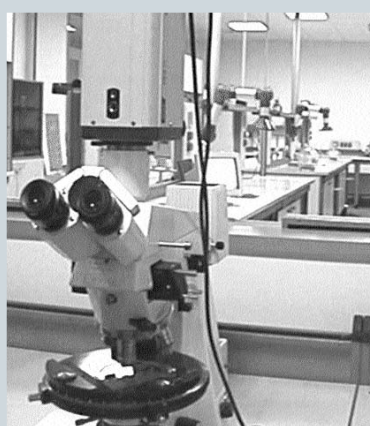


CONSERVATION OF ARCHITECTURAL HERITAGE,
HISTORIC STRUCTURES AND MATERIALS

Introduction



INTRODUCTION
ARC
LABORATORY HANDBOOK

VOLUME

1
/99

ISBN 92-9077-157-7

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International Centre for the Study of the Preservation
and Restoration of Cultural Property
Via di San Michele 13, 00153 Rome, Italy

Printed in Italy by ATEL S.p.A. ~ Roma
Graphic Design: www.ocom.it ~ Roma
Editing: Christopher McDowall and Cynthia Rockwell
Cover Design: Andrea Urland

Photographs: ICCROM Archive ~ Cover: E. Borrelli, M.T. Jaquinta, W. Schmid, A. Urland

This publication has received financial support from the World Heritage Fund.

INTRO

ICCROM PREFACE

In recent decades, the conservation of architectural heritage has increasingly drawn on new knowledge arising from the development of specialized scientific disciplines.

Condition assessment, diagnosis, conservation and restoration treatment, as well as long-term monitoring of performance, often call for specific investigation techniques and interdisciplinary study with specialized conservation laboratories.

Over the years, conservation scientists working with cultural heritage have developed analytical methods, procedures and testing techniques for the study of materials: their characteristics, causes of deterioration, alteration and decay processes. Such data are of essential support to planning any conservation or restoration activity, as well as preventive measures.

Informed use of the various available testing techniques and measuring procedures combined with the findings of other surveys and past experience allow for establishing a more complete basis for correct diagnosis, sound judgement and decision-making in the choice of the most appropriate conservation and restoration treatments within the general strategy for the historic building.

Conservation professionals today are fully aware of the fundamental role materials science and laboratory analysis play in the conservation process.

Thus a certain knowledge of the basic and most frequently used laboratory tests and a capacity to interpret their results should be an integral part of the general preparation of any professional working in the field of conservation.

With the Laboratory Handbook, ICCROM hopes to fulfil the long-felt need to provide a simple and practical guide where basic concepts and practical applications are integrated and explained.

MARC LAENEN
DIRECTOR-GENERAL

INTRODUCTION

INTRODUCTION

Concept

The ICCROM ARC Laboratory Handbook is intended to assist professionals working in the field of conservation and restoration of architectural heritage.

Target group

It has been prepared principally for architects and engineers, but may also be relevant for conservator-restorers, archaeologists and others.

Aims

- To offer simplified and selected material, structured to the needs of the target group: an overview of the problem area, combined with laboratory practicals and case studies.
- To describe some of the most widely used practices, and illustrate the various approaches to the analysis of materials and their deterioration processes.
- To facilitate interdisciplinary teamwork among scientists and other professionals involved in the conservation process.

Context

The handbooks have evolved from lecture and laboratory handouts developed and constantly updated for ICCROM's international or regional training programmes, principally the following mid-career professional courses:

- Conservation of Architectural Heritage and Historic Structures
- Technology of Stone Conservation
- Conservation of Mural Paintings and Related Architectural Surfaces
- Conservation of Architectural Surfaces

as well as in a series of collaborative laboratory activities, and consultancy and research projects.

Background

The Laboratory Handbook builds on valuable past experience. Certain ICCROM publications along similar lines, namely "Porous Building Materials: Materials Science for Architectural Conservation" by Giorgio Torraca (1982), and "A Laboratory Manual for Architectural Conservators" by Jeanne Marie Teutonico (1988) have been among the main references.

The long-term experience accumulated from other institutions, that of renowned experts and conservation/restoration practice in general have naturally also been a point of reference, as has the process of designing laboratory session modules for ICCROM courses in recent years; the latter has been enriched by feedback both from participants and from contributing leading specialists.

Structure

The concept behind the Laboratory Handbook is modular. Thus, it has been conceived as a set of independent volumes, each of which will address a particular subject area. The volumes relate particularly to topics covered in the International Refresher Course on Conservation of Architectural Heritage and Historic Structures (ARC), which are as follows:

- **DECAY MECHANISMS, DIAGNOSIS:** guiding principles of materials science, external climatic risk factors, atmospheric pollution, humidity, bio-deterioration, investigation techniques, surveying

- **CONSERVATION AND RESTORATION TREATMENTS OF BUILDING MATERIALS AND STRUCTURES**
THEIR TESTING, MONITORING AND EVALUATION: timber; earthen architectural heritage, brick, stone, mortars, metals, modern materials, surface finishes.
As an issue of broader interest, practical information and guidelines for setting up architectural conservation laboratories are also scheduled for publication.

In general, each volume includes:

- introductory information
- explanations of scientific terms used
- examples of common problems
- types of analysis (basic principles)
- practicals (laboratory tests)
- applications (case study)
- bibliography

Principles are explained, and some longstanding methodologies described. Up-to-date information on techniques, instruments and widely used reference standards has been included.

Further information on practical techniques not strictly related to laboratory work is also provided in some cases.

The practicals - involving basic tests and simple analyses - are conceived as part of ICCROM's laboratory sessions.

It is understood that architects will not generally be performing laboratory analyses on their own. However, knowledge of the types of analysis available for obtaining specific data, their cost, reliability and limitations is essential for today's conservation architects. They should also be aware of sampling requirements and techniques, able to understand and interpret results, and effectively communicate them to other colleagues in interdisciplinary teams.

The individual volumes are being prepared by various authors, depending on the subject and their specialization. The Scientific Committee is composed of specialists in the relevant fields, who also have strong ties with ICCROM through direct involvement in the training courses.

Special features

Progress in conservation science and technology means that currently available information must be regularly evaluated and updated. This is why the Handbook has been structured as a series of volumes.

This will allow:

- the authors to periodically update specific volumes to reflect changing methodology and technology in an easy, time-saving and economical way, thanks to the digital reprinting process
- the users to work selectively with the volume relating to the particular problem they are facing
- new volumes to be gradually added to the set in line with developing needs, until all the relevant subjects have been covered by the series.

We are aware that the information provided is not all-embracing but selective. It is hoped that feedback from users will assist us in improving and continually adapting the project to changing needs.

The project team - Ernesto Borrelli and Andrea Urland - welcomes any constructive criticism, comments and suggestions that might help us achieve this goal.

ANDREA URLAND
ARC PROJECT MANAGER

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ACKNOWLEDGEMENTS

This publication was made possible thanks to the financial contribution of the UNESCO World Heritage Centre.

The authors would especially like to express their gratitude to the members of the Scientific Committee, who kindly agreed to give this publication their support, sharing their expertise by reviewing the draft texts and providing valuable comments and suggestions.

The authors further wish to recognize the scientific collaboration of Beatrice Muscatello, the precious contribution of the advisors and the text editing by Christopher McDowall and Cynthia Rockwell.

They are equally grateful to the ICCROM staff for their whole-hearted collaboration and to those at the UNESCO World Heritage Centre for their support.

And finally we wish to thank all the others who have, in some way, contributed to the preparation and completion of this publication.

1998 - 99 VOLUMES:

1. Introduction
2. Porosity
3. Salts
4. Binders
5. Colour specification and measurement

INTRO ICCROM

The International Centre for the Study of the Preservation and Restoration of Cultural Property (ICCROM) is a leading voice in the conservation of cultural heritage around the world. Founded by UNESCO in 1956, and based in Rome, ICCROM is the only intergovernmental organization concerned with conserving all types of movable or immovable heritage.

ICCROM takes action today for heritage tomorrow. Its broad-ranging activities span five continents and cover a wide range of cultural heritage, from cave painting to sculpture, earthen architecture, plastered façades, libraries, archives and entire historic centres.

ICCROM tackles the challenges facing cultural heritage – neglect, pollution, catastrophe, theft or decay – and strengthens the conditions for its effective conservation. It provides an international platform for debate while also tailoring activities to regional needs.

It achieves this by collecting and disseminating information; coordinating research; offering consultancy and cooperation in field conservation and restoration; providing professional training; and promoting awareness of the social value of cultural heritage preservation amongst all sectors of society.

Above all, ICCROM integrates the conservation of our cultural heritage into global approaches to sustainable human development and community service.



Dr Harold
Plenderleith
(1898-1997)

INTRO ICCROM's LABORATORY: A BRIEF HISTORY

The new ICCROM Laboratory, inaugurated in 1997, is dedicated to Dr Harold Plenderleith, the Organization's founding director.

In 1966, during his tenure, a first basic laboratory was set up for didactic purposes at the original headquarters in Via Cavour.

In the 1980s, after ICCROM had moved to Via di San Michele, a new and better-equipped laboratory was installed under the coordination of Dr Giorgio Torraca, then ICCROM's Deputy Director. Many activities, including research, were launched in the framework of the newly created "Training and Research Units". Later, a smaller specialized laboratory was also developed by Jeanne Marie Teutonico for the architectural conservation courses.

ICCROM's current Laboratory (about 200 m²) is organized as a series of specialized areas, grouping together all the previous activities. The works were subsidized by a major financial contribution from the Italian Government.

The improved level of equipment and furnishing offers opportunities to expand and further raise the standard of work.

ISBN 92-9077-157-7



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CONSERVATION OF ARCHITECTURAL HERITAGE,
HISTORIC STRUCTURES AND MATERIALS

Porosity



Ernesto Borrelli

ARC
LABORATORY HANDBOOK
POROSITY

VOLUME 2 / 99

The ICCROM ARC Laboratory Handbook is intended to assist professionals working in the field of conservation of architectural heritage and historic structures. It has been prepared mainly for architects and engineers, but may also be relevant for conservator-restorers or archaeologists.

It aims to:

- **offer an overview of each problem area combined with laboratory practicals and case studies;**
- **describe some of the most widely used practices and illustrate the various approaches to the analysis of materials and their deterioration;**
- **facilitate interdisciplinary teamwork among scientists and other professionals involved in the conservation process.**

The Handbook has evolved from lecture and laboratory handouts that have been developed for the ICCROM training programmes. It has been devised within the framework of the current courses, principally the International Refresher Course on Conservation of Architectural Heritage and Historic Structures (ARC).

The general layout of each volume is as follows: introductory information, explanations of scientific terminology, the most common problems met, types of analysis, laboratory tests, case studies and bibliography.

The concept behind the Handbook is modular and it has been purposely structured as a series of independent volumes to allow:

- **authors** to periodically update the texts;
- **users** to work selectively with the volume relating to the particular problem they are facing
- **new volumes** to be gradually added in line with developing needs.

1998 - 99 volumes:

(1) Introduction, **(2) Porosity**, (3) Salts, (4) Binders, (5) Colour specification and measurement

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ISBN 92-9077-157-7

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Printed in Italy by ATEL S.p.A. ~ Roma

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HISTORIC STRUCTURES AND MATERIALS

ARC
Laboratory Handbook

Porosity

Ernesto Borrelli



Rome, 1999

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1 INTRODUCTION

Many building materials, both natural (stones) and artificial (bricks, lime and cement mortar) contain a certain volume of empty space. This is distributed within the solid mass in the form of pores, cavities, and cracks of various shapes and sizes. The total sum of these empty spaces is called porosity, a fundamental characteristic of building material that affects its physical properties (durability, mechanical strength, etc.).

The characteristics of pores in rocks depend mainly on their genesis (i.e. igneous^①, sedimentary^②, metamorphic^③ etc.) whereas the porosity in man-made building materials depends on their manufacture .

The knowledge of the pore structure is an important parameter for characterizing materials, predicting their behaviour under weathering conditions, evaluating the degree of decay and establishing the effectiveness of conservation treatments.

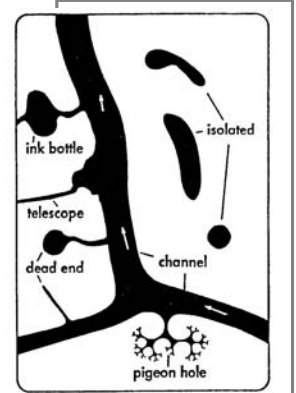




Fig. 1 - Pore models
(taken from Fitzner)

2 CLASSIFICATION OF PORES

Pores can essentially be classified according to their typology, geometry and size.

Typology

- **CLOSED PORES:** pores completely isolated from the external surface, not allowing the access of water in either liquid or vapour phase. They influence neither permeability nor the transport of liquids in materials but do affect their density and mechanical and thermal properties .
- **OPEN PORES:** pores connected with the external surface of the material and therefore accessible to water have a direct bearing on deterioration phenomena. Open pores permit the passage of fluids and retain wetting liquids by capillary action. They can be further divided into dead-end or interconnected pores (Fig. 1) .

Geometry

Pores can also be classified according to their shape:

- **SPHERICAL PORES, CYLINDRICAL PORES AND ELONGATED PORES**

or according to their genesis (Fig.2; photos 1-4 on inside back cover):

- **BASIC PORES:** pores inherent to the process of rock formation;
- **DISSOLUTION PORES:** pores deriving from the chemical dissolution of carbonates (see transformation of carbonates into soluble bicarbonates^④) sulphates, and organic materials (by transformation into CO₂);
- **FRACTURE PORES:** pores and microcracks deriving from intro and inter-crystalline mechanical fracture, linked to the tectonic deformation of rocks and due to stress caused by applied loads;

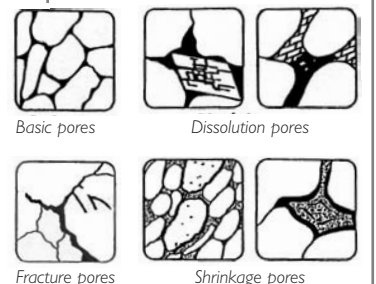


Fig. 2 - Pore types (taken from Fitzner)

① Rocks that have solidified from molten rock material both below and on the earth's surface (e.g. granite, gabbro, basalt, tuffs).

② Rocks formed by the accumulation and consolidation of sediment at relatively low temperature and pressure (sandstone, limestone, travertine).


③ Rocks that are the result of the structural transformation in the solid state of pre-existing rocks under conditions of high temperature and pressure (marble, gneiss, schist).

④ See Laboratory Handbook Salts p. 7

- **SHRINKAGE PORES:** pores deriving from the contraction of the various components of masonry materials, mainly artificial ones e.g. the shrinkage of mortars due to rapid water loss.

Size

There is considerable variability in pore sizes; they vary from a few angstrom^⑤(Å) to several millimetres. Pores of greater dimensions are defined as cavities rather than pores, and do not contribute to capillary action. Pores with radii^⑥ less than 10 angstrom are not considered permeable.

There are conflicting views concerning pore size classification. In practice, when conservation scientists speak of porosity, they are not generally referring to the values defined below, but to a dividing line of < 2.5 µm and > 2.5 µm between microporosity and macroporosity, which is more realistic when dealing with building material^⑦ .

International standards (IUPAC) s classify pores according to their radius as

MICROPORES:	radius	< 0.001 µm	(< 10 Å)
MESOPORES:	radius between	0.001 µm and 0.025 µm	(10 Å and 250 Å)
MACROPORES:	radius	> 0.025 µm	(> 250 Å)

The percentage distribution of pores of differing radius within the material is an extremely important parameter for the evaluation of its behaviour in contact with water and therefore for the forecast of freeze-thaw cycles, chemical reactivity etc.

There is obviously a great variation in porosity from one material to another. Igneous (e.g. granite, basalt) and metamorphic (e.g. marble, gneiss) rocks are not very porous with maximum porosity between 1% and 2%. Unless they are fractured, these low-porosity rocks are scarcely permeable. A lot of sedimentary rocks, however, and particularly calcarenites, have high porosity with maximum values even reaching 45%.

The % porosity and pore types of some common rocks are summarized in the following table:

Rock type	Genesis	Geological formation		% porosity (average value)	Predominant pore type
		pressure	temperature		
basalt	igneous	low	very high	≅ 1 - 3	macro
granite	igneous	high	very high	≅ 1 - 4	micro
tuff	igneous	low	high	≅ 20 - 30	micro
gneiss	metamorphic	high	high	≅ 0,4 - 2	micro
marble	metamorphic	high	high	≅ 0,2 - 0,3	micro
slate	metamorphic	high	medium-high	≅ 0,1 - 1	micro
coral stone	sedimentary	low	low	≅ 40 - 50	macro
limestone	sedimentary	low	low	≅ 15 - 20 ± equal	micro/macro
sandstone	sedimentary	low	low	≅ 10 - 15	macro

Table 1 - Porosity and pore type of some common rocks

⑤ 1 Å = 10⁻¹⁰m,
or 1µm = 10⁴ Å.

⑥ At a first approximation, all pores of any shape are considered equivalent to round pores of equal nature. The radius of the equivalent pores can be measured by several methods (see section 6).

⑦ These values are proposed by Russell (1927), De Quervain (1967), Ashurst & Dimes (1977), Zehnder (1982) and Veniale & Zezza (1987).

POROSITY

3 BASE TERMS

- **PORE VOLUME (V_p)**

is the fraction of the total volume of a solid occupied by the pores (i.e. the empty space of a solid).

- **APPARENT VOLUME (V_a)**

is the volume of a solid including the space occupied by pores.

(a piece of stone measuring 5 cm each side has an apparent volume of $5 \times 5 \times 5 = 125 \text{ cm}^3$)

- **% TOTAL POROSITY (P)**

is defined as the ratio between the volume of the pores (V_p) and the *apparent volume* (V_a) expressed as a percentage.

$$\% P = 100 \times (V_p / V_a)$$

(a piece of limestone measuring 5 cm each side with a total porosity of 22% contains 27.5 cm^3 of empty space)

- **OPEN PORE VOLUME (V_{op})** is the volume occupied by open pores.

- **% OPEN POROSITY**

is less than or equal to the total porosity and is defined as the ratio between the volume of the open pores (V_{op}) and the apparent volume (V_a) expressed as a percentage.

$$\% P = 100 \times (V_{op} / V_a)$$

It is also known as effective porosity: percentage of interconnected pore space

(a piece of limestone measuring 5 cm each side with a total porosity of 22% that contains 20 cm^3 of open pores has an open porosity of 16 %)

- **REAL VOLUME (V_r)**

is the difference between apparent volume (V_a) and pore volume (V_p)

$$V_r = V_a - V_p$$


(a piece of stone measuring 5 cm each side with a total porosity of 22% has a real volume of 97.5 cm^3)

- **SPECIFIC SURFACE**

(S_s) is the surface (m^2) of the walls of the open pores and is expressed per unit volume of the material (m^2/m^3).

4 MOVEMENT OF WATER IN POROUS BUILDING MATERIALS

The study of porosity is fundamental for understanding phenomena of water transport within pore structure and interactions between materials and water.

Water can penetrate a solid because there are interconnected channels (pores) inside the solid that facilitate its transportation. Stone material can absorb moisture from the environment in **vapour form** depending on the relative humidity and in **liquid form** when exposed to the direct action of water (rainfall, rising damp from the soil and water vapour condensation from the air) .

IN LIQUID FORM:

- a) By capillary suction: when an initially dry porous material comes into contact with water; it gets progressively wetter. First, it fills up the smaller pores, and then creates a liquid film on the surface of the larger pores, eventually filling these too;
- b) By diffusion, due to the passage of water from a higher to a lower water content area;
- c) By osmosis: when salts are present in water; they are dissociated into electrically charged particles (ions) that attract water through electric force.

IN VAPOUR FORM:


- a) By diffusion as vapour; from pores with a high water vapour content to pores with a lower one;
- b) By hygroscopic absorption, which can occur even at temperatures above dew point^⑧. This phenomenon is accentuated in the presence of soluble salts that are hygroscopic and can absorb water also under average conditions of relative humidity;
- c) By condensation: when the temperature of the material is less than dew point, water vapour then condenses within the pores. In small pores, condensation can take place before the temperature reaches dew point.

^⑧ The temperature at which the water vapour in the air is saturated. As the temperature falls the dew point is the point at which the vapour begins to condense as droplets of water.


POROSITY

5 DETERIORATION

The size of the pores, their distribution and geometry are fundamental factors in determining the properties of materials and their suitability for building applications.

The degree of porosity in different materials can be a positive characteristic for their use in some applications (e.g. very porous plaster allows for water vapour transmission) but may have adverse effects on their performance in others (e.g. very porous stone generally deteriorates more easily) .

One of the main causes of stone decay is the interaction between water and the porous structure. Water adsorption can induce weathering on stone materials in several ways:

- a) by chemical reaction (e.g. aggressive pollutants);
- b) by a physical mechanism: through mechanical stress due to freeze/thaw cycles;
- c) by acting as a transport medium for salts in dissolution and recrystallization processes within the pore space;
- d) by providing an essential substrate for biological growth .

6 METHODS OF MEASUREMENT

The three structural properties that are fundamental in describing porous materials are **porosity**, **pore size distribution** and **specific surface**; pore shape is also significant but less easily quantifiable.

As these properties are geometrical, they can be evaluated by **direct** observation. Other methods of assessment are termed **indirect**, as they are obtained from calculations based on other parameters 10.

Direct methods

Those methods that make it possible to directly observe the porous structure, using either a petrography microscope or scanning electron microscopy (SEM).

A) PETROGRAPHY MICROSCOPE ANALYSIS

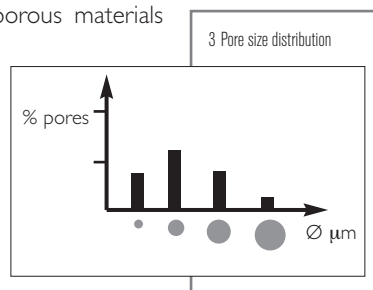
Direct observation by microscope of thin sections of porous materials makes it possible to evaluate total porosity which includes closed pores. This is a traditional method of studying the porosity on thin sections 9 of material, enabling the calculation of the area occupied by pores as a percentage of the total surface area under examination and, at the same time, recording their size distribution (Fig. 3).

The advantage of this technique is that it permits the direct quantification of what is visible. A further important characteristic is that specific data such as the size distribution of larger pores can only be obtained with this method 11.

It is limited in that a large number of thin sections from different layers and angles of the sample must be examined in order to obtain a statistically viable result from these measurements.

Although this analysis is suitable for larger pores, pores with radii smaller than 4 μm are not measurable. This depends on the resolution of the optical microscope, which limits the measurement to pores with radii ranging between 4 and 500 μm 12.

When combined with the digital analysis of the images, this technique automatically calculates the area occupied by pores comparing it to that occupied by the solid. For this reason, it is possible to carry out a large number of measurements and therefore obtain statistically valid data.



B) SCANNING ELECTRON MICROSCOPY ANALYSIS (SEM)

This is an effective technique for analysing materials that have a large number of micropores. It can be combined with the digital analysis of the images and computer-aided techniques to reconstruct three-dimensional images (3D-modelling), as opposed to the two-dimensional system in petrography microscopy. This makes it possible to delineate the empty space occupied by the pores and obtain direct information on their shape, size and three-dimensional distribution.

The advantage of this method is that it does not rely on fictitious pore models, which are generally assumed to be cylindrical (see Indirect methods p.8), but provides a true description of the pore structure. It is again necessary to examine a large number of thin slices 10 of the material for the result to be representative 13.

9 These are obtained by embedding the sample in a synthetic resin and cutting across its outer surface. The sections are then polished until their thickness is less than 30 μm . They provide information on mineralogical composition and microstructural characteristics such as porosity.

10 dimensions: 10mm x 10mm x 2mm.

The problem of the representativity of the sample is the same as in the preceding case. This is a highly sophisticated method and is only used in specific areas of research 14.

Indirect methods

Indirect methods measure certain derived properties, such as density, permeability to fluids (liquid or gas), liquid imbibition rates, adsorptive capacity and so on, in order to evaluate the porous structure 15.

Mercury porosimetry and nitrogen adsorption measurement are the two most common indirect methods in which porosity is essentially correlated to the pressure necessary to introduce a fluid into the pores of the material.

A) MERCURY POROSIMETRY MEASUREMENT 16

This technique makes it possible to measure the distribution of pore sizes inside the material. The mercury 1 is forced inside by applying steadily increasing pressure.

The principle of measurement is based on Washburn's equation:

$$r = 2\sigma \cos\theta / P$$

where:

P = pressure exercised

σ = surface tension of mercury

θ = contact angle between the mercury and the solid

r = pore radius

The distribution of the pores, as well as the total porosity values, the real and apparent density and the volume of intrusion can be obtained from the proportionality between pressure necessary for penetration and the dimension of the pores. The theory upon which the Washburn equation is based assumes that all pores are cylindrical. In the case of ink bottle pores, for example, their true dimensions are unobtainable, as the measurement only refers to the radius of the pore entrance.

Current instruments allow the pressure to reach 4000 bar 17 (400 MPa), which permits them to fill macro and micropores. However, this method cannot be recommended for very fragile materials.

The amount of sample necessary for the analysis ranges from 0.5 to 1 g and the current cost is around 150 US dollars per test.

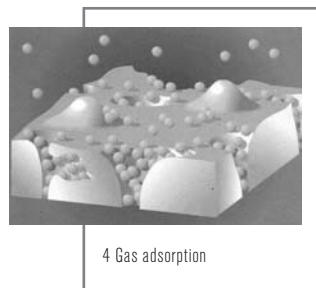
1 Mercury is used because of its non-wetting properties.

17 1 bar = 0.9678 atm

B) MEASUREMENT OF NITROGEN ADSORPTION (Fig.4) 17


This procedure is based on the quantity of gas adsorbed by a porous material at constant temperature and at increasing levels of pressure. A curve is obtained called the isotherm of adsorption which is correlated to the distribution of pore sizes within the solid. Various fluids can be used depending on the dimensions of the pores to be measured, but nitrogen has given the best results making it possible to determine micropores.

A gram of sample is necessary for the analysis.



Simple methods

All the previously mentioned methods, both direct and indirect, have the advantage of requiring only a small quantity of sample for the analysis but the initial cost of the instruments is extremely high.

Other indirect methods to study porosity can be used, based on the derived properties of the material, by measuring, for example, *water absorption by total immersion*, *water desorption*, *water absorption by capillarity*, and *water vapour permeability*. These tests, which are easy to carry out, make it possible to observe the behaviour of building materials in contact with water  15. Although simple, they are limited by the necessity to work on samples of a precise geometrical shape and size (e.g. cubes, cylinders, etc.). It is therefore seldom possible to take samples of this nature from a historic structure. Furthermore, several samples must be analysed to obtain a statistically viable result.

A) WATER ABSORPTION TEST BY TOTAL IMMERSION

This test measures the water absorption rate and the maximum water absorption capacity. The total quantity of water absorbed is related to the total open porosity, while the kinetics of the process depend principally on the distribution of the pore sizes.

B) WATER DESORPTION TEST

This measures the evaporation rate of saturated samples at room temperature and pressure. This is an extremely useful test that indicates the drying properties of the materials (i.e. whether they will dry quickly or remain wet for a long time). The presence of ink bottle pores, for example, has an adverse effect on the drying process due to their particular geometry.

C) WATER ABSORPTION BY CAPILLARITY

This test measures the capillary rise of water; the most common form of liquid water migration in building materials. It is inversely proportional to the diameter of the pores; the smaller the diameter, the greater the capillary absorption. Certain building materials, because of their low capillary absorption, are selected for specific uses, for example, as a barrier where masonry is in contact with the soil or as a base for wood fixtures to protect the structure from rising damp. (photo 5)



All the above tests may be correlated to the behaviour of masonry in contact with liquid water:

D) WATER VAPOUR PERMEABILITY

The permeability test is very important to predict the water vapour transmission capacity of added materials, especially plasters. It measures the quantity of water vapour that passes through a given thickness of material, limited by parallel surfaces, as a result of the partial difference in pressure of the water vapour between the two sides. The test is also a useful method to evaluate the suitability of paints as finishing layers which provide protection without reducing water vapour transmission.

Although all these measurements provide information relating to porosity, they are often used to make comparisons between quarried, weathered or treated stone materials.

MEASURING APPARENT VOLUME AND OPEN PORE VOLUME OF A STONE SAMPLE

Aim

The aim is to become familiar with a measuring procedure to obtain % open porosity, using basic equipment.

Equipment and chemicals

Oven

Technical balance

Desiccator

Beakers or plastic
containers

Soft cloth

Glass rods

Silica gel

Deionized water

① A cylinder of suitable size
should be chosen for the sample
with a scale that makes it easy to
read the change in volume.

Procedure

A) APPARENT VOLUME

- If the sample has a *regular* form (i.e. cube, cylinder), it is sufficient to measure the size and calculate the geometrical volume which, in this case, corresponds to the *apparent volume* of the sample.

In the case of small samples of *irregular* shapes and sizes :

- Wash the sample in deionized water before beginning this test to eliminate powdered material from the surface.
- Dry the sample in the oven for 24 hours at 60°C and then place it in a desiccator with dry silica gel to cool off.
- Weigh the sample. Then, repeat the drying process until the mass of the sample is *constant* (i.e. until the difference between 2 successive measurements, at an interval of 24 hours, is not more than 0.1% of the mass of the sample).
- Once the sample has been completely dried and the *constant mass* recorded (m_c), place it in a container or beaker on a base of glass rods and slowly cover with deionized water until the sample is totally immersed with about 2 cm of water above it.
- Take the sample out of the container 8 hours later; blot it quickly with a damp cloth to remove surface water and record its weight.
- Re-immerses the sample in the water and repeat the measurement until the difference in weight between 2 successive measurements at 24-hour intervals is less than 1% of the amount of water absorbed.
- Record the mass of the wet samples (m_s) and the time of measurement on the data sheet.
- Put the saturated sample in a graduated cylinder① filled with deionized water and measure the increase in volume indicated on the cylinder.

Calculation

Apparent volume V_a (cm³) corresponds to the observed increase in the volume of water measured on the graduated cylinder.

Procedure

B) OPEN PORE VOLUME

- Use the values recorded (m_c and m_s) to calculate open pore volume and % open porosity.

Calculation

- a) The open pore volume V_{op} (cm^3) corresponds to the volume of water absorbed by the sample. Since the density of water is 1 g/cm^3 at 4°C , the difference in weight (g) of the sample before and after being saturated corresponds to the open pore volume:

$$V_{op} = m_s - m_c$$

where

m_s = the mass of the saturated sample (g)

m_c = the dry mass of the sample (g)

V_{op} = open pore volume (cm^3)

- b) To calculate the % open porosity, use the following formula:

$$\% \text{ open porosity} = 100 \times (V_{op}/V_a)$$

where

V_{op} = calculated open pore volume (cm^3)

V_a = calculated apparent volume (cm^3)

WATER ABSORPTION BY TOTAL IMMERSION

Equipment and chemicals

Oven

Technical balance

Chronometer

Desiccator

Beakers or plastic containers

Soft cloth

Glass rods

Silica gel

Deionized water

Definitions

WATER ABSORPTION BY TOTAL IMMERSION: the quantity of water absorbed by a material immersed in deionized water at room temperature and pressure at successive time intervals (i.e. the rate of water absorption), expressed as a percentage of the dry mass of the sample.

WATER ABSORPTION CAPACITY: the maximum quantity of water absorbed by a material at room temperature and pressure under conditions of saturation, again expressed as a percentage of the dry mass of the sample.

Aim

The measurement of water absorption is a useful laboratory test to characterize porous building materials, evaluate the degree of deterioration, and monitor the effects of conservation treatments. Here is a simple method for such measurement that gives reliable results without the use of sophisticated equipment.

Procedure

Samples should be of a regular shape (cubes, cylinders, or prisms). In the case of cubes, the side should neither be less than 3 cm nor greater than 5 cm, so that the value of the ratio S/V (total surface to apparent volume) is between 2 and 1.2 cm^{-1} .

The number of samples required depends on the heterogeneity of the material being tested. In general a series of at least three samples is recommended. These should be as similar as possible in terms of physical properties and condition.

- Wash the samples in the deionized water before beginning this test in order to eliminate powdered material from the surface.
- Dry the samples in the oven for 24 hours at 60°C (this relatively low drying temperature will prevent the deterioration of organic substances in the case of treated samples). Then place the samples in a desiccator with dry silica gel to cool off.
- Weigh the samples. Repeat the drying process until the mass of the each sample is constant, that is, until the difference between 2 successive measurements, at an interval of 24 hours, is no more than 0.1% of the mass of the sample.
- Once the samples have been completely dried and the *constant mass* recorded (m_o), place them in a container or beaker; on a base of glass rods and slowly cover with deionized water until they are totally immersed with about 2 cm of water above them.
- At programmed intervals of time ^①, take each sample out of the container; blot it quickly with a damp cloth to remove surface water; then record the mass of the wet samples (m_i) and the time of measurement on the data sheet.
- Re-immerses the samples in water and continue measuring until the difference in weight between 2 successive measurements at 24-hour intervals is less than 1% of the amount of water absorbed.
- At this point, take the samples out of the water and dry them again in an oven at 60°C until they have reached *constant mass* (as above). Record this value (m_d) on the data sheet. Proceed with the calculations.

Calculation

- a) At each interval, the quantity of water absorbed with respect to the mass of the dry sample is expressed as:

$$M_i\% = 100 \times (m_i - m_o)/m_o$$

where

m_i = weight (g) of the wet sample at time t_i

m_o = weight (g) of the dry sample

- b) Record these values on a data sheet and on a graph as a function of time.

- c) Again, using the figures from the data sheet calculate the water absorption capacity (WAC) with the following formula:

$$WAC = 100 \times (m_{\max} - m_d)/m_d$$

where

m_{\max} = the mass (g) of the sample at maximum water absorption

m_d = the mass (g) of the sample after re-drying at the end of the test

As an example, a generic series of samples are recorded in Fig. I and Table I.

① The length of the intervals during the first 24 hours depends on the absorption characteristics of the materials:

a) Stone and brick should be weighed after the first 5 minutes of immersion and then every hour for the first 3 hours.

b) Mortar samples should be weighed a few minutes after immersion, and then at increasing intervals (15 min, 30 min, 1 hour, etc.) for the first 3 hours.

All samples should then be weighed 8 hours after the beginning of the test and then at 24-hour intervals until the quantity of water absorbed in two successive measurements is not more than 1% of the total mass.

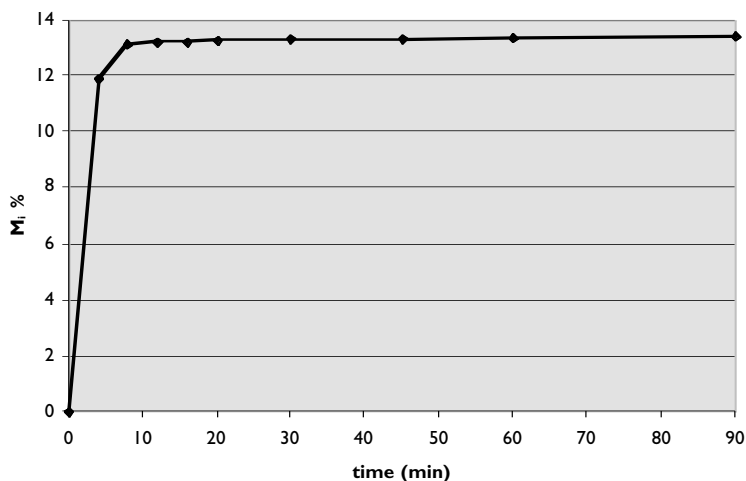


Fig. I - Water absorption by total immersion

MEASUREMENT INTERVALS	SAMPLE 1 $m_o = 124.70$ g		SAMPLE 2 $m_o = 113.88$ g		SAMPLE 3 $m_o = 122.03$ g		MEAN VALUES
t_i (min)	m_i (g)	M_i (%)	m_i (g)	M_i (%)	m_i (g)	M_i (%)	\bar{M}_i (%)
0	124,70	0,00	113,88	0,00	122,03	0,00	0,00
4	138,28	10,88	127,78	12,21	137,44	12,62	11,90
8	140,97	13,05	128,97	13,25	137,93	13,03	13,11
12	141,08	13,14	129,12	13,38	138,01	13,09	13,20
16	141,12	13,16	129,15	13,41	138,06	13,13	13,23
20	141,19	13,22	129,22	13,47	138,11	13,18	13,29
30	141,21	13,24	129,21	13,46	138,15	13,21	13,30
45	141,23	13,25	129,29	13,53	138,19	13,24	13,34
60	141,25	13,27	129,30	13,54	138,23	13,27	13,36
90	141,27	13,29	129,35	13,59	138,29	13,32	13,40
1440	141,35	13,35	129,42	13,65	138,31	13,34	13,45
2880	141,95	13,83	129,74	13,93	138,75	13,70	13,82

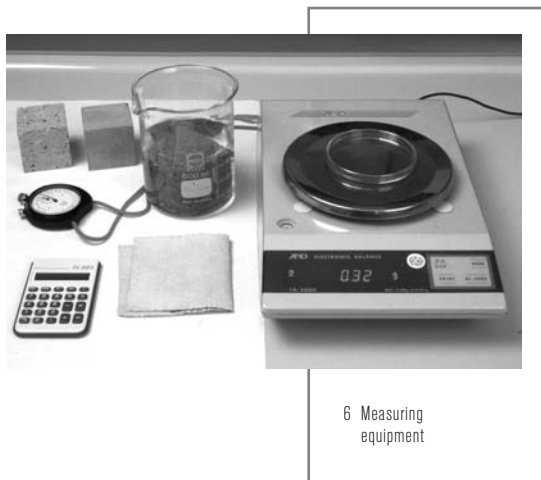
Table 1 - Water absorption by total immersion

where

m_o = weight of dry sample

m_i = weight of the wet sample at time t_i

M_i = $100 \times (m_i - m_o)/m_o$ calculated for each interval time and for each sample



POROSITY

WATER DESORPTION

Equipment and chemicals

Oven

Balance

Chronometer

Desiccator

Soft cloth

Glass rods

Silica gel

Definition

The variation in water content of the material, expressed as a percentage of the dry mass of the sample at a constant temperature and under fixed conditions of humidity, is measured over a period of time.

Aim

The aim is to indicate the drying capacity of porous materials.

Procedure

Samples should be of a regular shape (cubes, cylinders, or prisms). In the case of cubes, the side should neither be less than 3 cm nor greater than 5 cm, so that the value of the ratio S/V (total surface to apparent volume) is between 2 and 1.2 cm^{-1} .

The number of samples required depends on the heterogeneity of the material being tested. In general a series of at least three samples is recommended. These should be as similar as possible in terms of physical properties and condition.

- Soak the samples in water to the point of saturation (*see Practical 1*). Blot them with a damp cloth to remove surface water and weigh them (m_0).
- Place each sample inside the desiccator containing anhydrous silica gel with a cobalt chloride indicator. Store at a constant room temperature of $20 \pm 1^\circ\text{C}$.

The size of the desiccator and the number of samples for each desiccator must be determined, through preliminary tests, by the nature of the material, so that the relative humidity corresponding to the equilibrium of the silica gel is maintained within the desiccator during the whole testing phase. Check the relative humidity by placing a cobalt chloride strip indicator on the wall of the desiccator. The indicator must always remain blue throughout the test. If not, quickly replace the silica gel at the bottom of the desiccator.

- Remove samples periodically from the desiccator and weigh them. During the first 24 hours the length of interval will depend on the evaporation characteristics of the material, which is determined by a preliminary test to identify the initial evaporation rate.

Procedure continued

- Repeat weighing at 24-hour intervals, until the following formula has been verified:

$$1.0 \geq (m_o - m_{i-1}) / (m_o - m_i) \geq 0.90$$

where

m_o = mass (g) of sample at time t_o (hrs)

m_{i-1} = mass (g) of sample at time t_{i-1} (hrs)

m_i = mass (g) of sample at time t_i (hrs)

- Proceed with the desiccation of the samples in an oven at $60 \pm 5^\circ\text{C}$, until constant mass is reached. Mass is considered constant when the difference between two successive measurements at 24-hour intervals is less than or equal to 0.01% of the mass of the dry sample.
- Plot the experimental values to obtain a “drying curve” (water content as a function of time) (Fig. 1).

Calculation

The residual water content Q_i of the sample at time t_i is calculated with the following formula:

$$Q_i = 100 \times (m_i - m_{of}) / m_{of}$$

where

Q_i = water content at t_i expressed as a percentage of the final dry mass

m_i = mass (g) of sample at t_i (hrs)

m_{of} = mass (g) of the desiccated sample at the end of the drying test

Record the values of Q_i in a graph versus time t_i and draw the relative curve^①.

① The initial water content corresponds to the value of the absorption capacity WAC determined during the process of total immersion.

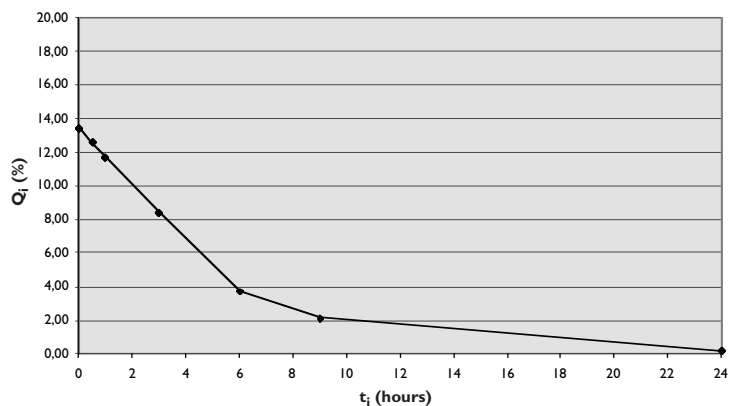


Fig. 1 - Water content versus time

PRELIMINARY SURVEY FOR THE CONSERVATION OF THE NORTH FAÇADE IN THE CHURCH OF S. MARIA FORMOSA-VENICE

(The author acknowledges the kind contribution of W. Schmid, ICCROM)

Background

This survey was organized in 1997, as part of the XII International ICCROM/UNESCO Course on the Technology of Stone Conservation (SC97). It was undertaken by a multidisciplinary group of conservation architects, conservator-restorers and engineers supervised by experts in the field.

(N.B. Only the part of the survey referring to the porosimetry analysis is reported here).

Aim

The aim was to develop a proposal for conservation treatment by assessing the conditions of the north façade, integrating *in situ* observations, historical data and scientific investigations and carrying out treatment trials.

A program of laboratory analysis was carried out in order to:

- assess the façade's existing condition and possible decay mechanisms,
- identify altered materials,
- better understand the original material.

Description

S. Maria Formosa (Fig. 1) is considered one of the earliest Venetian churches (7th century). However, over the centuries it has undergone so many modifications that it now appears in typical Renaissance style. It has a Latin cross plan with three aisles, deep side chapels, a presbytery, a semicircular apse and a central dome over the transept. The chapel on the right-hand side borders the canal whereas the north façade, to which the case study refers, overlooks Campo S. Maria Formosa.

The façade is divided into five parts by pilasters with four semicircular windows in the first order. Three busts are located in the upper part of the façade. The central bust stands at the base of an oculum at the centre of an open arch with volutes. Five statues are symmetrically positioned above the upper cornice.



Fig. 1 - S. Maria Formosa

① Porosity = 0,2 - 0,5 %

② Porosity = 0,5 - 1 %

③ Porosity = 25 - 28 %

Survey: materials, previous treatments and state of conservation of north façade

The façade of the church is made up of Istrian limestone^① blocks; the three busts of Carrara marble^②, while the five statues may have been originally carved out of Vicenza limestone^③.

Attention was mainly concentrated on the structural condition of the church although there was also significant decay and physical change of the stone surface: areas of black crusts, rainwashing and pigeon excrement as well as *biological growth*. Flaking and scaling was evident on all the stone surfaces. *Disaggregation* was apparent on the three marble busts and the upper right sculptures and *blistering* on the top central sculpture. *Erosion* was present mainly below the cornices, on the corners near the pilasters where there is rainwater washing.

There were quite a few small and medium sized broken or loose pieces on corners and projecting architectural elements. Cracks, fissures and fractures were related either to differential decay and bedding of the stone or to structural problems. Furthermore, many of the joints between the stone blocks were devoid of mortar, especially on the right side of the façade and in places where considerable water erosion had taken place.

Experimental

The scientific investigations focused on the essential problems of the façade due to limited time and funds. Apart from soluble salts analysis, biological identification, and petrography analysis, research concentrated on the porosimetry of Vicenza limestone in order to assess the degree of deterioration of the uppermost sculptures.

Sampling

Fig. 2 shows sampling locations with indications of relevant analyses.

POROSIMETRIC ANALYSIS

Using a mercury porosimeter the total porosity and open pore size distribution was calculated for sample n. 110 from a sculpture at the uppermost part of the façade and on a quarry sample of unweathered Vicenza limestone.

The total porosity of the quarried stone was 18.24% compared to the 27.29% of the statue (Fig. 3). This sharp increase of about 50% in total porosity is due to the extreme weathered condition of the stone.

The open pore size distribution of the two samples was determined because it plays an important role in the chemical and physical behaviour of porous materials.

A comparison of the pore size distributions of the two samples shows a marked difference (Fig. 4 and 5). The quarried limestone shows a bell-shaped curve with a maximum amount of pores in the range

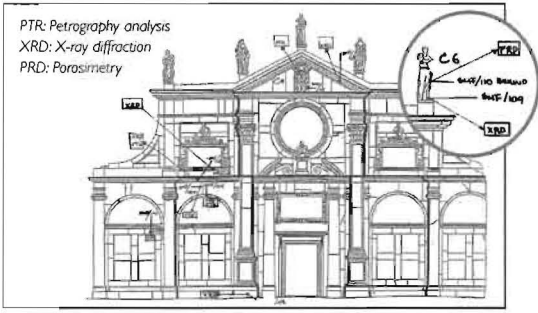


Fig. 2 - Sampling plan

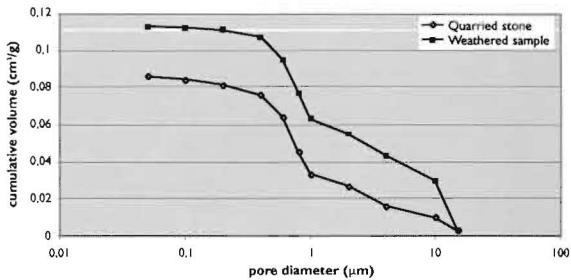


Fig. 3 - Mercury porosimetry analysis

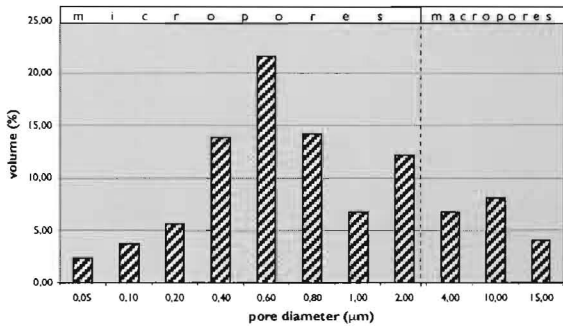


Fig. 4 - Quarried stone sample (Pore size distribution)

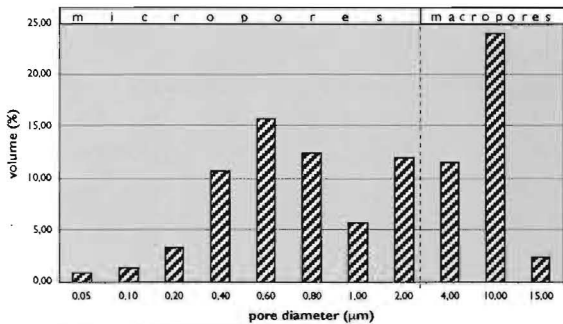


Fig. 5 - Weathered sample from the sculpture (Pore size distribution)

of **0.4 - 0.8 μm** . The *weathered* sample from the sculpture shows a bi-modal curve: the first part of the pore size distribution curve is similar to that of the quarried sample (**0.4 - 0.6 μm**), while the second part of the curve indicates higher open porosity in the range of **4 - 10 μm** .

Conclusions

It must be noted that all the statues require some form of consolidation to re-establish the cohesion of the material. The increase in the amount of larger open pores reflects the severe deterioration of the sculpture which may be due to several factors. For example, soluble salt crystallization cycles would greatly affect the pores due to the expansion and pressure caused by salt crystals. There are cement fills in the sculpture from where these salts probably originate. Other sources of salts may be from air pollution.

The severe weathering of these sculptures is visible to the eye, but the analysis made it possible to qualify and quantify the deterioration.

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Designation: D 4404-84 (reappr. 1992). Standard test method for determination of pore volume distribution of soil and rock by mercury intrusion porosimetry

Designation: D 4959-89. Standard test method for determination of water (moisture) content of soil by direct heating method

Designation: D 4525-90. Permeability of rocks by flowing air
Designation: D 653-90. Terminology relating to soil, rock and contained fluids

Designation: C 566-89. Test method for total moisture content of aggregate by drying

Designation: E 12-70 (reappr. 1991). Density and specific gravity of solids, liquids and gases

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Test n. I.1: Porosity accessible to water

Test n. I.2: Bulk and real densities

Test n. I.3: Air-permeability

Test n. I.4: Pore-size distribution (suction)

Test n. I.5: Pore-size distribution (mercury porosimeter)

Test n. II.1: Saturation coefficient

Test n. II.2: Coefficient of water vapour conductivity

Test n. II.3: Water absorption under low pressure (box method)

Test n. II.4: Water absorption under low pressure (pipe method)

Test n. II.5: Evaporation curve

Test n. II.6: Water absorption coefficient (capillarity)

Test n. II.8a: Water drop absorption

NORMAL documents (Commissione Normativa Manufatti Lapedei)

NORMAL 4/ 80: Distribuzione del volume dei pori in funzione del loro diametro

NORMAL 7 /81: Assorbimento d'acqua per immersione totale - Capacità di Imbibizione

NORMAL 11/ 85: Assorbimento d'acqua per capillarità - Coefficiente di assorbimento capillare

NORMAL 21/ 85: Permeabilità al vapore d'acqua

NORMAL 29/ 88: Misura dell'indice di asciugamento (Drying index)

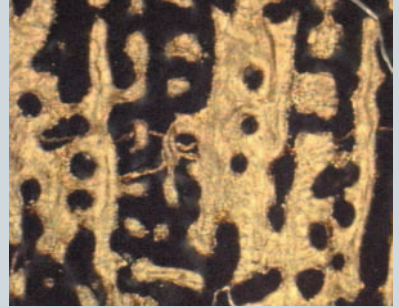
NORMAL 33/ 89: Misura dell'angolo di contatto

NORMAL 44/ 93: Assorbimento d'acqua a bassa pressione

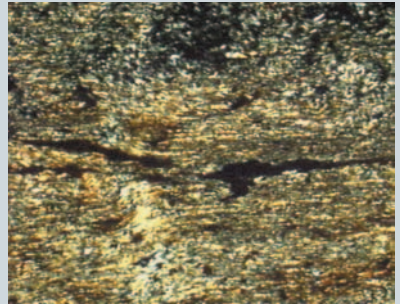
POROSITY

MICROPHOTOGRAPHS OF THIN SECTIONS Examples of macroporosity

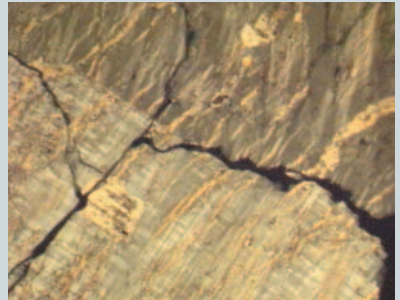
1
Coral stone



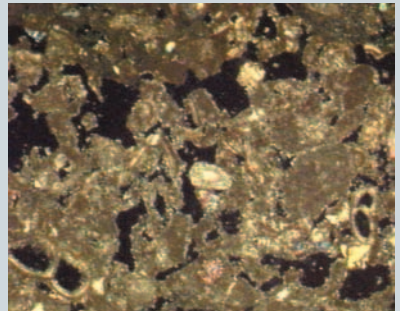
2
Slate



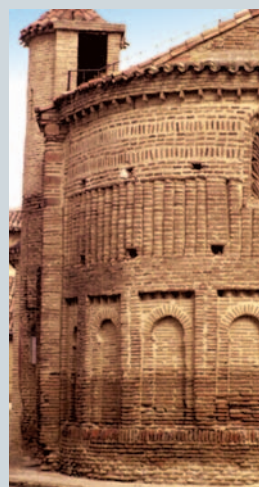
3
Granite



4
Organogenic limestone



ISBN 92-9077-157-7



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