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Conservation of Cultural Heritage — Guidelines to characterize natural stone used in cultural heritage

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee B/560, Conservation of tangible cultural heritage.

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

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Erhaltung des kulturellen Erbes - Leitfaden zur Charakterisierung von Naturstein in der Denkmalpflege

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Foreword

This document (EN 16515:2015) 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of an identical text or by endorsement, at the latest by October 2015 and conflicting national standards shall be withdrawn at the latest by October 2015.

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Introduction

The characterization of a stone is an essential step for a conservation plan, in order to decide on appropriate remedial interventions, to achieve better working practices, and technologies for conservation interventions including stone replacements. Therefore, it is important that it is achieved through a consistent and uniform methodology.

This document provides cultural heritage professionals with a guidance for a common methodology for the characterization of stones used in cultural heritage. This information is used to define nature (petrography or mineralogy and texture) and properties (chemical, physical and mechanical) of stone.

The characterization of stones used in cultural heritage should be carried out and interpreted by professionals experienced in the field of materials science and/or conservation/restoration.

1 Scope

This European Standard specifies a methodology for the characterization of sound or deteriorated stones by using the most appropriate analytical techniques on samples taken from the object.

This European Standard contains guidelines for the selection of methods to determine mineralogical, textural, physical, chemical and mechanical properties of natural stone used in cultural heritage monuments and objects. This information is used to define rock typology and to evaluate the stone's condition with respect to its conservation as well as for understanding of deterioration processes of natural stone. Where possible existing standards are referred to and guidance provided where different specimens are required and additional methods used. The methods described are generally destructive, however, non-destructive (NDT) methods are always preferable to methods with a minimum of destruction and those are always preferable to destructive methods.

Methods used for stone analysis can vary depending upon the objectives of the work. All investigation and analysis need be proportional to the significance of the building or artefact being investigated, its condition and the likely level of intervention. This European Standard will be used to determine the kind, extent, and objectives of the examination to be made.

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 1926, *Natural stone test methods — Determination of uniaxial compressive strength*

EN 1936, *Natural stone test methods — Determination of real density and apparent density, and of total and open porosity*

EN 12372, *Natural stone test methods — Determination of flexural strength under concentrated load*

EN 13755, *Natural stone test methods — Determination of water absorption at atmospheric pressure*

EN 14146, *Natural stone test methods — Determination of the dynamic modulus of elasticity (by measuring the fundamental resonance frequency)*

EN 14579, *Natural stone test methods — Determination of sound speed propagation*

EN 14580, *Natural stone test methods — Determination of static elastic modulus*

EN 15801, *Conservation of cultural property — Test methods — Determination of water absorption by capillarity*

EN 15803, *Conservation of cultural property — Test methods — Determination of water vapour permeability (δp)*

EN 15886, *Conservation of cultural property — Test methods — Colour measurement of surfaces*

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

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

EN 16322, *Conservation of Cultural Heritage —Test methods — Determination of drying properties*

EN 16455, *Conservation of cultural heritage — Dissolution and determination of soluble salts in natural stone and related materials used in cultural heritage*

3 Terms and definitions

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

3.1
investigation
gathering of information necessary for a conservation decision making process

3.2
sample
number of specimens of material, ideally representative, removed from the cultural property for scientific investigation

[SOURCE: EN 16085, modified]

3.3
sampling
process of removing a sample

3.4
specimen
single individual piece of stone forming one portion of a sample

3.5
sound material
stone showing an un-altered state under visual observation with the naked eye

3.6
deteriorated material
stone showing evidence of alteration under visual observation with the naked eye

3.7
analysis
investigation of natural stone which supplies important information to establish its nature and properties

3.8
petrography
description of the mineral content and the textural relationships of rock, based on observation of hand specimen and thin microscopic section

4 Preliminary operations

4.1 Initial survey

An initial survey of the object together with the objectives of the stone characterization and the description of the context are essential for the selection of the analytical methodology that is to be followed. Where the object is part of the immovable heritage a condition survey should be undertaken in accordance with EN 16096 or if the object is part of the moveable heritage a condition report should be made in accordance with EN 16095.

The condition report or survey should be supplemented by sufficient information to allow a preliminary assessment of the stone and its condition. This preliminary assessment should include general stone type, the macroscopic characteristics, such as colour and texture, structural aspects and apparent condition.

4.2 Sampling

Sampling shall be undertaken in accordance with EN 16085. In all cases, the location, the orientation, the number, the amount, the size and the shape of the specimens used should be documented in the test report.

In accordance with the initial survey, it should be stated whether the stone specimen to be characterized is derived from sound or deteriorated part of the stone.

5 Methodology to characterize sound and deteriorated material

5.1 Visual examination of specimens

A visual examination of the specimens is undertaken. When specimens are examined using a stereo-microscope this should be in accordance with EN 12407:2007, Clause 6. In the case of natural stone used in cultural heritage objects this enables an initial assessment of their condition, colour range or overall colour, porosity, grain, weathering patterns, along with other features such as cracks, veins, voids, fillings, bedding, cleavage, macro-fossils, staining, inclusions, fabrication issues, etc.

If further information is required an accurate and detailed petrographic assessment can be undertaken by an experienced professional at a later date.

5.2 Examination under petrographic microscope

Petrographic description of a stone material by examination of thin sections under an optical microscope in transmitted, polarized light should be carried out in accordance with EN 12407:2007 Clause 7.

The petrographic description of thin and polished sections provides information on composition, grain constituents or mineral, their type and their abundance, on texture, grain size, shape and orientation, micro-structures, discontinuities, veins, fissures, porosity, etc. In addition to the above, when applicable, petrographic examination of natural stone used in cultural heritage objects should identify weathered or otherwise altered constituents or minerals and describe the extent of that weathering or alteration.

The following information should also be included:

a) the stratigraphy of any surface layers (if present);

b) the width of every different weathering layer;

c) the adhesion and cohesion between the altered layers and the sound stone;

d) the presence of any signs of biodeterioration;

e) the character of the weathering, providing information on the decay process;

f) a basic hypothesis on the deterioration mechanism and agents that affect the stone.

g) surface soiling – its nature, level of adhesion and whether this seems to be contributing to deterioration or is merely disfiguring.

Any modification of the original characteristics of the sample during the preparation of the thin sections shall be avoided.

Excessive heating (temperatures of > 45° C) should be avoided during the preparation of the thin sections. Exposure to water should be kept to a minimum in order to avoid the occurrence of secondary hydration and the loss of water soluble compounds from the sample.

5.3 Mineralogical analysis by X-ray diffraction (XRD)

When optical examinations are insufficient, the mineralogical composition of the crystalline components of stones including clay products and salt efflorescence can be determined by powder X-ray diffraction. In case of clastic sediments, the cementing material should be analysed separately from the clastic fragments. In case of clay-containing stones, a specific methodology should be followed aiming to determine the nature of the clay phase and to enhance the intensity of peaks attributed.

In addition to the examination above listed, further information can be achieved by the following methods.

5.4 Micro-structural examination by Scanning Electron Microscopy

A Scanning Electron Microscope (SEM), equipped with Energy or Wavelength Dispersive Spectroscopy (EDS or WDS) allows morphological, microstructural and morphometric examination of the surface of the samples/sections as received or polished along with qualitative and quantitative elemental analysis, as well as mapping of the elements. In addition to the mapping of elements, SEM makes it possible to identify and map the location of soluble salt crystals.

Further information could be achieved by combining SEM and petrographic examination of thin/polished sections with image analysis.

5.5 Chemical analysis

Chemical analysis of natural stones supplies important information to establish their composition, properties and alteration which in turn can help to guide more in depth investigations, to determine the nature and distribution of contaminants, for example soluble salts, within the pores of the stone and their possible effect, to assess and understand weathering and other decay processes affecting the stone.

NOTE Chemical data of the major elements content and minor elements (content less than 0,1 %) plus trace elements present can be required to provide specific information on the geological provenance of stones. Relevant instrumental techniques include Scanning Electron Microscope (SEM-EDS/WDS), X-Ray Fluorescence Spectroscopy (XRF), Inductively coupled plasma spectrometry (ICP), Inductively coupled plasma-mass spectrometry (ICP-MS) and Neutron Activation Analysis (NAA). Additional information related to the mineralogy and chemistry of stone, their conditions of formation, secondary reactions, or unnatural treatments, can be achieved by FTIR and micro-Raman spectroscopies, based on interaction of light with molecular and/or lattice motions.

For the determination of the soluble salts see EN 16455.

5.6 Physical properties

5.6.1 General

When it is required, and is possible to collect sufficient material, the following physical properties shall be measured.

NOTE The availability of material can make it necessary to use specimen sizes that are different from those specified in the standards and can limit the tests which can be undertaken.

5.6.2 Density, porosity and pore size distribution

EN 1936 specifies methods of determining the real density, the apparent density, and the open and total porosity of natural stone.

These porosity properties can also be determined instrumentally in representative samples by mercury intrusion porosimetry (MIP) and nitrogen adsorption. The advantage of these techniques is that they can provide a more detailed description of pore structure (e.g. micro-porosity, pore size distribution, as well as adsorption and de-sorption isotherms).

5.6.3 Hydric behaviour

EN 13755 and EN 15801 specify the methods for determining the water absorption of natural stone by immersion in water at atmospheric pressure and the water absorption coefficient of natural stone by capillarity, respectively.

Measurement of water absorption under low pressure according to EN 16302 can be used for determining on site, without sampling, the water absorbed into the stone mass, through a graduated tube, as a function of time.

For the determination of the drying behaviour see EN 16322.

EN 15803 specifies the method for the determination of water vapour permeability (δp).

5.6.4 Colour

EN 15886 specifies a method for the measurement of surface colour.

5.7 Mechanical properties

5.7.1 General

When it is required and is possible to collect sufficient material the following mechanical properties shall be determined.

NOTE The availability of material can make it necessary to use specimen sizes that are different from those specified in the standards and can limit the tests which can be undertaken.

5.7.2 Compressive Strength

EN 1926 specifies a method for determining the uniaxial compressive strength of natural stones. The method determines the compressive strength of either cubic or cylindrical specimens through the application of a uniformly distributed load.

5.7.3 Flexural strength

EN 12372 specifies a test method for the determination of flexural strength of natural stones under a concentrated load. The method requires the appropriate preparation of prismatic specimens, which are placed on two rollers and are progressively loaded in the middle. The breaking load is measured and the flexural strength calculated.

5.7.4 Static modulus of elasticity

EN 14580 specifies a method for determining the static modulus of elasticity of natural stones, through uniaxial compression. The method incorporates the calculation of the static elastic modulus through the measurement of the longitudinal deformations of either prismatic or cylindrical specimens during uniaxial compressive stress.

5.7.5 Dynamic modulus of elasticity

EN 14146 specifies a method for determining the dynamic modulus of elasticity by measuring the fundamental resonance frequency.

5.7.6 Sound speed propagation

EN 14579 specifies the methodology for the determination of the ultrasonic longitudinal waves velocity in natural stone, both in laboratory and *in situ*. The dynamic modulus of elasticity can be consequently calculated.

6 Characterization report

The characterization report shall contain the following information:

a) unique identification number of the report;

b) the number, title and date of issue of the document;

c) the name and the address of the test laboratory (ies) and the address of where the test was carried out, if different from the test laboratory;

d) the name of the client (if appropriate);

e) the date when the specimens were prepared (if relevant) and the date of testing;

f) the number of specimens in the sample;

g) the dimensions of the specimens;

h) the orientation of the specimens with respect to their location in the object or monument and planes of anisotropy;

i) the type of test(s), techniques /methods and the relative protocol used;

j) the petrographic description of the stones and the values for all the properties measured, according to the requirement of the relevant standard (including tables and figures);

k) all deviations from (or modifications of) the standard methods and their justification;

l) any general remarks or comments.

Finally, an interpretation of the results may be included in the report (General remarks derived by the results).

Bibliography

[1] EN 12670, *Natural stone —Terminology*

[2] EN 12407:2007, *Natural stone test methods — Petrographic examination*

[3] EN 16095, *Conservation of cultural property — Condition recording for movable cultural heritage*

[4] EN 16096, *Conservation of cultural property — Condition survey and report of built cultural heritage*

[5] EN 16302, *Conservation of cultural heritage — Test methods — Measurement of water absorption by pipe method*

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