SLOVENSKI STANDARD
SIST EN 12407:2007

01-junij-2007

Nadomeġļa:
SIST EN 12407:2002

Preskušanje naravnega kamna - Petrografska preiskava

Natural stone test methods - Petrographic examination

Prüfverfahren für Naturstein - Petrographische Prüfung

Méthodes d'essai pour pierres naturelles - Examen pétrographique

Ta slovenski standard je istoveten z: **EN 12407:2007**

ICS:

73.020	Rudarstvo in kamnolomsko izkopavanje	Mining and quarrying
91.100.15	Mineralni materiali in izdelki	Mineral materials and products

SIST EN 12407:2007 **en;fr;de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 12407

March 2007

ICS 73.020; 91.100.15

Supersedes EN 12407:2000

English Version

Natural stone test methods - Petrographic examination

Méthodes d'essai de pierres naturelles - Examen pétrographique

Prüfverfahren für Naturstein - Petrographische Prüfung

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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 12407:2007: E

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Foreword

This document (EN 12407:2007) has been prepared by Technical Committee CEN/TC 246 “Natural stones”, 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of an identical text or by endorsement, at the latest by September 2007, and conflicting national standards shall be withdrawn at the latest by September 2007.

This document supersedes EN 12407:2000.

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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

A petrographic description of natural stone is important not only for the purposes of petrographic classification but also in order to highlight features influencing 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 not only from the point of view of 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 be, as far as possible, quantitative.

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 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 12670, *Natural stone – Terminology*

EN 12440, *Natural stone – Denomination criteria*

3 Principle

First a macroscopic description of the sample is undertaken. 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 a petrographic microscope in order to give a microscopic description of the sample; where appropriate an additional polished section shall be prepared.

4 Apparatus

4.1 Hand lens or stereoscopic microscope (if required)

4.2 Rock cutter with sliding guide, water cooled and with a vertical diamond disk 3 mm thick

4.3 Electrical heating plate

4.4 Glass plate 300 mm x 400 mm x 10 mm

4.5 Bristle brush

4.6 Multiple rectifier for thin slides for 16 slides, water cooled and semiautomatic stop. Thin sections can also be prepared by hand by a thin slides preparation expert

4.7 Electric solder

4.8 Multi Form mold 40 mm and plastic cup

4.9 Grinding and polishing machine

4.10 Petrographic microscope

4.11 Point counter or image analysis (if required)

4.12 A Rock Colour Chart or another colour reference chart (if required)

4.13 Reactives and products: Canada Balsam, Thermoplastic cement, Epoxi resin, Epoxi hardener, Carborundum (F220, F400, F600, F800), Supporting glass (28 mm x48 mm x1,8 mm), cover glass (24 mm x32 mm), diamond disks, xileno & ethanol, sodium cobalt nitrite, hydrofluoric acid, hydrochloric acid, sulphosodic alizarine solution.

5 Preparation of thin and polished sections

5.1 General

The 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 with reflected light microscope for the determination of opaque minerals or microprobe analysis) polished sections or polished thin sections shall be prepared. Polished sections and polished thin 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 side remains uncovered.

The section normally measures about 44 mm x 28 mm, but in the case of larger grain size stones, larger dimensions may be used (e.g. 75 mm x 50 mm) or several sections of normal dimensions can be prepared. If the rock 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 bedding planes, cleavage planes).

Preparation of thick samples

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 x 32 mm and 3 to 4 mm thick. In case of porous samples pores are filled with Canada balsam, 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 the stone.

Preparation of thin samples

Blocks are cleaned placed on the plate and then treated with Canada balsam until caramel colour. After cooling and scratching the balsam off the surface, they are grinded dry in the glass plate with carborundum avoiding the appearance of pores. The blocks are then cleaned with the bristle brush to eliminate all carborundum particles. They are later heated in the plate with the adhesive mounting them on the supporting glass with thermoplastic adhesive. The samples are then mounted in the rectifying machine until sample thickness is 1 mm. Then they are subjected to another wet grinding process on the glass plates with the different carborundum sizes, checking the thickness every now and then controlling polarizing colours of one reference mineral existing in the sample, ending the process once the stipulated colours have been achieved (first order white and grey for quartz and feldspar for example). Thin slides are then washed with water and dried with a cloth. Samples are then cut by the edges with a cutter up until the measures of the cover glass (generally 24 mm x 32 mm). Then a volume of balsam of approximately 0,5 ml is poured over the samples and heated on the plate to 60 °C, exerting pressure on the cover plate to favour a thorough cover of the balsam over the sample avoiding the formation of bubbles.

Balsam residues remaining between cover and supporting glass can be eliminated burning them with the solder. Finally the thin slides are washed with xilene, water and soap, are left to dry in open air and are labelled with a permanent marker on the cover glass.

To facilitate the identification of some minerals with similar colours or optical properties it might be necessary in many cases to use selective staining methods of its mineral components. Out of the many existing techniques two are so common that are always carried out in all samples unless otherwise stated; these are feldspar staining methods and carbonate staining methods which are described below. Any other staining method different from those mentioned shall be clearly stated in the working order.

5.2 Staining

5.2.1 Feldspar staining

In order to carry out the feldspar staining, the thin section shall be subjected to HF acid vapour under the extracting hood and during one minute, using a plastic recipient of the same size of the cover glass. Then three drops of a solution of sodium cobaltonitrite are dropped over the section and left to act during 4 to 5 min. Then the section is washed with water: the K-feldspar is stained with yellow tones whereas the other feldspars and quartz show no change.

5.2.2 Carbonate staining

If samples are suspected of containing carbonates, their selective staining can be carried out. Samples are attacked 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 in not frequent.

6 Macroscopic description

6.1 General

Macroscopic description shall be carried out both on fresh cut samples and on polished samples.

The following items shall be included in the macroscopic description.

6.2 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).

6.3 Fabric

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

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

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

6.7 Presence of macrofossils (when relevant)

6.8 Presence of xenolithic and mafic intrusions (when relevant)

7 Microscopic description

The following items shall be included in the microscopic description:

7.1 Fabric

7.2 Constituents

7.2.1 Minerals/Grains

NOTE 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).

7.2.1.1 Percentage by volume, specifying the method used (e.g. estimate, point counter).

7.2.1.2 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). Degree of sorting (in clastic rocks): very well sorted, well sorted, moderately sorted, poorly sorted, very poorly sorted.

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

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

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

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

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

7.2.1.8 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.

7.2.2 Groundmass:

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

7.2.2.2 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)

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

7.3 Discontinuities

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

7.3.2 Cracks and open fractures

7.3.2.1 Width (most frequent value, minimum and maximum)

7.3.2.2 Length (most frequent value, minimum and maximum)

7.3.2.3 Type (intergranular, intragranular, transgranular)

7.3.2.4 Orientation

7.3.2.5 Distribution

7.3.3 Filled fractures and veins

7.3.3.1 Width (most frequent value, minimum and maximum)

7.3.3.2 Length (most frequent value, minimum and maximum)

7.3.3.3 Type (intergranular, intragranular, transgranular)

7.3.3.4 Orientation

7.3.3.5 Distribution

7.3.3.6 Filling (extent, nature, structure)

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

7.4 Alterations.

7.4.1 Description of the minerals with alterations

7.4.2 Alteration grade of the rock

Templates for the petrographic description of the various types of rocks are given in Annex A (informative).

8 Petrographic definition

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

If the petrographic description provides insufficient data to assign a petrographic definition, further testing may be necessary, namely chemical or X-ray diffraction determinations.