



BSI Standards Publication

Conservation of cultural heritage — Extraction and determination of soluble salts in natural stone and related materials used in and from cultural heritage

National foreword

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Conservation of cultural heritage - Extraction and determination of soluble salts in natural stone and related materials used in and from cultural heritage

Conservation du patrimoine culturel - Extraction et détermination des sels solubles dans la pierre naturelle et les matériaux associés utilisés dans le patrimoine culturel

Erhaltung des kulturellen Erbes - Auflösung und Bestimmung von löslichen Salzen in Naturstein und artverwandten Materialien des kulturellen Erbes

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Contents		Page
Foreword.....		3
Introduction		4
1	Scope	5
2	Normative references	5
3	Terms and definitions	5
4	Principle.....	6
5	Reagents, materials and analytical equipment.....	6
6	Procedure	6
6.1	Sampling.....	6
6.2	Analysis procedure.....	6
6.2.1	General.....	6
6.2.2	Conductivity measurements.....	7
6.2.3	Ions measurement	7
7	Expression of results	7
7.1	Specific conductivity, γ ($\mu\text{S} \cdot \text{cm}^{-1}$)	7
7.2	Individual ions.....	7
8	Test report	8
Bibliography.....		9

Foreword

This document (EN 16455:2014) has been prepared by Technical Committee CEN/TC 346 "Conservation of cultural heritage", the secretariat of which is held by UNI.

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of an identical text or by endorsement, at the latest by March 2015 and conflicting national standards shall be withdrawn at the latest by March 2015.

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Introduction

Soluble salts are often present in stones and other porous inorganic building materials as alteration products of chemical or biological origin. They can originate from surface deposition of environmental pollutants or from water capillary transport from other external sources and also from materials themselves or from conservation interventions.

Soluble salts present in *porous inorganic materials* can, (depending on material properties, environmental conditions or possible treatments the surrounding conditions), initiate physical-chemical degradation processes.

This standard describes a procedure to extract soluble salts present in porous inorganic materials to determine the ions and to estimate the content of soluble salts.

There are several test methods for the analysis of salts, for example:

- a) recognition of salt crystals by microscopy;
- b) qualitative chemical reactions. These make it possible to identify the type of *anions and cations* present in a solution, after dissolution of salts in water (i.e. sulphates, nitrates, chlorides);
- c) semiquantitative tests. These are mostly based on colourimetric reactions that lead to the formation of coloured compounds formed by each *anion* and its specific reagent;
- d) X-ray diffraction, which provides information on the nature of a salt (in *crystalline form*) identifying the mineralogical composition of a compound;
- e) spectroscopic techniques like flame photometry, AAS (Atomic Absorption Spectrometry), ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry) for cations;
- f) ion chromatography which makes it possible to identify and quantify separately anions and cations.

1 Scope

This European Standard provides a methodology for the qualitative and quantitative analysis of anions and cations obtained by dissolution of soluble salts present in either natural stone or other porous inorganic materials constituting cultural heritage and in materials and products used for their conservation. The methodology requires samples to have been taken from the cultural property.

The main ions considered in this standard are:

Cl^- , NO_2^- , NO_3^- , SO_4^{2-} , Na^+ , K^+ , NH_4^+ , Ca^{2+} , Mg^{2+}

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15898:2011, *Conservation of cultural property — Main general terms and definitions*

EN 16085:2012, *Conservation of Cultural property — Methodology for sampling from materials of cultural property — General rules*

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 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15898:2011 and EN 16085:2012 and the following apply.

3.1

soluble salts

salts that readily dissolve in a solvent such as water in order to form a solution; the solubility is dependent on the salt and the temperature of water

3.2

conductivity

measure of the ability of water to conduct an electrical current; it is highly dependent on the amount of dissolved solids (such as salt) in the water

3.3

specific conductivity

conductivity of a solution measured between two electrodes 1 cm² in area and 1 cm apart

Note 1 to entry: The units are $\mu\text{S} \cdot \text{cm}^{-1}$.

3.4

porous inorganic material

material including natural stones e.g. sandstone limestone and marble; as well as artificial materials such as mortar, plaster brick and other materials

4 Principle

Soluble salts are extracted from a powdered solid sub-sample by dissolution in a fixed volume of ultra-pure water. The content of soluble salts in the sample can be determined by measurement of conductivity and qualitative and quantitative ion analysis after filtration.

5 Reagents, materials and analytical equipment

- 5.1 Ultra pure water (specific conductivity $\leq 1 \mu\text{S}\cdot\text{cm}^{-1}$)
- 5.2 Anion and cation reference solutions in the range of the expected results
- 5.3 Common laboratory glassware or plasticware
- 5.4 Pestle and agate mortar
- 5.5 Series of sieves in accordance with ISO 3310-1 or ISO 3310-2
- 5.6 0,45 μm filter
- 5.7 Magnetic stirrer
- 5.8 Flask shaker
- 5.9 Temperature controlled oven capable of maintaining a temperature at $(50 \pm 5) ^\circ\text{C}$
- 5.10 A balance capable of weighing to $\pm 0,01 \text{ mg}$
- 5.11 A conductivity meter capable of measuring to $\leq 1 \mu\text{S}\cdot\text{cm}^{-1}$
- 5.12 Instrument for the analysis of anions and cations

NOTE Ion Chromatography is the most widely applied technique.

6 Procedure

6.1 Sampling

General rules for sampling are described in EN 16085:2012. In all cases the location, the number and the amount of the samples used should be documented in the final report. During the sampling, environmental temperature ($^\circ\text{C}$) and relative humidity (%) shall be measured.

6.2 Analysis procedure

6.2.1 General

An appropriate amount (at least $100 \text{ mg} \pm 5 \text{ mg}$) of the sample shall be powdered until the powder grain size is less than 0,106 mm (140 mesh). The powdered sub-sample has to be dried in an oven at a temperature of $(50 \pm 5) ^\circ\text{C}$ for $(24 \pm 1) \text{ h}$ or until a constant mass has been reached. Constant mass is assumed to have been attained when the difference between two weights at an interval of $(24 \pm 2) \text{ h}$ is less than 0,1 % of the sub-sample. In a few cases, the available amount may not be sufficient to allow a correct quantification. The concentrations will be considered as a semiquantitative evaluation.

An amount, (P), $(100 \pm 5) \text{ mg}$ of the powdered and dried sub-sample has to be weighed with an accuracy of 0,1 mg and transferred quantitatively to a 100,0 mL (V) volumetric flask. Ultra pure water with known specific

conductivity (γ_b) has to be added to mark on the volumetric flask. The flask has to be sealed and gently shaken for $(24,0 \pm 0,2)$ h at room temperature (25 ± 5) °C.

NOTE $(72,0 \pm 0,2)$ h may be required for complete or near complete of some soluble salts.

The resulting mixture shall be allowed to stand (minimum 2 h) until the insoluble material settles.

6.2.2 Conductivity measurements

Prior to proceed to determine quantitatively the ions, to estimate the total content of soluble salts¹⁾ the specific conductivity of the clear solution (γ_a) shall be measured and compared with the conductivity of ultra pure water (γ_b).

6.2.3 Ions measurement

The solution from 6.2 is then filtered using a $0,45\mu\text{m}$ filter, and after calibration of the measurement system a portion of filtrate shall be collected and used to determine the concentration (C), in mg/L, of each individual ion.

7 Expression of results

7.1 Specific conductivity, γ ($\mu\text{S} \cdot \text{cm}^{-1}$)

$$\gamma = (\gamma_a - \gamma_b) \cdot \frac{100}{P}$$

where

γ_a is the specific conductivity of the sample solution ($\mu\text{S} \cdot \text{cm}^{-1}$);

γ_b is the specific conductivity of the pure water ($\mu\text{S} \cdot \text{cm}^{-1}$);

100 is the "reference mass" of 100 mg;

P is the actual mass of the solid powdered sample (mg).

7.2 Individual ions

Results of the ions shall be recorded as:

Percentage of the dry mass of the sample, $I\%$

$$I\% = \left[(C) \cdot \left(\frac{V}{F} \right) \cdot \left(\frac{1}{P} \right) \right] \cdot 100$$

where

I is the mass percentage [mg/mg] % of a single anion or cation analysed (Cl^- , ..., Ca^{2+});

C is the concentration in mg/L of a single ion in solution;

¹⁾ In the case of mortar samples and certain types of stone, e.g. gypsum rock, the results are not always reliable because of the possible dissolution or partial dissolution of components which may affect the conductivity.

- V is the water extraction volume (100 ml);
 F is factor for conversion from ml to L, $F = 1\,000$ ml/L;
 P is the actual mass of the solid powdered sample (mg).

$$\frac{\text{meq}}{\text{g}} = \left[\frac{(I\%)}{100} \cdot \left(\frac{1}{ew} \right) \right] \cdot 1000$$

where

ew is the equivalent weight of each single ion.

8 Test report

The report shall contain the following information:

- a) unique identification number of the report;
- b) the name, number, and date of issue of this European Standard;
- c) the name and address of the laboratory and the address where the test was carried out if different;
- d) the name and the address of the client (if appropriate);
- e) the nature of the sample, e.g. solid, powder, etc.;
- f) the date of delivery of the sample or of the specimens;
- g) the date when the specimens were prepared (if relevant) and the date of testing;
- h) the analytical method used, the limits of detection, and the measurement accuracy;
- i) the conductivity ($\mu\text{S} \cdot \text{cm}^{-1}$) and the ion content (meq/g);
- j) the stirring time in hours;
- k) any deviation from this standard and the reason for such deviation;
- l) remarks.

Bibliography

- [1] UNI 11087:2003 *Beni culturali - Materiali lapidei naturali ed artificiali - Determinazione del contenuto di sali solubili*

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