SLOVENSKI STANDARD
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Ohranjanje kulturne dediščine - Postopek za analitično vrednotenje in izbiro metod za čiščenje poroznih anorganskih materialov, uporabljenih pri tej kulturni dediščini

Conservation of cultural heritage - Procedure for the analytical evaluation and selection of cleaning methods for porous inorganic materials used in cultural heritage

Erhaltung des kulturellen Erbes - Methodologie für die Bewertung von Reinigungsmethoden - Verfahren für die analytische Prüfung der Objekte des kulturellen Erbes

Conservation du patrimoine culturel - Procédure pour l'évaluation analytique et le choix des méthodes de nettoyage des matériaux inorganiques poreux dans les bâtiments d'intérêt patrimonial

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Conservation of cultural heritage - Procedure for the analytical evaluation and selection of cleaning methods for porous inorganic materials used in cultural heritage

Conservation du patrimoine culturel - Procédure pour l'évaluation analytique et le choix des méthodes de nettoyage des matériaux inorganiques poreux dans les bâtiments d'intérêt patrimonial

Erhaltung des kulturellen Erbes - Methodologie für die Bewertung von Reinigungsmethoden - Verfahren für die analytische Prüfung der Objekte des kulturellen Erbes

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

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Contents

Page

European foreword

This document (prEN 17488:2020) has been prepared by Technical Committee CEN/TC 346 “Conservation of Cultural Heritage”, the secretariat of which is held by UNI.

This document is currently submitted to the CEN Enquiry.

Introduction

Cleaning is a conservation treatment aimed at the removal of material of natural or artificial origin, which has the potential to damage the substrate, or which may limit the correct interpretation of the object or parts thereof.

The actions required for cleaning involve a degree of risk for the object and therefore demand extreme caution. Cleaning needs to be effective while not being harmful to the object.

Cleaning operation needs to take into account the compatibility definition (EN 15898) as the "extent to which one material can be used with another material without putting significance or stability at risk". Extending the definition of compatibility to the cleaning action a "compatibility analysis" should therefore ascertain how cleaning actions (in terms of effectiveness and harmfulness) would impact on the significance and stability of the heritage object.

A successful cleaning strategy requires the careful consideration of a number of aspects which include but are not limited to the following:

— context and sensitivity of the object to be evaluated (for example presence of polychrome and gilt surfaces);

— conservation condition of the substrate, which may result in greater risk of harm during testing;

— the aim of the cleaning in terms of which materials are to be removed and which to be retained;

— function and form of the substrate (flat or carved surface).

These factors may exclude the use of one or more cleaning methods, which would be unsuitable.

This document takes into account the extreme variability of both the constituent materials and the conditions of the object, prescribing a procedure of analytical tests and comparing the extent of possible damage, which may result from each cleaning method tested.

"Harmfulness" indicates the level of risk of the variety of unwanted changes, which may appear not only in short-term but also in long term after cleaning.

This may include the deterioration of the substrate, the increase of porosity or surface roughness, the release of residual substances and formation of stains, which are not compatible with the material and or which could interfere with future conservation intervention.

The potential harmfulness of a cleaning method may be greater when it is applied to a deteriorated material.

Evaluation of effectiveness and potential harmfulness of cleaning methods need to be carried out on site by establishing a "trial area" as a preliminary step before any extensive work is started.

The process of cleaning requires careful evaluation throughout the work. This is initiated at the primary evaluation through the execution of trials and continues with the monitoring and optimization during the selected process. Trials aim to identify the method(s) of cleaning which produce an acceptable result at minimum risk to the object. Even an extensive sampling procedure may fail to identify all the conditions which exist on a cultural object and ongoing evaluation of the cleaning and the effects on the substrate are therefore vital. If necessary, cleaning needs to be halted to re-appraise the methods used or to require further testing where areas of increased sensitivity or uncertainty are uncovered. This document identifies the means by which cleaning methods may be selected and evaluated as part of conservation interventions.

Unwanted interactions may arise from different substrate and cleaning method combinations. Some features of a given substrate may cause it to be particularly damaged depending on the method and circumstances of its use. These specific combinations will increase likelihood of damage. In some

circumstances the synergistic effects can be easily predicted, e.g. a salt-laden wall masonry may be seriously affected by a water-based method, even if we know that generally this cleaning method is gentle and of low aggressiveness. The assessment of the synergistic effects should bear in mind that damage may emerge after some considerable elapsed time.

The initial assessment for a building or similar immovable object will take place on site with non-invasive systems. If necessary, it may be followed by appropriate micro-invasive or invasive laboratory analysis (Figure 1, Table 1).

Cleaning methods considered in EN 17138:2018 were divided into four categories: Water cleaning, mechanical cleaning, physical cleaning and chemical cleaning. Each method requires different considerations in order to select the most appropriate investigations.

Assessment of harmfulness for chemical cleaning methods needs additional investigations with respect to the possible interactions between the chemicals and the products to be removed, notably the formation of by-products which could be harmful for the substrate. As a consequence, the procedure for chemical cleaning will follow a different pathway (Figures 2 and 6, Table 1).

Evaluation tests of methods or materials which are under development should not be undertaken on objects of cultural heritage. The methods outlined in this document are instead to be used on analogous materials, similarly decayed, with enough material employed to fully evaluate pre and post cleaned conditions. In the case of testing for new cleaning methods, therefore, follow the procedure in Part B.

1 Scope

This document gives the test methodology for evaluation of both harmfulness and effectiveness of a cleaning method as applied to porous inorganic materials. These include materials such as natural or artificial stone, included those with painting layers as finishes. Evaluation admits the use of on site instrumental analyses and the taking of samples to be studied in laboratory.

Harmfulness evaluation has a priority over the effectiveness.

This document applies to:

a) all methods of cleaning, which have characteristics of parameterization and reproducibility (see EN 17138:2018). Part A;

b) all new methods that are under development. Part B.

This document applies to evaluate the optimum methods for cleaning and the optimization of the parameters of the selected cleaning process.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 15801, *Conservation of cultural property — Test methods — Determination of water absorption by capillarity*

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

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

EN 16515:2015, *Conservation of Cultural Heritage — Guidelines to characterize natural stone used in cultural heritage*

EN 17138:2018, *Conservation of Cultural Heritage — Methods and materials for cleaning porous inorganic materials*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

With reference to the integrity of the object surface, the nature of the analysis and the diagnosis may be described as one of the following:

3.1
non-invasive
contact between object surface and apparatus used for measurements is avoided. No material is removed from the object

3.2
micro-invasive
no visible damage on the surface

Note 1 to entry: A small amount of material is collected (1 mg or less).

3.3
invasive
consists of the removal of powder or of a fragment from the surface or from the bulk of the object

With regard to the sampled material, the analysis could be:

3.4
not destructive
samples could be re-used for several analytical purposes

Note 1 to entry: Samples scarcely modified (i.e. cross sections, spot test, ESEM sputtered samples) are also included in this category.

3.5
destructive
sampled material is modified (burned, solubilized, reacted, etc.)

3.6
effectiveness
ability in the removal of the unwanted material

3.7
harmfulness
any undesired change in the substrate to be cleaned, except the removal of the unwanted material

3.8
trial areas
set of areas where testing the planned cleaning method

3.9
unwanted material
material to be removed indicated by the restoration design because of legibility or conservation reasons

4 Abbreviations

The following list of analytical techniques is considered:

4.1 UVF: Fluorescence induced by Ultraviolet Radiation

4.2 pXRF: Portable X Ray Fluorescence

4.3 RLOM: Reflected light Optical Microscopy

4.4 UVOM: Ultraviolet Optical Microscopy

4.5 ESEM-EDS/WDS: Environmental Scanning Electron Microscopy with Energy or Wavelength Dispersive Spectroscopy

4.6 FTIR: Fourier Transmission Infrared Spectroscopy

4.7 Raman: Raman Spectroscopy

4.8 IC: Ionic conductivity

4.9 pH-m: pH measurement

4.10 WDT: Water drop test

5 General procedure (Part A)

5.1 Overview of the procedure

To facilitate the analytical evaluation of cleaning methods in each individual case the following systematic procedure is proposed. It consists of a number of consecutive stages as described in the flow chart of Figure 1. Due to the wide variability of each individual case the whole sequence proposed shall be adapted time by time and is up to the responsibility of the professional in charged.

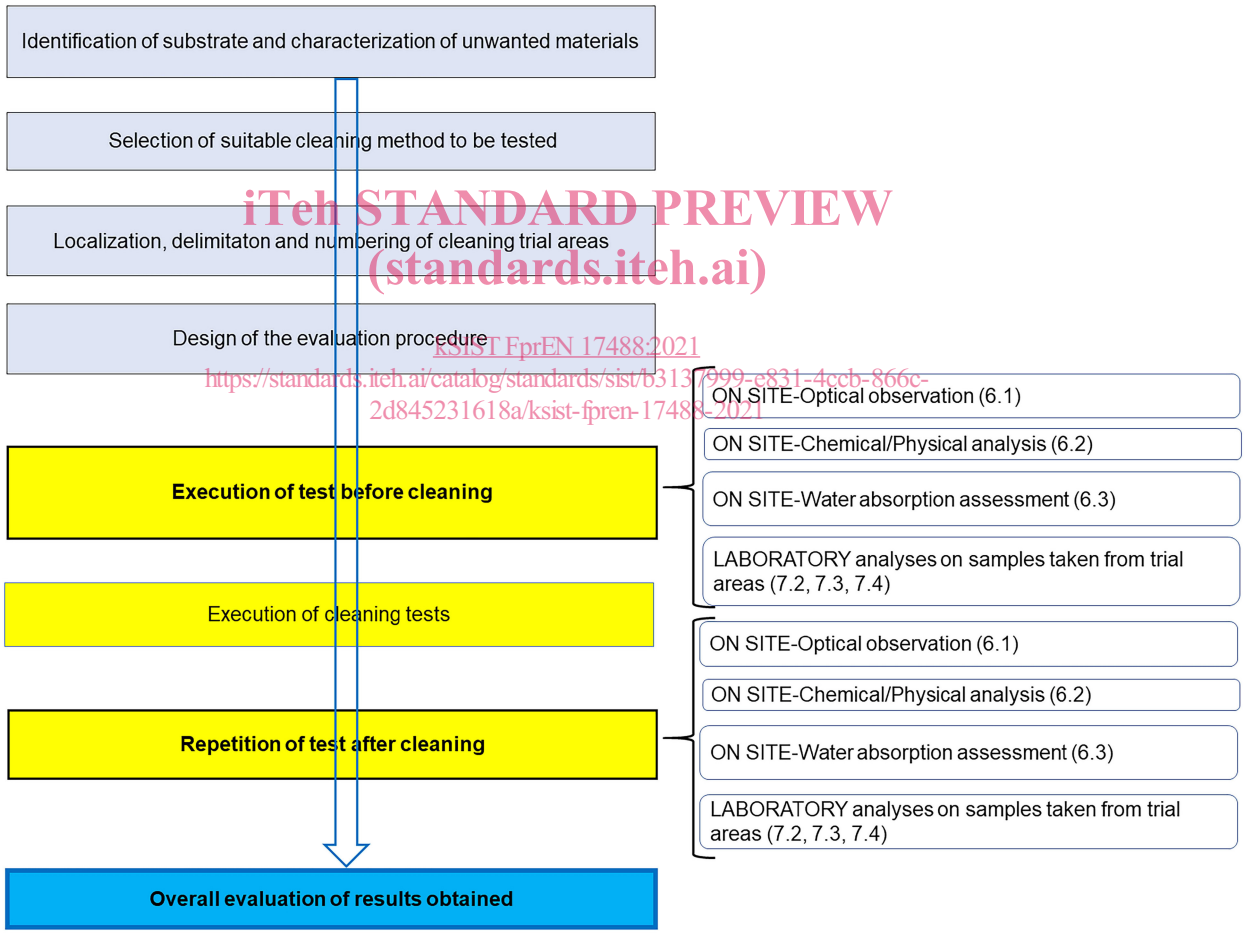

Figure 1 — Flow chart of the procedure for cleaning evaluation

5.2 Identification of substrate and characterization of unwanted materials

The chemical, mineralogical and the structural characteristics of the material constituting the object shall be preliminarily identified (EN 16515:2015) as well as the extent, nature and thickness of the unwanted materials to be removed.

The layer of unwanted materials and the altered surface of substrate shall be distinguished from each other at the investigation stage, so that the level of cleaning can be selected (see EN 17138:2018, 5.2).

The investigation should commence with the use of non-invasive systems, followed by appropriate laboratory investigations, micro-invasive or invasive, when and if required.

The preliminary investigations using non-invasive systems should be carried out on a defined test area selected as representative of the main surface material composition and cleaning problem.

The non-invasive systems considered are:

a) portable microscope for optical observations;

b) portable XRF equipment for the identification of chemical elements;

c) portable fluorescence induced by UV radiation equipment for the identification of fluorescent compounds. In the presence of organic materials, identified by UV fluorescence, a portable FTIR spectrometer should be used.

For laboratory investigations the procedure reported in EN 16515:2015 (Examination of specimens under stereo-microscope, petrographic examination, mineralogical analysis by X-ray diffraction XRD, examination by ESEM, chemical analysis) shall be performed.

The identification of organic components, not considered in EN 16515:2015, shall be performed by FTIR spectroscopy and when necessary by GC-MS (gas chromatography and mass spectrometry).

5.3 Selection of suitable cleaning methods to be tested

After the identification of the substrate and of the composition of unwanted materials a selection of suitable cleaning methods, according to the results obtained, is carried out on the “trial area”.

For the application of this document the following cleaning methods are considered (see EN 17138:2018):

a) water cleaning (restricted to the following: nebulous spray or intermittent mist spray, water spray at low pressure, steam cleaning, aqueous poultices or packs with absorbent material);

b) mechanical cleaning (restricted to the following: micro blasting, wet jet micro blasting, cryogenic cleaning);

c) chemical cleaning (application of organic solvents, application of acidic or alkaline solutions, application of chelating agents, application of surfactants, gel cleaning, application of ion exchange resins);

d) physical cleaning (laser cleaning).

5.4 Localization, delimitation and numbering of the cleaning trial areas

Trial areas should be selected in order to be representative of the whole (constituting materials, surfaces and materials to be removed); if possible, trial areas should be situated in unobtrusive locations. The trial areas should be the minimum size possible, taking into account the selected cleaning method, the significance of the object and the evaluation procedure while still being representative.

The trial test area(s) selection process should also take into account:

— the decay patterns including the type of substrate and the nature of unwanted material;

— the object size;

— the number of trial test required;

— the location where the impact of cleaning trials has the least detriment to the significance of the object, in case the trial should over-clean or otherwise damage an area;

— further optimization of the parameters of the selected cleaning process.

The description of trial area selection and procedure is reported in Annex A which is normative.

5.5 Design of the evaluation procedure

The evaluation procedure is composed of a sequence of tests, described in Clauses 6 and 7, which shall be carried out before and after cleaning respectively. The tests are also used to establish the level of cleaning that can be achieved.

A flow chart of observations and analyses is reported in Figure 2 and Table 1.

The whole sequence reported is the ideal pathway for evaluation of harmfulness and effectiveness of cleaning methods in the trial areas. The application of only part of the sequence is dependent on the special features (peculiarity) of the case study under evaluation and is the responsibility of the professional in charge. Any variation shall be specified in the test report.

Investigation and interpretation of results shall be performed by professional(s) (e.g. conservation scientist, conservators/restorers) with competencies in materials science and in the harmfulness of cleaning methods.

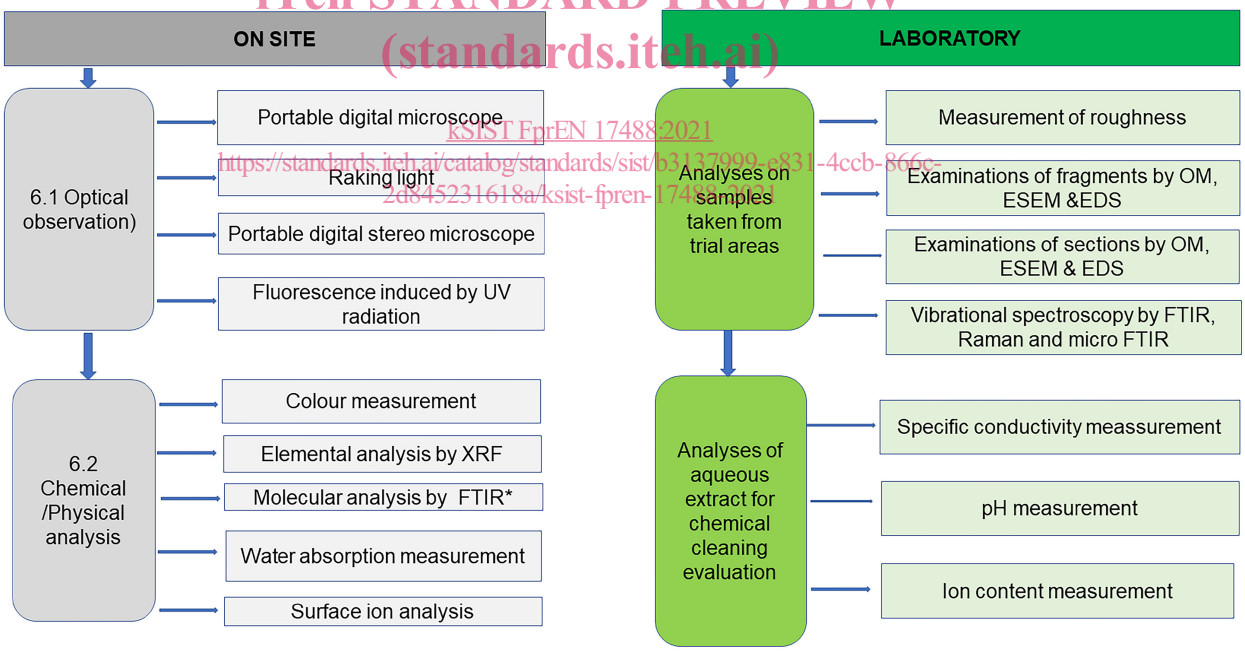

Figure 2 — Flow chart of observations and analysis related to cleaning methods

Table 1 — Observations and analysis in relation to the different cleaning classes

6 On site surface investigations **6.1 Optical observation**	**Water cleaning**	**Mechanical /physical cleaning**	**Chemical cleaning a**
6.1.1 Portable digital microscope	X	X	X
6.1.2 Raking light	X	X	X
6.1.3 Portable digital stereo microscope	X	X	X
6.1.4 Fluorescence induced by UV radiation	X	X	X
6.2 Chemical/physical analysis			
6.2.1 Measurement of colour	X	X	X
6.2.2 Elemental analysis by portable XRF	X	X	X
6.2.3 Molecular analysis by portable FTIR	X	X	X
6.2.4 Surface ion analysis	X	X	X
6.3 Water absorption assessment	X	X	X
7 Laboratory analysis on samples			
7.3.1 RLOM and ESEM-EDS on fragments	X	X	X
7.3.2 RLOM and ESEM-EDS on thin cross section	X	X	X
7.3.3 Vibrational spectroscopy (FTIR, micro-FTIR and Raman)	X	X	X
7.4 Surface morphology analysis (roughness)	X	X	X
7.5 Wet chemical analysis (on aqueous extract)			
7.5.2.2 Specific conductivity measurement	X		X
7.5.2.3 pH measurement	X		X
7.5.2.4 Ion content measurement	X		X
[a] Chemical cleaning is considered separately from the other methods due to the potential for deleterious reactions between chemicals and the unwanted materials to be removed.			

Some significant examples of potential drawbacks or side effects are listed below:

i) water cleaning can affect variations in distribution and concentrations of soluble salts (if present);

ii) mechanical/ cleaning can affect the roughness of the surface with possible change in colour, appearance and the water absorption behaviour;

iii) physical cleaning can cause discolouration of the surface;

iv) chemical cleaning can affect variations of pH, conductivity and concentrations of soluble salts.

Chemical cleaning methods, which provoke the formation of dangerous by-products (e.g. soluble salts residues, acidic substances etc.), shall generally be avoided.

In particular some of the chemicals mentioned in EN 17138:2018 shall be carefully monitored during *in situ* trial tests. We refer to:

a) strong alkaline and acid compounds, which were used in the past but are not commonly currently used for cultural heritage objects. Substances in the pH range between 5,5 and 8,0 are the most commonly specified. A notable exception to this regards the use of ammonium carbonate or bicarbonate (at pH within 8 and 10), which have been successfully used for many years particularly on limestones and marbles affected by gypsum formation crusts and on surfaces affected by proteinaceous residues, oily and greasy materials;

b) chelating agents used at the right pH (between 6 and 10) can be very selective, very effective and generally not harmful. In the case of polychrome surfaces pH shall not be above 8, which is the critical threshold limit for the dissolution of some of the most common pigments;

c) strong acid cationic resins release H^+ which can affect calcareous-based materials;

d) strong basic anionic resins release OH^-, which act negatively on mural painting substrates when pigments sensitive to an alkaline environment are present.

As a consequence of the previous considerations for chemical cleaning it is of importance to monitor any change of pH and by-product formation as a result of the selected cleaning process.

In addition to the common tests for each of the cleaning methods (water, mechanical and physical cleaning) the following measurements on the aqueous extract shall be considered:

i) pH;

ii) specific conductivity;

iii) qualitative and quantitative analyses of soluble salts.

6 On site surface investigations

6.1 Optical observation

6.1.1 Portable digital microscope

With the help of a portable digital microscope it is possible to observe the surface (stone, mortars, painting layers, finishes) and the decay products. This pre-test could help to optimize sampling. It also assists in assessment of the results of the cleaning tests and how the different systems affect the surface morphology, in the preliminary studies and during the development of the restoration works. It is possible to use several magnification and specific lights (visible, ultraviolet) to characterize different surfaces and identify also residues and overpaints.

Acquisition of digital images shall be obtained in order to document the surface condition and also identify residues.

6.1.2 Raking light

Light directed under a very small incidence angle (usually between 5° and 20°) to the object surface emphasizes its surface morphology and its relief variations. This observation provides information on the initial condition of the surface (roughness, surface defects etc.) before cleaning tests and any changes after the cleaning. The observations on target areas shall be photographed under any appropriate magnification.