Natural stone test methods — Determination of Knoop hardness

The European Standard EN 14205:2003 has the status of a British Standard

ICS 73.020; 91.100.15

British Standards

National foreword

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The UK participation in its preparation was entrusted to Technical Committee B/545, Natural stone, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 9 and a back cover.

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English version

Natural stone test methods - Determination of Knoop hardness

Méthodes d'essai pour les pierres naturelles - Détermination de la dureté Knoop

Prüfverfahren für Naturstein - Bestimmung der Härte nach Knoop

This European Standard was approved by CEN on 1 September 2003.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 14205:2003 E

Contents

page

Foreword

This document (EN 14205:2003) 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of an identical text or by endorsement, at the latest by May 2004, and conflicting national standards shall be withdrawn at the latest by May 2004.

This standard is one of the series of standards for tests on natural stone.

Test methods for natural stone consist of the following parts:

EN 1925, *Natural stone test methods – Determination of water absorption coefficient by capillarity*

EN 1926, *Natural stone test methods – Determination of compressive strength*

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

EN 12370, *Natural stone test methods – Determination of resistance to salt crystallisation*

EN 12371, *Natural stone test methods - Determination of frost resistance*

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

EN 12407, *Natural stone test methods – Petrographic examination*

EN 13161, *Natural stone test methods – Determination of flexural strength under constant moment*

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

EN 13373, *Natural stone test methods – Determination of geometric characteristics on units*

EN 13919, *Natural stone test methods – Determination of resistance to ageing by* SO_2 *action in the presence of humidity*

EN 14066, *Natural stone test methods – Determination of resistance to ageing by thermal shock*

prEN 14147, *Natural stone test methods – Determination of resistance to ageing by salt mist*

prEN 14157, *Natural stone test methods – Determination of the abrasion resistance*

prEN 14158, *Natural stone test methods – Determination of rupture energy*

prEN 14231, *Natural stone test methods – Determination of the slip resistance by means of the pendulum tester*

prEN 14581, *Natural stone test methods – Determination of thermal expansion coefficient*

prEN 14579, *Natural stone test methods – Determination of sound speed propagation*

prEN 14580, *Natural stone test methods – Determination of the static elastic modulus*

No existing European Standard is superseded.

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1 Scope

This European Standard specifies a method of determining the hardness of minerals in natural stone using the Knoop indenter. This method is especially useful for carbonate rock.

2 Principle

After carrying out a series of indentations by means of a Knoop indenter, the corresponding values of Knoop microhardness are calculated and the microhardness distribution is given.

3 Symbols

P - load on the indenter, in newtons

L - length of the largest diagonal of the indentation, in millimetres

HK - Knoop microhardness, in Megapascals

*HK*25 - Knoop microhardness corresponding to a cumulative frequency of 25% (“lower quartile”), in Megapascals

*HK*50 - Knoop microhardness corresponding to a cumulative frequency of 50% (“median value”)

*HK*75 - Knoop microhardness corresponding the cumulative frequency of 75% (“upper quartile”)

4 Apparatus

A microdurimeter essentially made of the following parts:

- sample holder with a mechanism for horizontal movement in two orthogonal directions by means of adjustable screws which also measure the amount of movement;
- Knoop indenter (see Figure 1);
- set of interchangeable weights from (0,1 to 5,0) Newtons;
- device for applying the load on the indenter at different speeds;
- microscope with a micrometer for measuring the indentation width and length with an accuracy of 0,5 micrometres.

5 Preparation of the specimens

5.1 Sampling

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

5.2 Number of specimens

At least one polished section shall be prepared approximately 20 mm width, 30 mm length and 10 mm thickness. Other sizes may be used provided that there is enough space on the polished face to carry out the necessary sequence of indentations.

In the case of stones showing anisotropy planes (e.g. bedding, foliation) at least two polished sections shall be prepared: one with the polished face parallel to the anisotropy planes and the other perpendicular.

In the case of very inhomogeneous coarse-grained stones, it is necessary to prepare a larger section than the previously described one or alternatively, to use several sections of standard dimensions (so that for the specimen be sufficiently representative).

6 Test procedure

The test consists of carrying out:

- 20 indentations, in line and with 1 mm between them, for fine-grained, visibly uniform types of stone;

- three series (20 + 10 + 10) of indentations for heterogeneous, medium-grained or coarse-grained stone; in each series the distance between the indentations will be 1 mm;

- eight series of 10 indentation each for very heterogeneous or very coarse grained stones; in each series the distance between the indentation shall be not less than 2 mm.

Proceed as follows:

1) place the chosen weight (see 4) on the tray to apply the load;

NOTE 1 The load applied is equal for all types of rock (the recommended load is 1,96 N since this creates indentations that are measurable in both hard and soft rocks).

2) position the specimen on the sample holder and focus by means of the microscope lens;

3) use the two directional stage holder to bring the point chosen as the first point of the random sequence of 20 aligned indentations to the centre of the field. For visibly anisotropic stones, choose an alignment perpendicular to the planes of anisotropy;

4) rotate the lens to bring the indenter over the first point of the sequence and activate the indenter's automatic descent mechanism;

NOTE 2 The speed at which the load is applied is the same for all types of rock (the recommended speed is that at which complete application of the load of 1,96 N is achieved in 40 s).

5) once the load has been applied, lift the indenter, rotate the lens into the operating position and then measure the length of the largest diagonal of the indentation by means of the graduated scale of the microscope;

6) using the two directional stage holder, move the sample holder one millimetre in the direction chosen for the alignment of the measurements: the indenter is now in position for the second point. For visually uniform fine-grained stones repeat the above operations until 20 indentations have been made and measured;

NOTE 3 In order to avoid non-random (i.e. "unconsciously intentional") positioning, move the sample holder indenter 1 mm between one measurement and the next without looking through the microscope.

7) in the case of heterogenous, medium-grained or coarse-grained stones, carry out another two series of 10 measurements, still 1 mm apart, in any direction different from the initial one (for a total of 40 indentations);

8) in the case of very heterogeneous or very coarse-grained stones, it is necessary to carry out another set of 40 indentations on at least another specimen or on the same specimen (if a polished section of larger dimensions is used). In both cases the distance between the indentations shall be not less than 2 mm.

7 Expression of results

For each indentation Knoop hardness *HK* (in MPa) is expressed by means of the following formula:

$$HK = 14{,}23\frac{P}{L^2} \quad (1)$$

where:

P is the load on the indenter, in Newtons;

L is the length of the largest diagonal of the indentation left by the indenter, in millimetres.

The hardness values obtained are arranged in increasing order and are plotted against the order of rank. The abscissa scale can also be graduated in percentages: this gives a diagram of the cumulative frequency of the microhardness values of the stone ("hardness distribution diagram", see Figure 2). The parts of the diagram parallel (or sub-parallel) to the abscissa indicate contents of the components with well-defined hardness values, whereas sloping parts of the diagram indicate the contents of the components with hardness values that vary gradually between two extremes.

The hardness distribution diagram gives a very detailed information on stone hardness; however, for comparison purposes, a more synthetic expression of the results is needed, making reference to the following characteristic values (see Figure 2):

- microhardness value corresponding to the cumulative frequency of 25% (*HK*25 or "lower quartile");
- microhardness value corresponding to the cumulative frequency of 50% (*HK*50 or "median value");
- microhardness value corresponding to the cumulative frequency of 75% (*HK*75 or "upper quartile").

NOTE The ratio between *HK*75 and *HK*25 provides an indication on the uniformity of the stone from point of view of the hardness: the nearer the ratio is to 1, the more uniform the stone.

8 Test report

The test report shall contain the following information:

a) unique identification number of the report;

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

c) name and address of the test laboratory and the address where the test 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:

- petrographic name of the stone;
- commercial name of the stone;
- country and region of extraction;
- name of the supplier;

- direction of any existing plane of anisotropy (if relevant to the test) to be clearly indicated on the sample or on each specimen by means of two parallel lines;

- name of the person or organization which carried out the sampling;

- surface finish of the specimens (if relevant to the test).

f) date of delivery of the sample or of the specimens;

g) date when the polishes section(s) were prepared and the date of testing;

h) number and the dimensions of the polished sections;

i) orientation of the polished face of each section with respect to the anisotropy planes;

j) load applied on the indenter and the load application rate;

k) individual microhardness values *HK*, calculated on the basis of the sequence of 20 or 40 or 80 measurements, and the cumulative frequency diagram ("hardness distribution diagram");

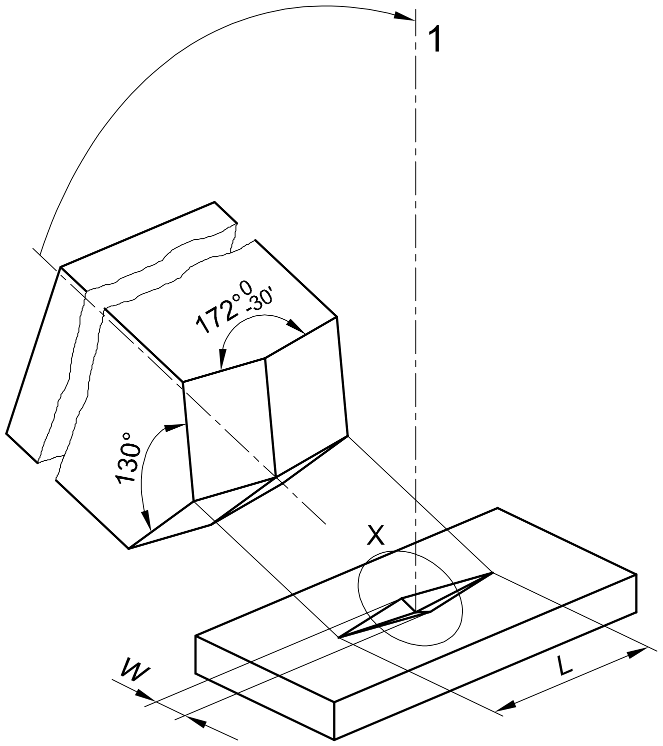
l) mean value of the individual microhardness values ($\overline{HK}$);

m) characteristic microhardness values:
HK25 (lower quartile), *HK50* (meadian value), *HK75* (upper quartile);

n) all deviations from the standard and their justification;

o) remarks.

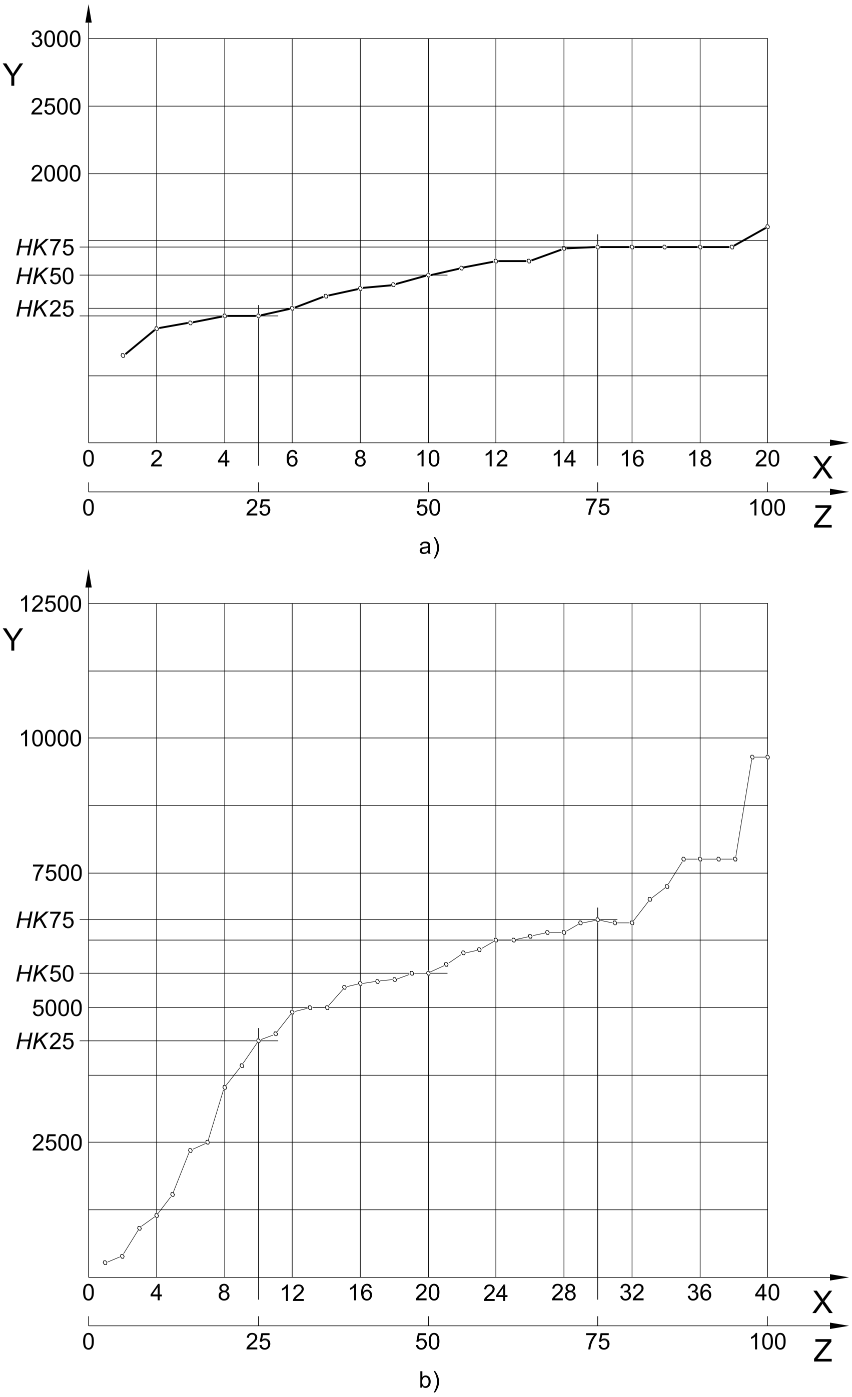
The test report shall contain the signature(s) and role(s) of the responsible(s) for the testing and the date of issue of the report. It shall also state that the report shall not be partially reproduced without the written consent of the test laboratory.



Key

1 operating position
L length
W width
d depth of the indentation

Figure 1 — Knoop indenter and its indentation



Key

X *n (*order of rank of the hardness values)

Y *HK* (Knoop microhardness in Megapascals)

Z *F* (cumulative frequency in percent)

*HK*25 microhardness value corresponding to the cumulative frequency of 25% ("lower quartile")

*KH*50 microhardness value corresponding to the cumulative frequency of 50% ("median value")

*HK*75 microhardness value corresponding to the cumulative frequency of 75% ("upper quartile")

NOTE The microhardness values are plotted in increasing order, not in the order in which the measurements were taken.

Figure 2 — Representation of the results of a microhardness test (hardness distribution diagram):
a) of a fine-grained, visibly uniform stone (e.g. marble)
b) of a medium-grained or coarse-grained non-uniform stone (e.g. granite)

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