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Preskušanje naravnega kamna - Petrografska preiskava

Natural stone test methods - Petrographic examination

Prüfverfahren für Naturstein - Petrographische Prüfung

Méthodes d'essai pour pierres naturelles - Examen pétrographique

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Natural stone test methods - Petrographic examination

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Prüfverfahren für Naturstein - Petrographische Prüfung

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (prEN 12407:2016) has been prepared by Technical Committee CEN/TC 246 “Natural stones”, the secretariat of which is held by AENOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 12407:2007.

Introduction

A petrographic description of natural stones is important not only for the purposes of petrographic classification but also in order to highlight features affecting its chemical, physical and mechanical behaviour. In the same way the determination of the stone's origin could be necessary (e.g. in the case of restoration of historical monuments). It is therefore essential to characterize the natural stones for their mineral components and of their fabric and structure but also in terms of any features as: colour, presence of veins, of fossils, of discontinuities, etc.

To ensure that the petrographic classification is objective, it is essential that the characterization of the material is, as far as possible, quantitative.

The interpretation of the results obtained from the petrographic examination of natural stone should include evidences of a possible relationship between petrographic features and technical properties (pores/cleavages/ schistosity with water absorption/ gelivity/ flexure resistances etc.).

1 Scope

This European Standard specifies methods for making technical petrographic descriptions of natural stone, except for roofing slates. For this product, the method for the petrographic examination is defined in EN 12326–2. Although chemical and physical methods of analysis are required for petrographic classification of some stone types, these methods will not be described in this standard.

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 12670, *Natural stone — Terminology*

EN 12440, *Natural stone — Denomination criteria*

3 Principle

A macroscopic description of the sample is undertaken at first. The macroscopic description may involve a visual inspection aided by a hand lens or a stereoscopic microscope. Then one or more thin sections prepared from the sample are examined using an optical polarized transmitted light microscope in order to give a microscopic description and modal analysis (volume proportions of mineral phases) of the sample; where appropriate an additional polished section shall be prepared.

Modal analysis is the most accurate determination of quantitative mineralogical composition of natural stones. It attributes to each mineral phase a certain percentage (vol.%). Modal analysis is obtained by point counting following a standard procedure on thin sections (44 × 28 mm^2) or using Shvetsov's diagrams (or any other similar diagrams commonly used by petrographers).This is a common method to determine the mode (volume percentage of each mineral) in which the identity of the mineral phase, in a series of equally spaced points on a grid, is determined and recorded.

4 Symbols

vol.% unit of the volume percentage of the mineral phase present in the sample.

5 Apparatus

5.1 Hand lens or stereoscopic microscope (if required).

5.2 Water cooled rock cutter equipped with a continuous rim and sliding guide.

5.3 Automatic grinding machine or manual grinding machine with a cast-iron lapidary.

5.4 Electrical heating plate.

5.5 Bonding press.

5.6 Extractor hood.

5.7 Glass plate c. 300 mm × 400 mm × 10 mm.

5.8 Soft bristle brush.

5.9 Ultrasonic tank.

5.10 Polarized transmitted light optical microscope.

5.11 Point counter and image analysis system (if required).

5.12 Reactives and products: emery with grain size varied (30 μ to 10 μ), Epoxy resin, Canada Balsam, Supporting glass (28 mm x48 mm x1,8 mm), cover glass (24 mm x32 mm), diamond disks, xylene and ethanol, amaranth, sodium cobaltinitrite, hydrofluoric acid, hydrochloric acid, sulphosodic alizarin.

To facilitate the identification of some minerals with similar colours or optical properties it might be necessary in many cases to use different techniques such as: selective mineral staining methods, **optical cathodoluminescence, XRD, SEM-EDS or WDS microprobe**. If required, these methods should be clearly stated in the working order referring to widely-referenced manuals, but taking into account that such additional techniques are not part of the petrographic analysis described in this standard.

6 Preparation of thin and polished sections

6.1 General

Sampling is not responsibility of the test laboratory except where specially requested.

The dimensions of the sample shall be large enough to be representative of the petrographic characteristics of the stone being examined.

One or more thin sections are then prepared.

A thin section is a portion of material mounted on a slide and mechanically reduced to a thin sheet measuring 0,030 ± 0,005 mm in thickness, and normally protected by a slide cover. For special purposes (observations under reflected light microscope for the determination of opaque minerals or microprobe analysis) polished thin sections shall be prepared. Polished sections have one side polished with alumina polishing paste (5 μm to 12 μm grade) and diamond paste (6 μm, 3 μm and 1 μm). The polished face is left uncovered.

The section normally measures about 44 mm × 28 mm, but in the case of coarser grain size stones, larger dimensions may be used (e.g. 75 mm × 50 mm) or several sections of normal dimensions can be prepared. If the rock texture is anisotropic it is necessary to prepare at least two sections with different orientation with respect to the anisotropy (e.g. parallel and perpendicular to anisotropy planes).

Preparation of thick sections

The sample shall be sufficiently coherent so as not to disintegrate when cut. If the stone is brittle or fragile, it will be necessary to strengthen it by means of impregnation, preferably in a vacuum, with resins with an index of refraction approximately 1,54 (e.g. epoxy resins).

Using the rock cutter several small blocks are cut of 44 mm × 32 mm and 3 to 4 mm thick. In case of porous samples pores are filled with Canada balsam (or any synthetic resin with approximately equivalent refractive index), heated on the plate to approximately 100 °C and then cooled.

Blocks are then successfully ground using diamond discs between 1 and 2 min depending on the type and hardness of their constituent minerals.

Preparation of thin sections

The detailed description of preparation of thin sections is given in Annex A (informative).

6.2 Staining

6.2.1 Feldspar staining

The detailed description of feldspar staining preparation and procedure is given in Annex B (informative).

6.2.2 Carbonate staining

If samples are suspected of containing carbonates, their selective staining can be carried out. Samples are etched with a HCl solution 1:20 and then immersed in a alizarine solution during 3 min. Then samples are washed with water and are left to dry in open air. Calcite will then stain with a dark red colour, dolomite will maintain its original colour and other carbonates will be stained between rose and violet colour depending on the case. It is possible to discriminate other carbonates than calcite using other chemical attacks, but this is not frequent.

7 Macroscopic description

7.1 General

Macroscopic description shall be carried out on a fresh broken samples and, if considered necessary, on polished samples.

The following observations shall be included in the macroscopic description:

7.2 The general colour or range of colours of the hand specimen. The colour can be estimated by visual impression or defined using a colour reference chart (Rock Colour Chart is recommended).

7.3 Rock structure (joints, bedding, stylolites, etc).

7.4 Grain size (e.g. coarse, medium or fine).

7.5 Open and refilled macroscopic cracks, pores and cavities (when relevant).

7.6 Evidence of weathering and alteration: staining by sulphide alteration, diffusion of iron hydroxides, alteration of feldspars etc. (when relevant).

7.7 Presence of macrofossils (when relevant).

7.8 Presence of xenolithic and/or autolithic inclusions (when relevant).

8 Microscopic description

The following observations shall be included in the microscopic description:

8.1 Fabric.

8.2 Constituents:

8.2.1 Minerals/Grains.

For the determination of the opaque minerals polished sections should be used.

For each mineral or grain identified, the characteristics listed below shall be specified (when relevant).

8.2.1.1 Percentage by volume (vol.%), specifying the method used (e.g. estimate, point counter). The detailed principle and procedure are given in Annex C (informative).

8.2.1.2 Dimensions: mean value and range of variation (if necessary for the groundmass and also for the larger crystals or grains). The range of sizes to be used will be: Very coarse (>10 mm), Coarse (4 mm - 10 mm), Medium (1 mm - 4 mm), Fine (<1 mm). These values could not be used for clastic rocks that require the widely-used Wentworth scale. In addition, the coarse-medium grain size limit for igneous rocks is placed at 5 mm. Degree of sorting (in clastic rocks): very well sorted, well sorted, moderately sorted, poorly sorted, very poorly sorted.

8.2.1.3 Habit (e.g. idiomorphic, anhedral).

8.2.1.4 Shape (e.g. isometric, anisometric, flattened, elongated). Detrital grains in sedimentary rocks shall be described in terms of sphericity and roundness.

NOTE For marble see also EN 16306 *Natural stone test methods — Determination of resistance of marble to thermal and moisture cycles.*

8.2.1.5 Boundaries (e.g. straight, lobate, dentate).

8.2.1.6 Distribution (e.g. homogeneous, heterogeneous, in layers, in patches).

8.2.1.7 Orientation (e.g. isotropic, shape preferred orientation, dimensional preferred orientation, isorientation of lamellar or tabular grains, isorientation of elongated, prismatic grains).

8.2.1.8 Evidence of weathering and alteration: staining by sulphide alteration, diffusion of iron hydroxides, chloritization of biotite; sericization of feldspars, radioactive decay of minerals such as zircon or allanite etc.

8.2.2 Groundmass.

8.2.2.1 In volcanic rocks mesostasis which can be glassy, ipocrystalline, microcrystalline, devitrified.

8.2.2.2 In sedimentary rocks have to be distinguished matrix (microcrystalline pelitic, carbonatic or silicic mud which includes grains when present, or fills the interstices) and cement (amorphous to crystalline materials partially or completely filling cavities).

8.2.3 Organogenic remains: e.g. organic or replaced (pyrite, apatite etc.) organogenic remains.

8.3 Discontinuities:

8.3.1 Pores, microcavities (size, shape, relative abundance and filling material if present).

8.3.2 Cracks and open fractures.

8.3.2.1 Width (most frequent value, minimum and maximum).

8.3.2.2 Length (most frequent value, minimum and maximum).

8.3.2.3 Type (intergranular, intragranular, transgranular)

8.3.2.4 Orientation.

8.3.2.5 Distribution.

8.3.3 Filled fractures and veins.

8.3.3.1 Width (most frequent value, minimum and maximum).

8.3.3.2 Length (most frequent value, minimum and maximum).

8.3.3.3 Type (intergranular, intragranular, transgranular).

8.3.3.4 Orientation.

8.3.3.5 Distribution.

8.3.3.6 Filling (extent, nature, structure).

8.3.3.7 Nature (e.g. stylolites, late veins).

8.4 Alterations:

8.4.1 Description of the minerals with alterations.

8.4.2 Alteration grade of the rock.

Templates for the petrographic description of the various types of rocks are given in EN 12670.

9 Petrographic classification

On the basis of the data generated from the macroscopic and microscopic examination relating to grain size, fabric and mineralogical composition a petrographic classification shall be assigned to the stone sample, using EN 12670 and fixing at least the rock group/family.

If the petrographic description provides insufficient data to assign a petrographic classification, further testing may be necessary, namely chemical and minerochemical determinations.

10 Test report

The test report shall contain the following information:

a) unique identification number for the report;

b) number, title and date of issue of this European Standard;

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

d) name and address of the client;

e) it is the responsibility of the client to supply the following information:

   1) commercial name of the stone, in accordance with EN 12440;

   2) country and region of extraction;

   3) name of the supplier;

   4) direction of any mega and mesoscopic plane of anisotropy (if relevant to the test) to be clearly indicated on the sample by means of two parallel lines; to obtain a complete indication of the