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Natural stone test methods — Determination of sensitivity to accidental staining

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The UK participation in its preparation was entrusted to Technical Committee B/545, Natural stone.

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Natural stone test methods - Determination of sensitivity to accidental staining

Méthodes d'essai pour les pierres naturelles - Détermination de la sensibilité au tachage accidentel

Prüfverfahren für Naturstein - Bestimmung der Empfindlichkeit gegen unbeabsichtigte Fleckenbildung

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EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 16301:2013) 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of an identical text or by endorsement, at the latest by September 2013, and conflicting national standards shall be withdrawn at the latest by September 2013.

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1 Scope

The European Standard specifies a method to assess the sensitivity of natural stones when exposed to accidental staining. It defines a procedure for the application of the stains, the cleaning and the assessment of the surface appearance after cleaning. It also covers the possibility to assess the efficiency of a surface treatment. Note that the method does not intend to present any de-staining technique.

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 ISO 11664-2, *Colorimetry – Part 2: CIE standard illuminants (ISO 11664-2)*

EN ISO 11998, *Paints and varnishes – Determination of wet-scrub resistance and cleanability of coatings (ISO 11998)*

ISO 1065, *Non-ionic surface-active agents obtained from ethylene oxide and mixed non-ionic surface-active agents – Determination of cloud point*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
surface finishing
final surface texture applied to a stone during processing

3.2
matt finished surface
surface treatment to produce a very flat, uniform, but no polished finish

Note 1 to entry: Matt finished surface can be obtained by means of a silicium carbide bonded polishing disk with grain size F 400 mesh.

3.3
surface treatment
application of certain materials (i.e chemicals) to the exposed face of a slab

[SOURCE: EN 12670:2001, 2.4.29]

3.4
impregnation
protection of the surface of a stone by a pore-sealing product or a chemical coating

3.5
staining agent
liquid used to produce stains on the surface of the stone

4 Principle

A defined set of staining agents is applied on a defined stone surface. The mode of application is specified. The specimens are conditioned and washed with a standardised cleaning machine. The stones are dried and the possible remainders of the stains are assessed by a visual observation.

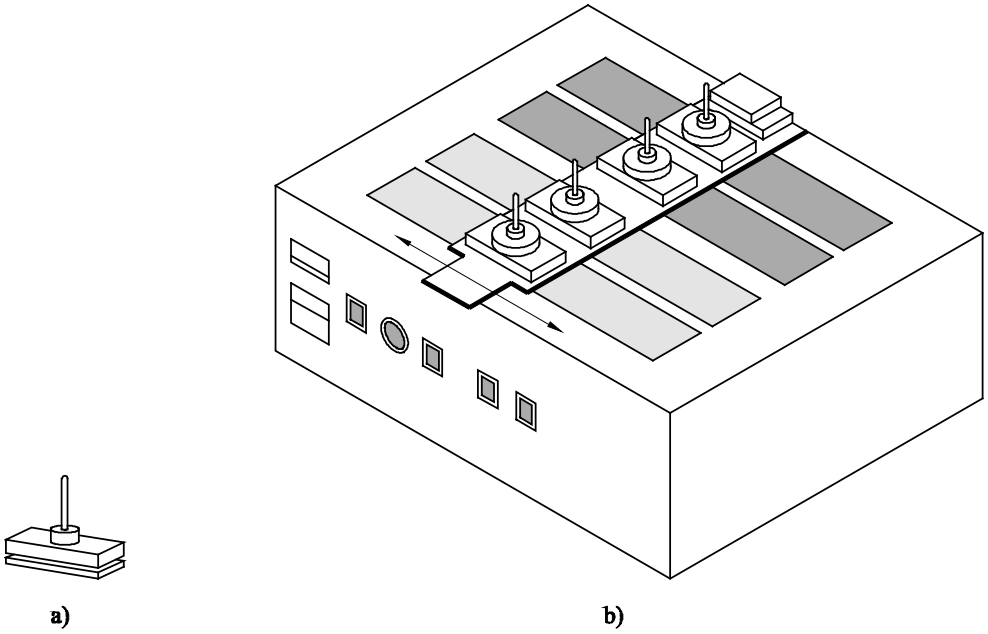
5 Apparatus

5.1 **Ventilated oven** capable of maintaining a temperature of (40 ± 5) °C and (70 ± 5) °C

5.2 **Pipette**, 0,10 ml – 1,00 ml

5.3 **Wet Abrasion Scrub Tester**, according to EN ISO 11998. The machine (Figure 1) works with a sinus-shaped movement with a frequency of 35 cycles per minute, and amplitude of approximately 350 mm. The total weight of the scrubbing arm with dry cloth and its holder shall be (350 ± 10) g.

5.4 **Weights** to achieve the specified load on the scrubbing arm.



Key

a) scrubbing arm with dry cloth and its holder
b) the machine

Figure 1 — Example of washability apparatus for cleaning test

5.5 Cleaning cloth

a) Dimensions of the cloth 120 mm × 38 mm (may be different depending on the cleaning equipment).

b) Thickness: (2 ± 0,5) mm.

c) Area weight: (300 ± 50) g/m^2.

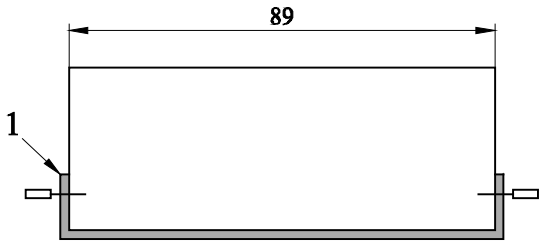
d) A thinner cloth may be used in layers to achieve the above stated properties.

e) At least 50 % viscose (regenerated cellulose fibres).

f) At least 20 % cotton.

5.6 Holder for the cleaning cloth, e.g. wooden block (Figure 2). The scrubbing arm of the machine is equipped with e.g. a wooden block measuring 89 mm × 38 mm × 32 mm. The block holds a piece of cleaning cloth measuring 120 mm × 38 mm. The standard dimensions of the scrubbing area of the brush in the standards mentioned above are 89 mm × 39 mm.

Dimensions in millimetres



Key

1 cloth

Figure 2 — Cleaning cloth on wooden block

5.7 Camera, digital (a minimum of 2,5MPixels, uncompressed), for documentation of unstained and stained test specimens.

5.8 Daylight or illuminants D65 (6500K) according to EN ISO 11664-2.

5.9 Stand for repeatable photography conditions.

5.10 Cleaning solution pH-neutral, without abrasive, multipurpose cleaner, surfactant-based anionic type or a product basing the following composition:

- 12,5 % (m/m) of a sodium primary (C10-C14) polymer alkyl aryl sulphonate;
- 12,5 % (m/m) polyethylated derivatives of primary or secondary (C8-C16) alcohols with 5 to 15 ethoxylated groups having a cloud point of 25 °C to 75 °C in 1 % (m/m) aqueous solution (determination of cloud point is described in ISO 1065);
- 5,0 % (m/m) ethanol;
- 70 % (m/m) deinonized or distilled water.

The cleaning agent shall be stored in a glass bottle in a cool dark place and should be used within 1 year of the day of preparation.

5.11 Hand spray pump for applying the cleaning solution.

5.12 Staining agents the set of staining agents to be used for the test is defined in Annex A.

6 Preparation of specimens

6.1 Sampling

The method of sampling shall be stated in the test report and shall be chosen so that the samples are representative of the batch to be tested.

6.2 Test specimens

6.2.1 Surface finish

For identification test and comparison, the specimens shall have a “matt finished surface”.

NOTE Schist and slate are exceptions to this rule. They can be tested on natural cleft surface.

For “technological testing”, surfaces with a roughness > 0,5 mm are not suitable for testing due to the risk of damaging the apparatus.

6.2.2 Dimensions

One sample set consists of three test specimens sized, minimum 300 mm by 100 mm, and thickness as in use (maximum 30 mm). Alternative dimensions can be used if different cleaning equipment is used.

6.2.3 Testing environment

The test specimens shall be placed in (23 ± 5) ºC and (50 ± 5) % relative humidity during the entire stain exposure.

Daylight or artificial light D65 is used during the photography and the evaluation.

6.2.4 Pre-conditioning and treatment of the specimens

Marble and limestone shall be dried in (40 ± 5) °C for one week before the test. Other stone types shall be dried in (70 ± 5) °C for at least 24 h.

Commercially used stone products are sometimes impregnated in some way to make them easier to clean. The traditional treatment of carbonaceous stones is soft soap, which partially seals the pores and makes the surface a bit more hydrophobic. For standard test the stones shall be non-treated as a reference. If a carbonaceous stone type is tested, one set should also be treated with soft soap 2,5 vol. % solution in water, or similar which is recommended by the Stone Industry federations. The soap treatment is not mandatory, but highly recommended.

Instruction for the treatment with soft soap:

— the solution is sprayed on with a hand-pumped spray;

— after 3 h: The next layer is applied;

— after another 21 h: The excess shall be rinsed off under running water.

The test specimens are kept in (23 ± 5) ºC and (50 ± 5) % relative humidity for a minimum 16 h before applying the staining agents.

If a surface treatment/coating chemical or impregnation chemical shall be tested, the procedure of applying this shall be documented and follow the manufacturer’s instruction.

7 Procedure

7.1 Documentation of the test specimens

Document the test specimens by digital photography before the stains are applied. Use daylight or artificial light D65. The photographs should be scaled.

7.2 Staining agents to be used in the standard test

The set of staining agents to be used for the test is defined in Annex A.

NOTE For specific uses, it may be required to use also urine and blueberry soup (Annex A).

Other products can be used and prepared according to instructions by the manufacturer or commissioner.

7.3 Staining procedure

All samples have to be stained in the same way.

The stains shall have the same size and shape.

All staining shall be performed at (23 ± 5) ºC and (50 ± 5) % relative humidity. The samples shall remain in this atmosphere during the whole exposure time, usually 15 min and (48 ± 1) hours as a default value.

Other time schedules may be used provided that the commissioner asks for it.

The diameter of the stains shall be (35 ± 5) mm.

NOTE Most water based stains, like Cola, wine and coffee has the same surface properties and a suitable stain volume is 0,70 ml. This volume is also sufficient for ketchup which shall be spread out to the same size. A stain like oil has completely different surface properties and immediately floats out on most stone materials. To get a stain in the same size as for the water based products a volume of 0,15 ml is suitable.

Surface treated samples may need the staining agent to be spread out to reach the optimal 35 mm Ø.

Dimensions in millimetres

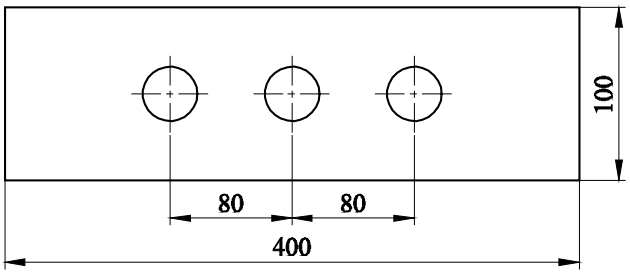


Figure 3 — Optimal sample size and stain pattern

7.4 Cleaning procedure

— Gently remove possible liquid residues of the stains with absorb and dry paper.

— Apply the correct load to the cleaning arm, (350 ± 10) g (arm, cloth and cloth holder).

— Prepare cleaning solution with 15 ml of the cleaning agent *(ref. 5.10)* in 1000 ml deionized or distilled water.

— Spray cleaning solution on the stains 4 min before the cleaning.

— Soak the cleaning cloth in cleaning solution.

— Mount the cleaning cloth and the holder to the scrubbing arm.

— The stained surfaces are then washed with a “Wet Abrasion Scrub Tester”.

— Ten cleaning cycles (10 strokes back and forth) shall be used in all tests.

— Rinse the surfaces with 150 ml water to remove residues of the cleaning solution.

— Condition the test specimens for 24 h in (23 ± 5) ºC and (50 ± 5) % relative humidity before they are photographed and evaluated.

— Photograph the test specimens in using the same scaling and light conditions used to photograph the test specimen before staining.

8 Evaluation of staining and expression of the result

A visual observation of the residual staining shall be made according to the following classification:

1) unchanged

2) light change

3) moderate change

4) severe change

The visual assessment is to be performed in daylight or artificial light D65.

Etching effect (gloss change) from acidic staining agents shall be assessed separately. The same classification is used and the result is reported separately. The evaluation can be done in a reflecting light.

Note that an etching effect is, in cases, also observed as a colour change. The use of a gloss meter is not recommended.

See Annex B, with photos exemplifying the difference between the grades.

The evaluation is performed by three persons individually. The three stains on each test specimen are evaluated as a group. Each stain type is evaluated separately. Present the mean value as an integral number (1, 1, 2 = 1). If the result of two evaluators differs more than one grade, the evaluators have to come to consensus by discussion.

9 Precision

No inter-comparison trial has been performed between different laboratories. The repeatability is based on the fact that three different persons evaluate the changes by using 3 categories. The acceptable difference between the persons is maximum 1 category. If there is more than 1 scale deviation between any of the evaluators they shall discuss until consensus is reached.

10 Test report

The test report shall include the following information (when relevant):

a) name and address of the testing laboratory;

b) identification number of the test report;

c) name and address of the organisation or the person who ordered the test;

d) purpose of the test;

e) method of sampling and other circumstances (date and person responsible for sampling);

f) name and address of the manufacturer or the supplier of the tested object(s);

g) name and other identification marks of the test specimens;

h) description of the test specimens;

i) date of supply of the test specimens;

j) date of test;

k) reference to this method;

l) condition of the test specimens (i.e. orientation of stone fabric in relation to the test object and the surface finishing);

m) surface coating or impregnation if used;

n) staining agents used;

o) cleaning liquid used;

p) time of exposure;

q) any deviations from the test method;

r) test result as defined in Clause 8;

s) photographs of the stains, indicating the staining agent used;

t) statement about the uncertainty of measurement;

u) 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

Annex A
(normative)

Specification of staining agents

The choice of the standard set of stains cover a range of strong colour (coffee, wine), high penetration and viscosity (oil), acidity (cola, citric acid, ketchup).

Red wine	>80 % Cabernet Sauvignon, pH 3,0 – 4,0
Cooking oil	100 % olive-oil without any colorants
Instant Coffee	Brewed and freeze-dried coffee powder 4 g is solved in 100 ml hot (>80 ºC) deionised water. Let cool down to about 23 ºC before use
Ketchup	pH 3 – 4
Cola	“regular Coke”, not light or with modified taste pH 2
Citric acid	Dilute for pH 1,5 – 1,8
OPTIONAL	
Artificial urine	Urea 25 g Sodium chloride 9 g Disodium hydrogen phosphate, anhydrate 2,5 g Potassium dihydrogen phosphate, anhydrate 2,5 g Ammonium chloride 3 g Creatine 2 g Sodium sulphite 3 g Distilled water up to 1 l
Lime concentrate	pH 1,5 – 1,8
Blueberry soup	Commercially available 10 g dry soup is solved in 50 ml hot de-ionised water. pH 3

Annex B
(informative)

Colour pictures to facilitate the evaluation of staining

The photos give examples of some common stones on the market. Depending on the type of staining agent and surface, it has been possible to visualise all grades of the evaluation scale from 1 to 4.

NOTE In some cases, the ultrabasite is difficult to illustrate by photo since the stains are partly a change in colour and partly an effect of etching.

Classification of the stains shown in the figures:

1 unchanged

2 light change

3 moderate change

4 severe change

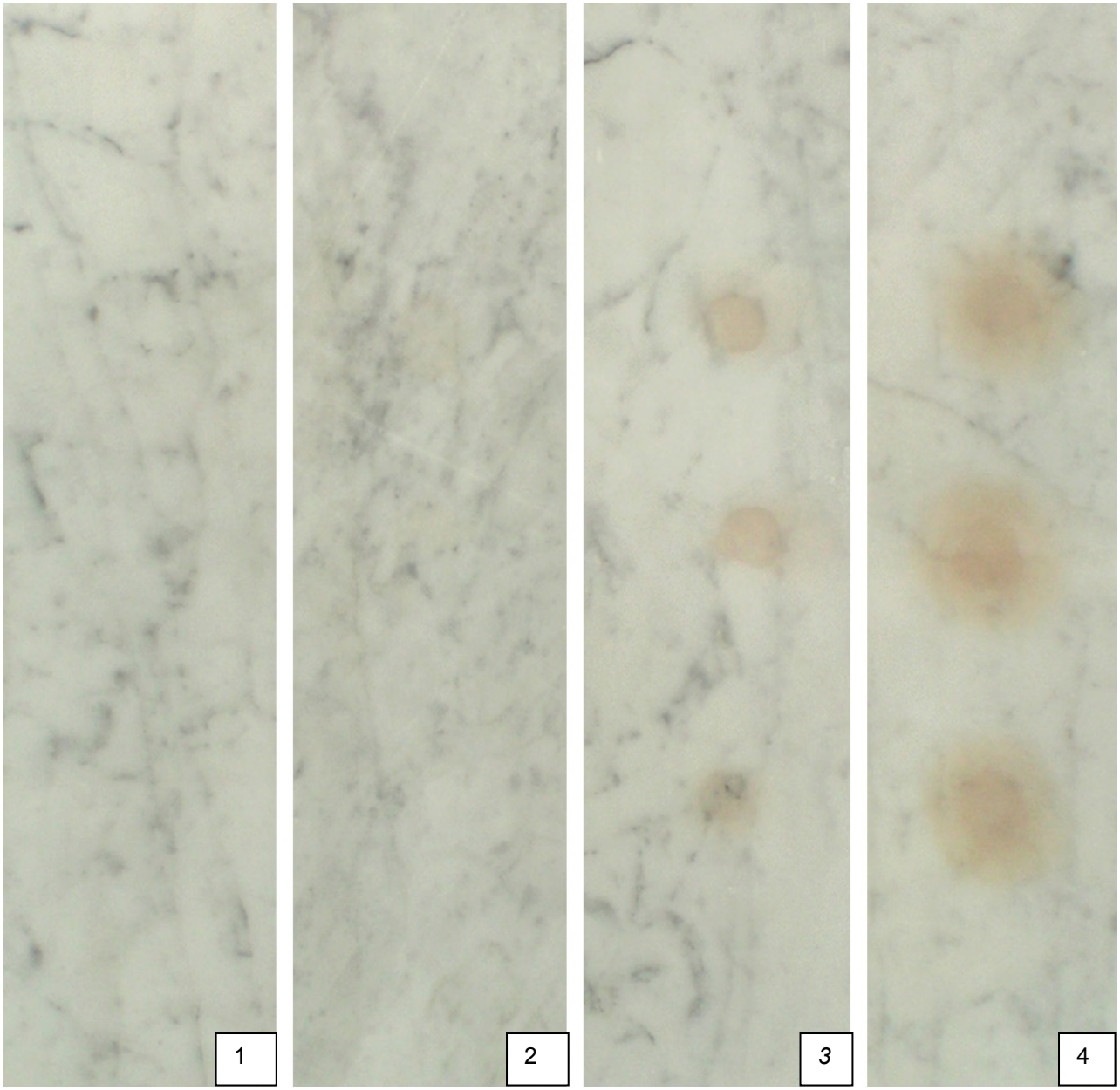


Figure B.1 — Marble

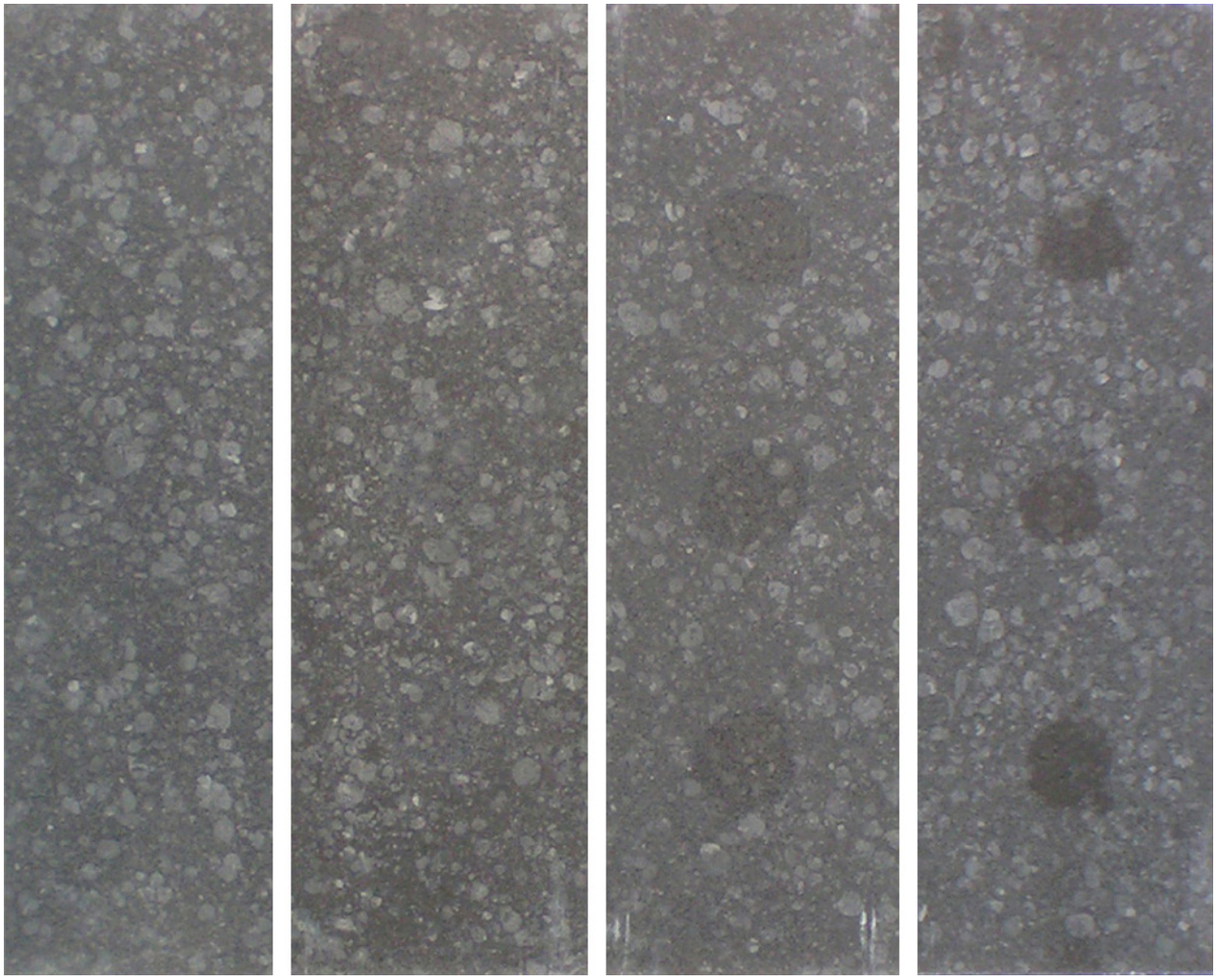


Figure B.2 — Ultrabasite

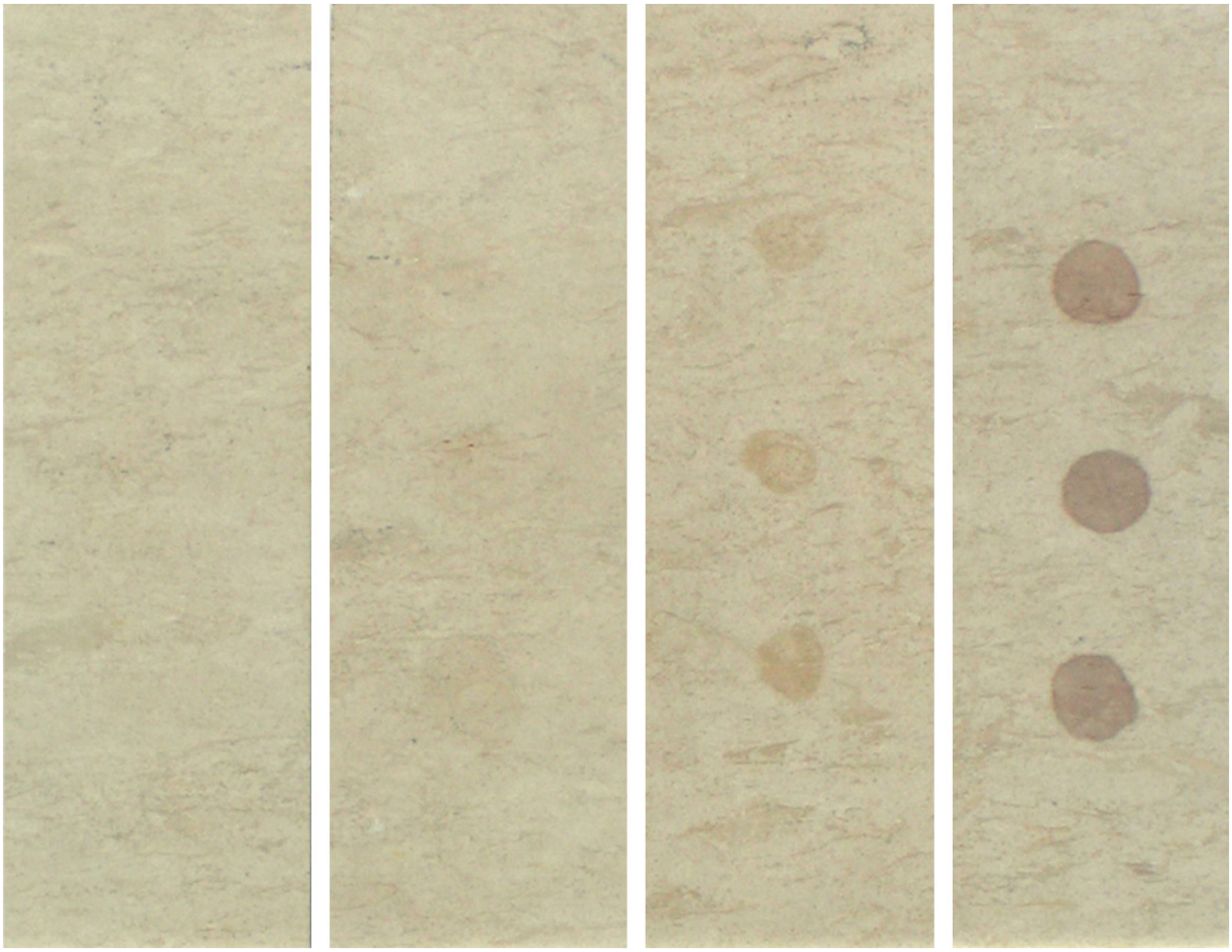


Figure B.3 — Limestone

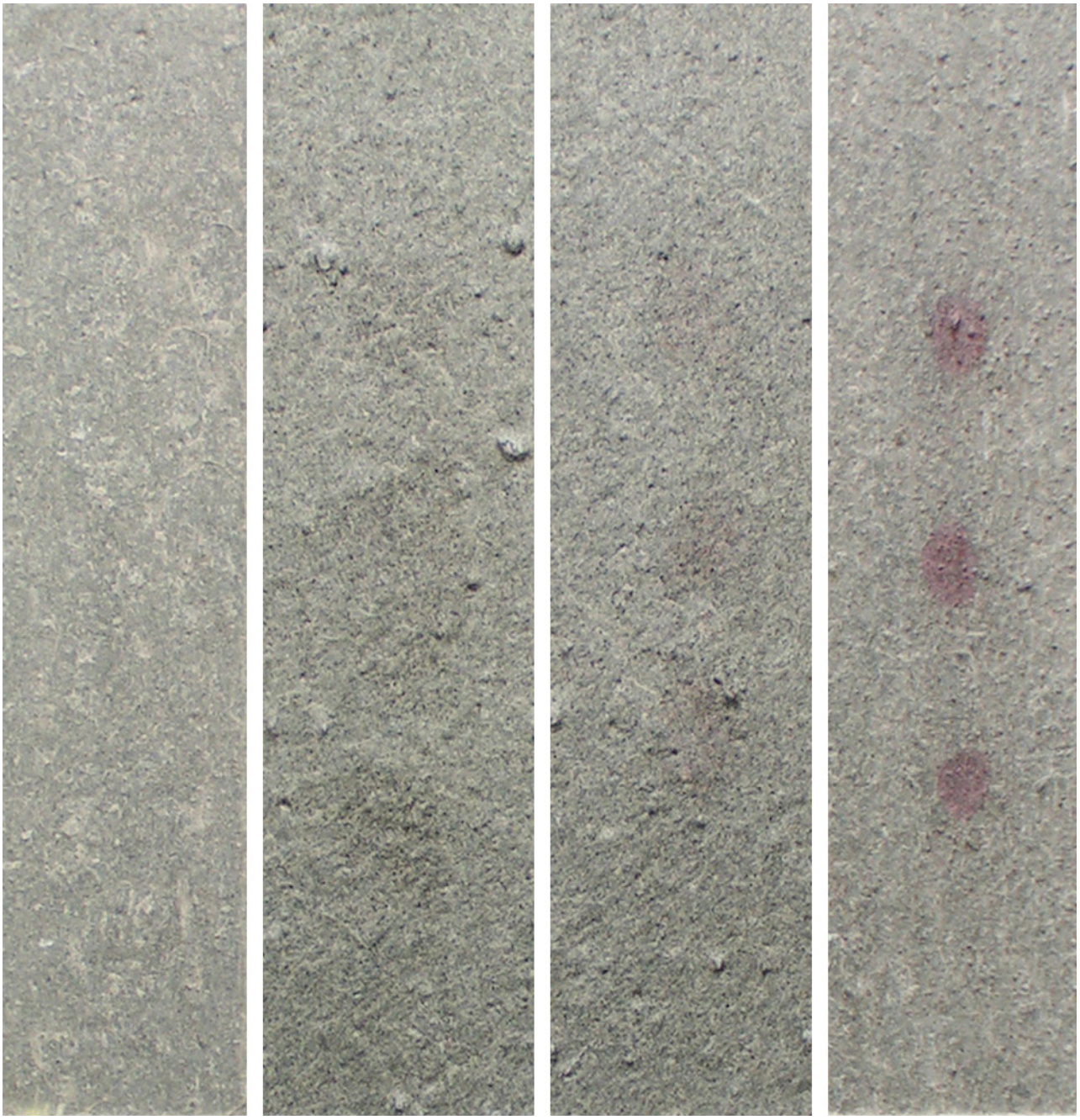


Figure B.4 — Schist

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[1] EN 12059, *Natural stone products – Dimensional stone work – Requirements*

[2] EN 12720, *Furniture – Assessment of surface resistance to cold liquids*

[3] ISO 11664-2, *Colorimetry – Part 2: CIE standard illuminants*

[4] EN ISO 4628-1, *Paints and varnishes - Evaluation of degradation of coatings - Designation of quantity and size of defects, and of intensity of uniform changes in appearance - Part 1: General introduction and designation system (ISO 4628-1)*

[5] EN 12670:2001, *Natural stone – Terminology*

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