument manufacturers.

For example:

E_1 measured signal for the lower reference sample concentration C_1

E_2 measured signal for the higher reference sample, concentration C_2

E_x measured signal for sample

$$C_{x \text{ sample}} = C_1 + \frac{(C_2 - C_1)(E_x - E_1)}{(E_2 - E_1)} \cdot d$$

where

C_1 is the lower reference sample concentration, in percentage (%)

C₂ is the higher reference sample, concentration, in percentage (%)

C_x is the sample concentration, in percentage (%)

E₁ is the measured signal for the lower reference sample concentration C₁

E₂ is the measured signal for the higher reference sample, concentration C₂

E_x is the measured signal for sample

d is the dilution factor

If the concentration of the sample is, greater than 0,001 % then subtract the concentration value found for the blank solution from the calculated concentration of the sample.

10. Alternative method of determination of the content of active soluble salts

10.1. Preparations of reagents for magnesium determination

10.1.1. Magnesium test solution (1,0 mg Mg/ml)

Dissolve 1,000 g of magnesium metal in a slight excess of hydrochloric acid solution consisting of one part hydrochloric acid (relative density 1,18) to three parts distilled or deionized water, and dilute to 1 l with distilled or deionized water in the graduated flask (6.7). Before weighing, etch the metal ribbon or foil in diluted hydrochloric acid, wash it with water and then dry it with alcohol followed by ether. Adjust the mass with scissors.

10.1.2. Preparation of EDTA test solution (0,5 %)

Dissolve 5 g of ethylenediamine tetra acetic acid (EDTA) (disodium salt, dihydrated) in warm distilled or deionized water, filter if necessary, cool and dilute it to 1 l. Store in a polyethylene bottle (6.10). Standardize against the magnesium test solution described in 10.1.1 using methyl thymol blue complexone, where M_{Mg} is the number of milligrams of Mg equivalent to 1 ml of EDTA

10.2. First titration (calcium plus magnesium)

Pipette (6.6) a 10 ml aliquot of the soluble salts extract into a 500 ml conical flask. Add 20 drops of hydrochloric acid (relative density 1,18), followed by 10 ml of ammonia solution (relative density 0,88) and dilute with distilled or deionized water to about 200 ml. Add about 0,04 g of methyl blue complexone indicator, prepared by grinding together 0,2 g of methyl thymol blue complexone with 20 g of potassium nitrate. Titrate with EDTA test solution, prepared as described in 10.1.2 from a 10 ml burette (6.5), the colour change being from blue to colourless. Measure the volume titrated, where the volume of EDTA used in the titration is denoted by X.

10.3. Second titration (calcium)

Pipette (6.6) a 10 ml aliquot of the soluble salts extract a 500 ml conical flask. Add 20 drops of hydrochloric acid (relative density 1,18), followed by 10 ml of potassium hydroxide solution (approximately 250 g/l), and dilute to about 200 ml with distilled or deionized water. Add about 0,015 g of calcein indicator prepared by grinding together 0,1 g of calcein with 10 g of potassium chloride. Titrate with EDTA test solution, prepared as described in 10.1.2 from a 10 ml burette (6.5), the colour change being from fluorescent green to pink. Measure the volume titrated, where the volume of EDTA used in the titration is denoted by X.

10.4. Magnesium content

Calculate the mass of water soluble magnesium as a percentage of the mass of the original sample from the formula:

$$Mg = \frac{(y - x) M_{mg}}{10} \text{ in percentage \%}$$

Where y is the volume of the EDTA used in the titration described in 10.2 and x is the volume of EDTA described in 10.3.

10.5. Determination of sodium and potassium

Compare a portion of the soluble salts extract with test solutions containing 5 µg/ml by mass of sodium and 10 µg/ml by mass of potassium in a flame photometer or by atomic absorption spectroscopy. Calculate the sodium and potassium contents by reference to previously prepared calibration graphs.

11. Expression of results

Report the sum of sodium and potassium contents to the nearest 0,01 % and the magnesium to the nearest 0,01 %.

12. Test report

The test report shall contain the following information:

- a) the name, title and date of issue of this European Standard;
- b) the name of the organization that carried out the sampling and the method used;
- c) the date of testing;
- d) the type, origin and designation of the masonry unit by reference to DKS 2801-1;
- e) the date of delivery of the specimens;
- f) the number of specimens in the sample;
- g) the method of analysis, detailing whether chemical or alternative instrumental methods were used;
- h) the active soluble salts content of the sample, expressed as a percentage to the nearest 0,001 % for the ions of magnesium, sodium, and potassium;
- i) express the sum of sodium and potassium contents to the nearest 0,01 % and the magnesium to the nearest 0,01 %;
- j) Remarks, if any.