



INTERNATIONAL CENTRE FOR THE STUDY OF THE PRESERVATION AND THE RESTORATION OF CULTURAL PROPERTY  
CENTRE INTERNATIONAL D'ETUDES POUR LA CONSERVATION ET LA RESTAURATION DES BIENS CULTURELS

**ICCROM**

# ARC

## A LABORATORY MANUAL FOR ARCHITECTURAL CONSERVATORS

Jeanne Marie Teutonico

**A LABORATORY MANUAL  
FOR  
ARCHITECTURAL CONSERVATORS**

Jeanne Marie Teutonico



**I C C R O M**

Rome 1988

## CONTENTS

<u>Foreword</u>	v
<u>Preface</u>	vii
<u>General Principles: Laboratory Science</u>	
1. Sampling	3
2. Measurement and Error: Precision, Accuracy, Statistics	7
3. Measurement: Mass (Use of the Balance)	11
4. Measurement: Length (Use of the Vernier Caliper and Micrometer)	16
5. Measurement: Volume	21
6. Measurement: Solutions	26
7. Measurement: pH	30
<u>Porous Building Materials</u>	
8. Water Absorption by Total Immersion	35
9. Water Drop Absorption	41
10. Penetration of Water: Capillary Action	43
11. Porosity of Granular Beds	45
12. Porosity in Solids: Indirect Measurement by Water Absorption	50
13. Porosity in Solids: Hydrostatic Weighing	52
14. Movement of Salts	56
15. Salt Crystallization	57
16. Qualitative Analysis of Water-soluble Salts and Carbonates	58
17. Semiquantitative Analysis of Water-soluble Salts	68
<u>Earthen Building Materials</u>	
18A. Particle Size Analysis: Part I Sieving Procedure	73

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Earthen Building Materials (continued)

18B. Particle Size Analysis: Part II Sedimentation Procedure: Hydrometer Method	83
--	----

19. Plastic Limit of Soils	96
----------------------------	----

20. Liquid Limit of Soils	102
---------------------------	-----

Stone, Brick and Mortars

21. Mortar Analysis: Simple Method	113
------------------------------------	-----

22. Analysis of Calcium Carbonate Content in Mortars: Calcimeter Method	117
--	-----

23. Mixing Mortars for Conservation	119
-------------------------------------	-----

24. Cleaning Building Stone	123
-----------------------------	-----

25. Repointing of Stone and Brickwork	127
---------------------------------------	-----

26. Investigation of the Carbonation Process in Lime Mortars by Means of Phenolphthalein	129
---	-----

Architectural Surfaces:

Renders, Plaster (Intonaco). Paint

27. Sampling of Architectural Surface Materials	137
---	-----

28. Imbedding a Sample for Cross Section	139
--	-----

29. Cutting and Polishing a Cross Section of Surface Material; Observation of the Sample	143
---	-----

30. Simplified Method for Imbedding and Polishing a Cross Section of Plaster or Paint	148
--	-----

Wood

31. Wood Structure	155
--------------------	-----

32. Swelling and Shrinkage of Wood	158
------------------------------------	-----

33. Cross Sections and Identification of Wood	161
---	-----

34. Test Loading of Wood	163
--------------------------	-----

35. Preparation and Application of Traditional Oil House Paint	167
---	-----

FOREWORD

After his appointment as director of ICCROM in 1977, Sir Bernard FEILDEN proposed to emphasize the interdisciplinary character of conservation by introducing scientists to field work and architects to laboratories. Consequently it was decided to introduce a series of laboratory exercises designed for the needs of architectural conservators and conservation architects in the program of the International Architectural Conservation Course at ICCROM.

The present publication can be seen as an outcome of this initiative. It should be conceived as an introduction to the work in an architectural conservation laboratory, aimed at helping the reader to understand the character and behavior of building materials, their identification, and the diagnosis of their state of conservation. It is believed that this manual would be useful not only to courses in conservation, but also to schools of architecture and engineering, as well as institutions interested in establishing a low-cost architectural conservation laboratory.

The material included has been drawn from different sources, and thanks are due to the many ICCROM lecturers and experts who have contributed to it. The training programs of the Scientific Principles of Conservation Course, run at ICCROM under the direction of Giorgio TORRACA, and the Istituto Centrale del Restauro in Rome provided a basis. The first selection and preparation of exercises was undertaken by Simonetta PERONI, then assistant to the Architectural Conservation Course. Since 1984, this material has been completely revised, expanded, further elaborated and edited by Jeanne Marie TEUTONICO, who has dedicated much time to the development of laboratory training in the Architectural Conservation Course at ICCROM. She has been assisted by Cynthia ROCKWELL and Monica GARCIA in the editing and publication of the manual, and by Mehr Azar SOHEIL in the line drawings and graphic presentation.

Special thanks are due to the Finnish Government whose financial contribution made the publication of this manual-a reality.

Dr Jukka Jokilehto  
Coordinator of  
Architectural Programs

## PREFACE

This manual is intended to provide a basic introduction to the analyses of building materials that can be carried out in an architectural conservation laboratory.

Certainly, it is neither possible nor desirable to turn every architectural conservator into a laboratory technician. The aim here is to make architectural conservators more aware of the potential offered by the conservation laboratory for the understanding of materials and structures. Such potential ranges from simple tests, which the conservator can conceivably carry out alone, to more complicated procedures for which the aid of a specialist might be required. In either case, a greater familiarity with laboratory tests and techniques will better enable the architectural conservator to obtain the information needed for the critical evaluation of historic structures.

This manual has been prepared with such goals in mind. The present edition is the result of four years of research, re-evaluation, and experience in a teaching situation. It begins with a section on general principles which provides an introduction to laboratory concepts, methods, and equipment. This is followed by a compendium of laboratory analyses and exercises, organized by building material. All of these can provide useful information with only simple equipment.

The laboratory procedures have been collected from various standardized sources (ASTM, NORMAL, RILEM, etc.) and then adapted by the author to suit the specific needs of the architectural conservator. This process has often necessitated a complete re-thinking and re-working of the parent methodologies. Too frequently, procedures are simply "transferred" from related disciplines (i.e. conservation of objects or paintings) and have little relevance to the scale, materials, deterioration processes, and environmental conditions faced by the architectural conservator. Thus, though original sources of experiments have been cited in the bibliographic references, the resulting procedures sometimes bear little resemblance to their origins.

In certain cases for which no standardized tests exist, the analyses have been based on procedures developed by various conservation experts or teaching laboratories (cited in the bibliographic references). These have been similarly modified by the author to suit the aims and format of the manual.

It is important; to emphasize that the analyses contained in this compendium have been adapted to the exigencies of a basic conservation laboratory. All are designed to give scientifically reliable results with simple and inexpensive equipment, thus respecting the economic realities and working conditions of many architectural conservation projects.

GENERAL PRINCIPLES: LABORATORY SCIENCE

## Ex. 1 SAMPLING

### INTRODUCTION/AIM

This exercise introduces the sampling process and the variables that affect it. It is important to remember that the experimental process begins in the field when the samples are taken. As in the laboratory, experimental design (i.e. the sampling procedure) is critical. The conservator must consider factors such as: *WHEN* should samples be taken? Under what conditions? Using which instruments? *HOW MANY* samples should be taken? Of what size? *WHERE* should the samples come from? *WHAT* information is desired (i.e. why are the samples being taken)? It is essential to keep in mind that both techniques and results must be reproducible.

### DEFINITIONS

- (1) Types of Comparison: The experimenter must gather enough data to make relevant comparisons. These can be external in which evidence about the subject building is compared with other buildings OR internal in which individual pieces of evidence are compared with the building as a whole.

In general, internal comparisons are more reliable because very few procedures are standardized.

- (2) Types of Sampling:

Random sampling: A process in which samples are chosen at random; spot-checking (e.g. quality control in factories).

Non-random sampling: A process in which samples are drawn from a particular area or group (e.g. if deterioration on a building occurs in a localized area, all samples may be drawn from there if the purpose of the investigation is to discover the cause of deterioration).

Population: The entire sample group.

### MATERIALS

50 coins or chips; red nail polish; plastic bag; graph paper; notebook; calculator.

Ex. 1 (continued)

# PROCEDURE

1. Work in teams of three persons: one person (R) to record the data and the others (E1 and E2) to collect the samples.
2. Mark 5 of the coins with red nail polish.
3. Place 5 marked and 45 unmarked coins in the plastic bag. Shake the bag to mix the coins.
4. Let E1 reach into the bag and pull out a random quantity of coins from the bag. Let R record the number of marked and unmarked coins in the sample on the attached chart.
5. Return the sample pulled by E1 to the plastic bag. Mix the coins and let E2 pull a random quantity of coins from the bag. Let R record the number of marked and unmarked coins in the sample on the attached chart.
6. Repeat steps (4) and (5), alternating E1 and E2, until the ratio of marked/unmarked coins (keep a running total as indicated on the chart) equals or closely approximates the actual ratio of the population.
7. Express the results mathematically by completing the chart.
8. Express the results graphically showing the ratio as a function of the number of samples.
9. Repeat the entire procedure using a sample population of 20 marked coins out of 50.

# DISCUSSION

- (A) How did the following factors affect the sampling results:
- 1) Actual composition of the population (actual ratio of marked to unmarked coins).
  - 2) Number of coins in each sample (relationship of sample size to actual composition of the population).
  - 3) The sample collector (compare E1 and E2).
  - 4) The method of sample collection.
- (B) What were the possible sources of error?
- (C) What general statements can you make about the reliability of sampling and the factors that affect it?

Ex. 1 (continued)

Population: Marked coins \_\_\_\_\_ Unmarked coins \_\_\_\_\_

Sample	Marked coins	Total coins	Ratio fraction	Ratio dec.	$\Sigma$ Ratio fraction	$\Sigma$ Ratio dec.
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						

Ex. 1 (continued)

Population: Marked coins \_\_\_\_\_ Unmarked coins \_\_\_\_\_

Sample	Marked coins	Total coins	Ratio fraction	Ratio dec.	$\Sigma$ Ratio fraction	$\Sigma$ Ratio dec.
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						
E1						
E2						

## Ex. 2 MEASUREMENT AND ERROR: PRECISION, ACCURACY, STATISTICS

### AIM

To become familiar with the standard statistical evaluation of data. This exercise is designed to introduce the concepts of precision and accuracy and to provide a basic framework for the mathematical manipulation of laboratory data.

### DEFINITIONS

- (1) Data Treatment: Much of a conservator's data is numerical. These numbers must be (a) sorted and (b) questioned through statistics to arrive at some sort of assumption. In this regard, it is essential to understand the difference between precision and accuracy.

Precision: Degree of agreement of repeated measurements of the same quantity. It is a statistical value, the calculation of which is described below. Precision and reproducibility are synonymous.

Accuracy: The agreement between the result of a measurement and the true or real value of the quantity measured. (A measurement that is accurate is not only reproducible but also the "right" answer.)

"Accuracy" has to do with the closeness (of data) to the truth, "precision" only with the closeness of readings to one another.

### (2) Calculations:

Mean: The average. Sum up all the data and divide by the number of values.

Mode: The most common piece of data.

Median: The value right in the middle.

The dispersion of data about the mean gives an idea of the precision of a series of measurements. There are several ways to measure dispersion, including:

Range or Spread: The difference between the least and the greatest values in a set of data.

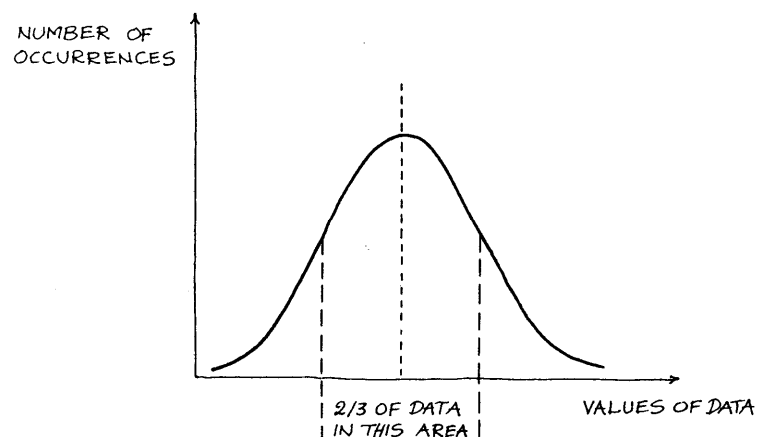
Absolute Deviation: For each piece of data, the difference between its value and the mean. The difference may be positive (greater than the mean) or negative, (smaller than the mean). This does not affect its "absolute value".

Ex. 2 (continued)

Mean Absolute Deviation: An average of the absolute deviations of the sample data.

Mean Relative Deviation:  $\frac{\text{Mean Absolute Deviation}}{\text{Mean}}$

The Standard Deviation: The most widely employed system. It establishes an interval in which at least 2/3 of the values will fall.



Calculation of Standard Deviation:

First, calculate the Variance. This is the sum of all the squares of the absolute deviations divided by the number of values.

The Standard Deviation (indicated by the Greek letter sigma  $\sigma$ ) is the square root of the variance.

$$\sigma = \sqrt{\frac{\sum (x-u)^2}{N}}$$

or, for small amounts of data, the Standard Deviation is:

$$\sigma = \sqrt{\frac{\sum (x-u)^2}{N-1}}$$

u = mean  
x = individual item  
N = number of elements

u = mean  
x = individual item  
N = number of elements

Ex. 2 (continued)

#### EXAMPLE OF PRECISION CALCULATIONS

Results	Absolute Deviations	Square Deviations
17.1	(+)0.1	.01
17.4	(+)0.4	.16
16.6	(-)0.4	.16
16.0	(-)1.0	1.00
17.7	(+)0.7	.49
84.8	2.6	1.82

Mean =  $84.8 \div 5 = 16.96$   
 $\approx 17.00$

Median = 17.1

Range = from 16.0 to 17.7 with a mean of 17.0; it is written 17.0  
+0.7 -1.0

Mean Absolute Deviation:  $2.6 \div 5 = 0.5$

Variance:  $1.82 \div 5 = .36$

Standard Deviation:  $\sqrt{.36} = 0.6$   
 $= 17.0 \pm 0.6$   
3 out of 5 results fall in this range.

#### PROCEDURE

Do the following exercise: The weight of a silver ring is measured 10 times on a rather large balance on which the weight may be measured up to 0.01 of a gram. The results are as follows:

Results	Absolute Deviations	Square Deviations
4.01		
4.02		
4.02		
4.01		
4.01		
4.01		
4.02		
4.03		
3.99		
4.01		

A. Fill in the chart.9

Ex. 2 (continued)

B. Calculate the:

Mean:

Median:

Range:

Mean Absolute Deviation:

Variance:

Standard Deviation:

C. Graph the results.

Ex. 3 MEASUREMENT: MASS (Use of the Balance)

AIM

Balances are mechanical devices used to determine the mass of objects. Many kinds of balances are available, ranging from rough measuring devices which are sensitive to 0.1 g to the analytical balances sensitive to fractions of a microgram.

The choice of a balance obviously depends upon its designated use. The following exercise introduces two balances typically used in the architectural conservation laboratory: a Sartorius single-pan, top-loading balance (precision 0.1 g, maximum load 1000 g), and a Mettler balance of a similar design (precision 0.01 g, maximum load 1200 g).

#### DEFINITIONS

**Mass:** An invariant measure of the quantity of matter in an object. The SI (International System of Units) unit of mass is the kilogram, but gram quantities are more usual in the laboratory. Technicians properly use the term mass in discussing measurements made with a balance.

**Weight:** The forces of attraction exhibited between an object and the earth. Weight equals mass times the gravitational attraction. Mass is proportional to weight, so we ordinarily interchange the terms, but the unit of weight is the newton.

**Capacity:** The largest load on one pan for which the balance can be brought to equilibrium.

**Readability:** The smallest fraction of a division at which the index scale can be read with ease.

**Sensitivity:** The change in load required to produce a perceptible change in indication. It is, therefore, a ratio and not to be used to discuss the quality of a measurement.

**Significant Figure:** A digit that shows a quantity in the position that it occupies in the whole numerical term. A zero is not significant when it is used to locate the decimal point; it is significant, however, when it fixes quantity, for example, when it, indicates that the value is nearer to 0 than to 1.

E.g. In the number  $1.500 \times 10^1$ , the<sup>y</sup> zeroes are, significant because they show that the quantity is nearer to  $1.500 \times 10^1$  than to  $1.501 \times 10^1$  or  $1.499 \times 10^1$ .

Ex. 3 (continued)

A number should contain no more than one doubtful digit. For a number such as 2.987 with four significant digits, only the 7 should be doubtful.

#### PROCEDURE

1. Examine the attached diagram of the Sartorius balance which outlines its parts and their use.
2. Check the balance zero.
3. Weigh the sample objects (A) given to you. Record the weights in Data Sheet 1.
4. Weighing materials in a beaker:

##### Procedure (a):

Weigh the beaker empty ( $W_1$ ). Place sample 2A in the beaker and weigh ( $W_2$ ). Subtract  $W_1$  from  $W_2$  to obtain the weight of the sample ( $W_3$ ). Record your results in Data Sheet 2.

##### Procedure (b):

Use the Tare knob on the balance. Place the beaker on the pan and turn the Tare knob until it reads zero. Place sample 2 in the beaker and weigh. Record the weight in Data Sheet 2.

Are the weights obtained using procedure (a) and procedure (b) the same? What are the possible sources of error?

5. Empty the pan of the balance. Return the Tare knob to zero.
6. Lock the pan of the balance.
7. Repeat Steps 1 through 6 using the Mettler balance and Samples 1B through 6B. -

#### BIBLIOGRAPHY

ICCROM, Scientific Principles of Conservation Course, Course exercises, 1976-77.

Shugar, G. - Shugar, R. - Bauman, L. - Bauman, R.S. Chemical Technicians' Ready Reference Handbook. New York: McGraw Hill, 1981.

Ex. 3 (continued)

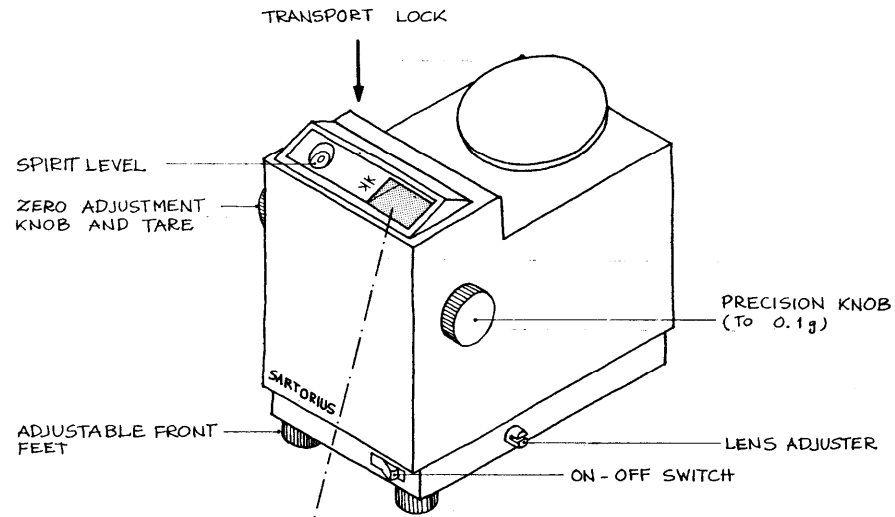
#### DATA SHEET 1

SAMPLE	WEIGHT	SAMPLE	WEIGHT
1A		1B	
2A		2B	
3A		3B	
4A		4B	
5A		5B	
6A		6B	

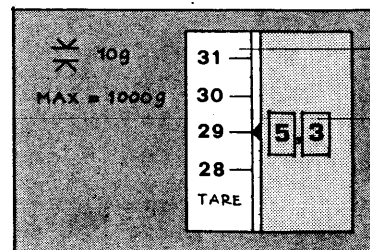
#### DATA SHEET 2

SAMPLE	2A	2B
Beaker + Sample ( $W_2$ )		
Beaker alone ( $W_1$ )		
Sample weight ( $W_3$ )		
Sample weight with TARE		

Ex. 3 (continued)



**SARTORIUS**



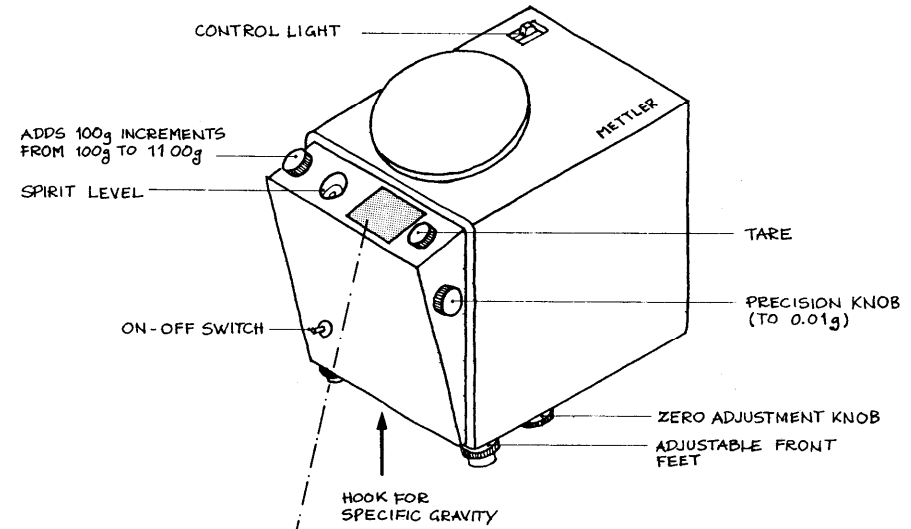
DETAIL OF DIAL

OPTICAL SCALE  
READS 00 TO 99 (990g)

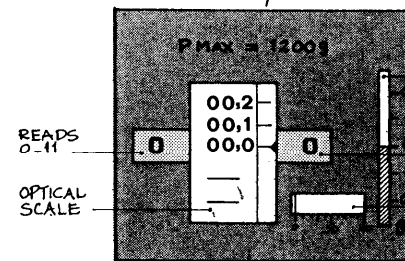
DIGITAL SCALE  
ADJUST BY TURNING PRECISION  
KNOB UNTIL BLACK ARROW  
CORRESPONDS TO A LINE ON THE  
OPTICAL SCALE

READ: 295.3

Ex. 3 (continued)



**METTLER**



DETAIL OF DIAL

PLUS/MINUS SCALE  
USED TO VERIFY WEIGHTS

DIGITAL SCALE  
ADJUST BY TURNING PRECISION KNOB  
UNTIL BLACK ARROW CORRESPONDS TO  
A LINE ON THE OPTICAL SCALE

ANALOG SCALE REPEATER  
0-100g FACILITATES DOSAGE

Ex. 4 MEASUREMENT: LENGTH (Use of the Vernier Caliper and Micrometer)

AIM

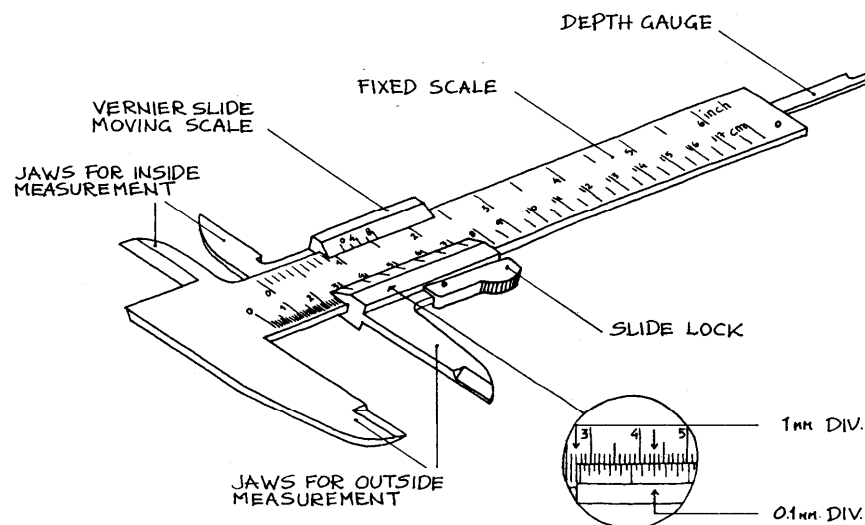
There are many types of laboratory tools for the precise measurement of length. Among these are the vernier caliper and the micrometer or screw gauge. The purpose of this exercise is to make you familiar with the care and use of these instruments.

DEFINITIONS

**Calipers:** Calipers consist of a pair of hinged steel jaws which are used for measuring the dimensions, both internal and external, of small objects where a scale rule cannot be applied directly.

**Vernier scale:** Invented by Pierre Vernier in the seventeenth century, the vernier scale is fitted to many types of measuring and surveying instruments. It enables measurements to be made by direct reading to 0.1 mm without having to estimate fractions of a division.

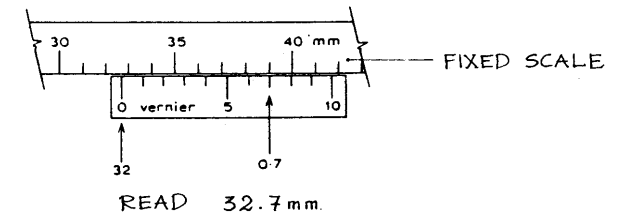
**Vernier calipers:** Vernier calipers consist of a fixed steel scale marked in millimeters (from 16 to 20 cm in length) with a fixed jaw at one end, and a sliding jaw carrying a scale (the vernier) which is 9 mm long and divided into 10 (or 20) equal parts.



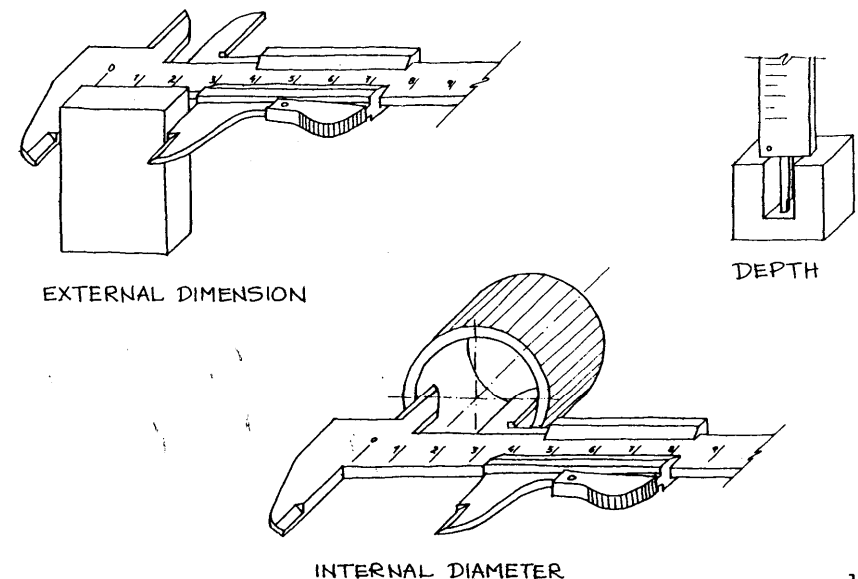
Ex. 4 (continued)

To measure with the vernier caliper:

1. Close (or open) the jaws so that they very lightly clamp the object at the desired point of measurement.
2. Read the fixed scale. The nearest scale division to the left of the zero on the vernier indicates the number of whole millimeters being measured.
3. Read the vernier. The line of the vernier that directly coincides with a line on the fixed scale indicates the number of tenths (or twentieths) of a millimeter to be added. See the example below.

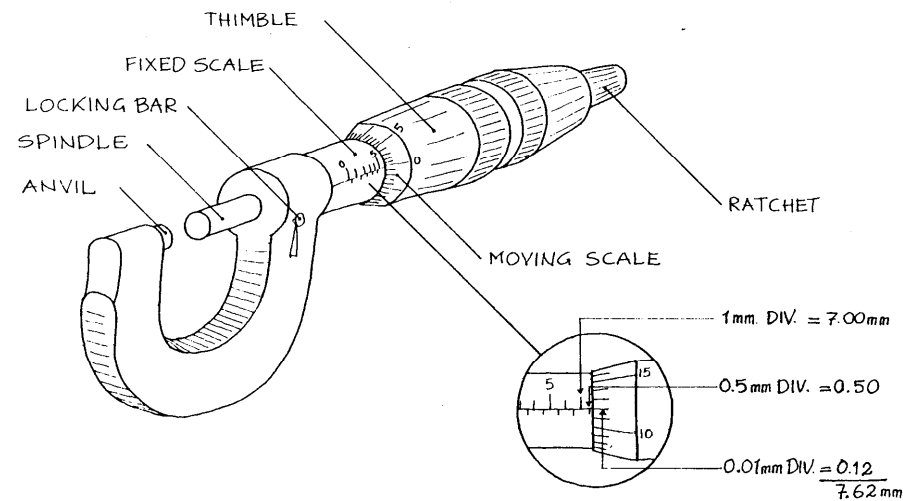


Vernier calipers have jaws for both internal and external measurements. They can also be used for depth measurement as indicated below.



Ex. 4 (continued)

**Micrometer or Screw Gauge:** An instrument used for the measurement of lengths between 0.01 mm and a few centimeters. It is thus more sensitive than the vernier caliper but has a narrower range. Though there are diverse types, a typical micrometer is illustrated below. The range of this instrument (0-25 mm, 25-50 mm, 50-75 mm) is usually indicated on the face of the U-shaped piece.

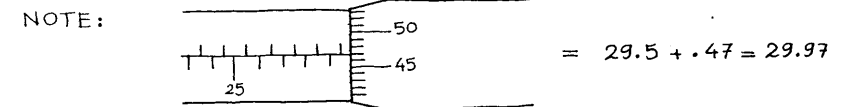
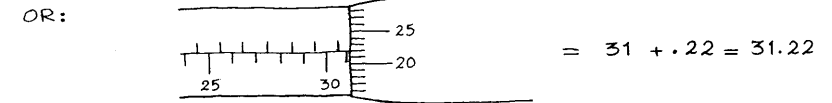
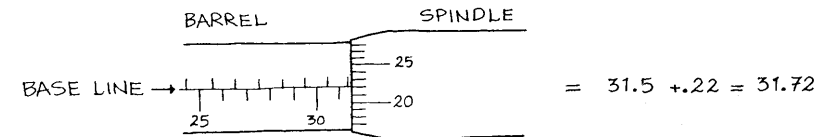


To measure with the micrometer:

1. Make sure the faces of the spindle and the anvil are clean and free of dust.
2. Place the object to be measured in the mouth of the micrometer.
3. Gently rotate the thimble until the object is lightly clamped between the anvil and the spindle (the thimble will stop moving). Make final adjustments with the ratchet knob. When the ratchet knob begins to slip round, you are ready to read the scales.

Ex. 4 (continued)

4. The length of the object is read by adding (a) the number of millimeters and half millimeters indicated by the length of the fixed scale uncovered by the thimble and (b) the number on the moving scale which most nearly coincides with the base line on the fixed scale. See examples below.



Ex. 4 (continued)

PROCEDURE

1. Measure the samples given to you with the vernier caliper.

Record the dimensions below:

**Sample A:** length \_\_\_\_\_  
width \_\_\_\_\_  
height \_\_\_\_\_

**Sample B:** length \_\_\_\_\_  
external diameter \_\_\_\_\_

**Sample C:** length \_\_\_\_\_  
external diameter \_\_\_\_\_  
internal diameter \_\_\_\_\_

2. Make sure the instrument is free from dust and return it to its case.
3. Using the micrometer, measure the second set of samples given to you. Record the results below

Sample	Thickness
D	
E	
F	

4. Make sure the micrometer is clean and return it to its case.
5. Compare your results with those of others in the lab. What are the possible sources of error?

BIBLIOGRAPHY

Head, K.H. Manual of soil laboratory testing. London: Pen-tech Press, 1980.

ICCROM, Scientific Principles of Conservation Course, Course notes.

Lucarelli, S. Notes on the use of measuring instruments. Rome: ICCROM, 1979.

Shugar, G. - Shugar, R. - Bauman, L. - Bauman, R.S. Chemical technicians' ready reference handbook. New York: McGraw Hill, 1981.

Ex. 5 MEASUREMENT: VOLUME

AIM

There are many different types of laboratory equipment for measuring volume. This exercise is intended to make you familiar with the volumetric equipment most frequently used in the architectural conservation laboratory.

DEFINITIONS

Units of Volume      Liter (L): the volume occupied by one kilogram of water at 4°C and standard atmospheric pressure; this volume is equivalent to one cubic decimeter.

Milliliter (mL): one-thousandth of a liter.

Cubic centimeter (cm<sup>3</sup>): can be used interchangeably with milliliter without effect.

Meniscus: The curvature exhibited at the surface of a liquid that is confined in a narrow tube such as a cylinder, buret, or pipet.

It is common practice to use the bottom of the meniscus in calibrating and using volumetric glassware (see illustration on page 22).

VOLUMETRIC EQUIPMENT

General Care: Only clean glass surfaces will support a uniform film of liquid; all volumetric glassware must therefore be clean and free of grease. Water should flow continuously on the glass surface without leaving any drops.



Ex. 5 (continued)

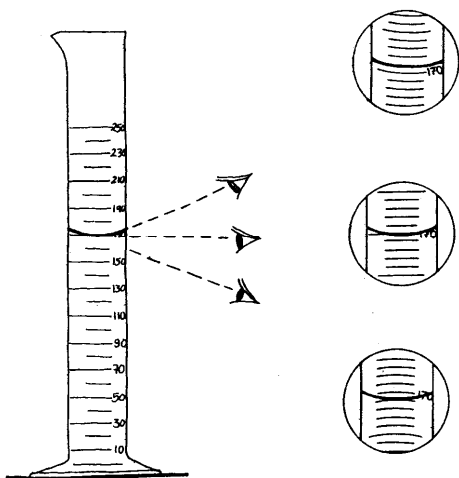
**Cleaning:** Volumetric glassware can normally be cleaned by washing and brushing with a detergent solution. The cleaned glass should then be rinsed first with tap water and, finally, 4 or 5 times with distilled water.

(Stubborn dirt may be removed with chromic acid. The preparation and use of this reagent, however, is quite dangerous and should only be done by trained technicians.)

Volumetric glassware should be dried at room temperature (i.e. on a rack), never in a hot oven which may alter calibration.

Types of Glassware:

- (a) Graduated Cylinders: Perhaps the most common type of volumetric equipment. These range in size from 25 mL to 1000 mL. When using any graduated cylinder to measure a volume of liquid, remember to read the bottom of the meniscus. As shown below, it is important to have the eye at the same level as the meniscus to avoid parallax errors.



- (b) Volumetric Flask: A type of volumetric equipment calibrated to contain a specified volume when filled to the line etched on the neck.

Ex. 5 (continued)

- (c) Pipet: This piece of volumetric equipment is designed for the transfer of known volumes from one container to another. Pipets that deliver a fixed volume are called *volumetric or transfer pipets* (see below). Other pipets, calibrated in convenient units so that any volume up to a maximum capacity can be delivered, are known as *measuring pipets*.

Proper use of a transfer pipet (see diagram below):

- 1) *Make sure the tip of the pipet is below the surface of the liquid to be transferred.*

*Draw the liquid up into the pipet using an appropriate pipet filler (never the MOUTH!).*



- 2) *When the liquid is past the calibration mark, remove the suction device and place the index finger over the exposed end of the pipet.*

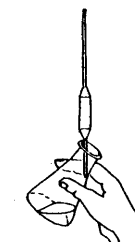


- 3) *Release pressure on the index finger until the meniscus reaches the calibration mark. Stop the flow by reapplying pressure. Drain the drop on the tip by touching it to the wall of the liquid-holding container.*



- 4) *Transfer the pipet to the receiving container. Touch a wall of the receiving container with the tip of the pipet and allow the liquid to drain slowly.*

*Touch off the last drop of liquid against the wall of the container. Do not blow out the pipet (it is calibrated for a small amount of liquid to remain).*

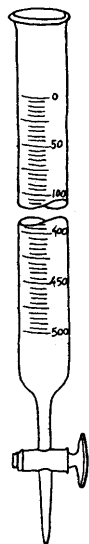


Ex. 5 (continued)

- (d) Buret: Like the measuring pipet, this piece of volumetric equipment is designed to deliver any volume up to its maximum capacity. It is fitted with a stopcock for control of liquid flow. The buret is most commonly used for titration processes.

Titration: A process by which a substance to be measured is combined with a reagent and quantitatively measured. Ordinarily, this is accomplished by the controlled addition of a reagent of a known concentration to a solution of the substance until the reaction between the two is judged to be complete. The volume of the reagent is then measured.

*Single-dispensing  
buret with  
graduated etched  
scale, standard  
taper, and stopcock.  
Such burets are  
available in general  
or standardized  
models with  
volumes from 50 to  
500 cm<sup>3</sup>.*



Ex. 5 (continued)

PROCEDURE

1. Become familiar with the various pieces of volumetric equipment given to you.
2. Practice reading the meniscus of the liquid volume in the graduated cylinder.
3. Practice transferring a volume of liquid from one container to another using the pipet.
4. Prepare the solutions outlined in Exercise 6.

BIBLIOGRAPHY

ICCROM, Scientific Principles of Conservation Course, Course notes.

Shugar, G. - Shugar, R. - Bauman, L. - Bauman, R.S. Chemical technicians' ready reference handbook. New York: McGraw Hill, 1981.

## Ex. 6 MEASUREMENT: SOLUTIONS

### AIM

Most of the time, substances are not used pure. Instead, they are diluted in other substances to make solutions. This exercise will introduce you to various types of solutions and how to make them.

### DEFINITIONS

- Solution:** A homogeneous system of two or more substances which may be present in varying amounts.
- Solute:** That substance which is dissolved or has gone into solution (e.g. sugar into water). It constitutes less than 50 percent of the solution.
- Solvent:** That substance which does the dissolving (e.g. water dissolving sugar). It constitutes more than 50 percent of the solution.
- Composition:** Mass of solute per unit mass of solvent.
- Concentration:** Amount of solute per unit volume of solvent.

### CALCULATIONS

There are many ways to express and prepare the concentration of laboratory solutions. These include:

Mass Percent: Grams of solute per 100 grams of solution (w/w\*).

E.g. 25 g of NaCl in 100 g of H<sub>2</sub>O is a 20% by mass solution.

$$\text{Why? } \frac{\text{Mass of solute}}{\text{Mass of solution}} = \frac{25 \text{ g}}{25 \text{ g} + 100 \text{ g}} = \frac{25}{125} = 20\%$$

\* The density of water at 4 °C is 1 g/cm<sup>3</sup> = 1 g/mL. The terms milliliter and cubic centimeter (cc) are therefore usually interchangeable. One mL of water is also assumed to weigh approximately 1 gram (though it varies somewhat according to the temperature). Thus, in the case of mass percent solutions where the solute is water, 100 g of water can be assumed equal to 100 mL of water.

## Ex. 6 (continued)

Volume Percent: Milliliters of solute per 100 milliliters of solution (used in the case of mixing liquids; a v/v percent).

E.g. 25 mL of acetone (1 part) diluted in 75 mL of H<sub>2</sub>O (3 parts) = 25% by volume solution.

$$\text{Why? } \frac{\text{Volume of solute}}{\text{Volume of solution}} = \frac{25 \text{ mL}}{25 + 75 \text{ mL}} = \frac{25}{100} = 25\%$$

### Molar and Molal Solutions:

To understand these types of solutions, one must first grasp the concept of the mole.

One *mole* (also called the gram-molecular mass) is the number of grams equal to the molecular weight of a substance. The gram-molecular mass can be calculated by adding the individual atomic masses (see the attached Periodic Table of Elements based on C = 12 g/mole\*) of the component parts of a molecule.

E.g. to calculate the mass in grams of one mole of NaCl (table salt):

$$\begin{aligned} \text{Na} &= 22.99 \text{ g/mole} \\ \text{Cl} &= 35.45 \text{ g/mole} \\ \text{NaCl} &= 58.44 \text{ g/mole} \end{aligned}$$

\* Since the Periodic Table of Elements has been standardized around Carbon<sub>12</sub> at 12 g/mole, a mole of any element contains the same number of atoms. This number is called AVOGADRO'S NUMBER and equals  $6.02 \times 10^{23}$ .

Molality (m): The number of moles of solute per 1000 grams of solvent. This is a composition term.

E. g. 58.44 g of NaCl dissolved in 1000 g of H<sub>2</sub>O is a 1m solution.

29.22 g of NaCl dissolved in 1000 g of H<sub>2</sub>O is a 0.5m solution.

Molarity (M): The number of moles of solute per liter or 1000 mL of solution. This is a concentration term.

E.g. If 58.44 g of NaCl are dissolved in H<sub>2</sub>O and the volume of the solution is 1000 mL, it is a 1M solution.

Molar concentrations are also indicated by square brackets. [HCl] = 1 means that a solution of HCl is one molar.

MOLARITY is probably the most common system for calculating the concentration of solutions.

Ex. 6 (continued)

#### MATERIALS/EQUIPMENT

Balance, volumetric glassware (see Exercise 5), sodium chloride, sodium sulfate, copper sulfate, alcohol, water.

#### PROCEDURE

1. Prepare 200 g of a 10% by mass solution of NaCl in water.
2. Prepare 200 mL of a 25% by volume solution of alcohol in water.
3. Prepare 100 mL of a 0.2M solution of copper sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in water.
  - Calculate formula weight.
  - Weigh desired quantity of solute.
  - Transfer to volumetric flask; add water to line.
4. Prepare 250 mL of a 0.2M solution of sodium sulfate ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ) in water.

For preparing solutions of definite molarity, the formula is:  
molecular mass of compound  $\times$  molarity wanted  $\times$  number of liters =  
grams of compound needed to make solution.

#### BIBLIOGRAPHY

ICCROM, Scientific Principles of Conservation Course, Course notes, 1977.

Shugar, G. - Shugar, R. - Bauman, L. - Bauman, R.S. Chemical technicians' ready reference handbook. New York: McGraw Hill, 1981.

Ex. 6 (continued)

#### Periodic Table of Elements

	1A																	2
1	H																	He
	1.00797																	4.0026
2	3 Li	4 Be															10 Ne	
	6.939	9.0122															20.18	
3	11 Na	12 Mg															18 Ar	
	22.9898	24.312															39.948	
4	19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
	39.102	40.08	44.956	47.90	50.942	51.996	54.9380	55.847	58.9332	58.71	63.54	65.37	69.72	72.59	74.9216	78.96	79.909	83.80
5	37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
	85.47	87.62	88.905	91.22	92.906	95.94	(99)	101.07	102.905	106.4	107.870	112.40	114.82	118.69	121.75	127.60	126.9044	131.30
6	55 Cs	56 Ba	57 La*	58 Hf	59 Ta	60 W	61 Re	62 Os	63 Ir	64 Pt	65 Au	66 Hg	67 Tl	68 Pb	69 Bi	70 Po	71 At	72 Rn
	132.905	137.34	138.91	178.49	180.948	183.85	186.2	190.2	192.2	195.09	196.967	200.59	204.37	207.19	208.980	(209)	(210)	(222)
7	87 Fr	88 Ra	89 Ac†															
	(223)	(226)	(227)															

\*Lanthanide Series

58	59	60	61	62	63	64	65	66	67	68	69	70	71
Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
140.12	140.907	144.24	(145)	150.35	151.96	157.25	158.924	162.50	164.930	167.26	168.934	173.04	174.97

†Actinide Series

90	91	92	93	94	95	96	97	98	99	100	101	102	103
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr
232.038	(231.036)	238.03	(237.048)	(244)	(243)	(247)	(247)	(251)	(254)	(257)	(258)	(259)	(260)

Groups designated in roman numerals.  
Periods designated in arabic numerals.

## Ex. 7 MEASUREMENT: pH

### AIM

It is often important for an architectural conservator to know whether a solution is acid, basic, or neutral. Such information is also critical in the analysis of meteorological agents such as rain or snow which could cause decay of building materials if excessively acid or alkaline (basic). The following experiment aims to explain the concept of pH and its measurement.

DEFINITIONS (Refer to BIBLIOGRAPHY for complete information). A solution is:

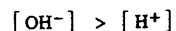
Acid if there are more hydrogen ions than hydroxyl ions.



Neutral if there are equal numbers of hydrogen and hydroxyl ions.



Basic (or Alkali) if there are more hydroxyl ions than hydrogen ions.



$[H^+] \times [OH^-]$  is a constant =  $10^{-14}$ .

pH is a value that represents the acidity or alkalinity of a solution.

It is defined as the logarithm of the reciprocal of  $[H^+]$ .

- A very acid solution has a pH of 1 (e.g. 0.1M HCl).
- A very basic solution has a pH of 14 (e.g. 1M caustic soda).
- A neutral solution has a pH of 7.

A buffer solution is one that tends to remain at a constant pH.

### MEASUREMENTS

The pH of a solution may be measured in three ways:

- 1) By means of *indicator solutions*, that is by dyes that change color at a given pH (see attached table).
- 2) With pH test paper or "strips". These are commercially available paper strips which have been impregnated with an indicator (test papers are available for every value of pH). The strip is wet with the solution to be tested and immediately compared with the standard color chart provided for each paper and range. The pH can be visually determined by comparison of colors.
- 3) With an electronic device known as the pH-meter.

## Ex. 7 (continued)

### EQUIPMENT/MATERIALS

pH indicator strips, pH meter, various indicator solutions, various test solutions, beakers, glass stirring rods, blotting paper.

### PROCEDURE

1. Using the pH strips, measure the pH of the solutions given to you. Record your results on the table below.
2. Repeat this procedure using the electronic pH meter. Record your results on the same table.

Solution	pH with strip	pH with meter
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

3. Check the pH range of the indicators given to you by adding a drop of the sample solutions of varying pH. Note color changes.

### BIBLIOGRAPHY

ICCROM, Scientific Principles of Conservation Course, Course notes, 1977.

Shugar, G. - Shugar, R. - Bauman, L. - Bauman, R.S. Chemical technicians' ready reference handbook. New York: McGraw Hill, 1981.

Torraca, G. Solubility and solvents for conservation problems. Rome: ICCROM, 1975.

INDICATOR SOLUTIONS

Name of indicator	pH range	Color change	Preparation
Methyl violet	0.2-3.0	Yellow to blue	0.05% dissolved in water
Cresol red	0.4-1.8	Red to yellow	0.1 g in 26 Ml 0.01 m NaOH + 200 mL H <sub>2</sub> O
Thymol blue	1.2-2.8	Red to yellow	Water + dil NaOH
Orange IV	1.3-3.0	Red to yellow	H <sub>2</sub> O
Benzopurpurin 4B	1.2-4.0	Violet to red	20% EtOH
Methyl orange	3.1-4.4	Red to orange-yellow	H <sub>2</sub> O
Bromphenol blue	3.0-4.6	Yellow to blue-violet	H <sub>2</sub> O + dil NaOH
Congo red	3.0-5.0	Blue to red	70% EtOH
Bromcresol green	3.8-5.4	Yellow to blue	H <sub>2</sub> O + dil NaOH
Methyl red	4.4-6.2	Red to yellow	H <sub>2</sub> O + dil NaOH
Chlorphenol red	4.8-6.8	Yellow to red	H <sub>2</sub> O + dil NaOH
Bromcresol purple	5.2-6.8	Yellow to purple	H <sub>2</sub> O + dil NaOH
Litmus	4.5-8.3	Red to blue	H <sub>2</sub> O
Alizarin	5.6-7.2	Yellow to red	0.1 g in MeOH
Bromthymol blue	6.0-7.6	Yellow to blue	H <sub>2</sub> O + dil NaOH
Phenol red	6.6-8.2	Yellow to red	H <sub>2</sub> O + dil NaOH
Thymol blue	8.0-9.6	Yellow to blue	H <sub>2</sub> O + dil NaOH
o-Cresolphthalein	8.2-9.8	Colorless to red	0.04% in EtOH
Phenolphthalein	8.3-9.8	Colorless to red	70% EtOH
Thymolphthalein	9.4-10.5	Yellow to blue	70% EtOH
Alizarin yellow R	10.0-12.0	Yellow to red	95% EtOH
Indigo carmine	11.4-13.0	Blue to yellow	50% EtOH
Trinitrobenzene 135	12.0-14.0	Colorless to orange	70% EtOH

(Shugar, 518)

POROUS BUILDING MATERIALS

## Ex. 8 WATER ABSORPTION BY TOTAL IMMERSION

### AIM

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

### DEFINITIONS

Water Absorption by Total Immersion: The quantity of water absorbed by a material immersed in deionized water at room temperature and pressure, expressed as a percentage of the dry mass of the sample.

Water Absorption Capacity: The maximum quantity of water absorbed by a material under the cited conditions, again expressed as a percentage of the dry mass of the sample.

### EQUIPMENT

Oven, dessicator, balance, plastic trays, cloth, glass stirring rods, silica gel, deionized water.

### PROCEDURE

1. Samples should be of regular form: cubes, cylinders or parallelepipeds. In the case of cubes, the side should not be less than 3 cm nor greater than 5 cm, so that the value of the ratio  $s/v$  (surface to apparent volume of the sample is between 2 and 1.2). Wash the samples in deionized water before beginning the test in order to eliminate powdered material from the surface.

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.

2. Dry the samples in an oven for 24 hours at 60°C; this relatively low drying temperature will prevent the deterioration of any organic substances employed in the case of treated samples. After drying, place the samples in a dessicator with silica gel to cool.

Ex. 8 (continued)

3. Weigh ( $M_0$ ) the samples. Repeat the drying process until the mass of the sample is constant; that is, until the difference between 2 successive weighings, at an interval of 24 hours, is not more than 0.1% of the mass of the sample.
4. Once the samples have been completely dried and the constant mass recorded, place them in a tray; preferably on supports (for example, glass stirring rods), and slowly pour deionized water into the tray until the samples are totally immersed and covered by about 2 cm of water.
5. At chosen intervals of time, take each sample out of the water, blot quickly with a damp cloth to eliminate surface moisture, and then weigh. Record the value obtained in the data sheet. Re-immerses the sample in water.
6. The choice of the intervals of time during the first 24 hours is dependent 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 at 8 hours from the beginning of the test and then every 24 hours until the quantity of water absorbed in two successive weighings is not more than 1% of the total mass.

7. At each interval, the quantity of water absorbed (WA) with respect to the mass of the dry sample is expressed as:

where  $M_n$  = weight of the wet sample at time  $t_n$   
 $M_0$  = weight of the dry sample

$$\frac{\Delta M}{M_0} \% = \frac{M_n - M_0}{M_0} \times 100$$

Record these values for water absorption in Data Sheet 1.

8. 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 (see steps 2 and 3). Record this value ( $M_d$ ) in the data sheet. Proceed to the Calculations.

Ex. 8 (continued)

CALCULATIONS

1. Using the data you have recorded, calculate the mean value of the water absorption (the sum of all values for WA divided by the number of samples) at each time interval. Express the results numerically in Data Sheet 2 and in graph form as a function of time (Graph 1).
2. Again, using the figures from Data Sheet 1, calculate the Water Absorption Capacity (WAC) with the following formula:

$$WAC = \frac{M_{max} - M_d}{M_d} \times 100$$

where:  $M_{max}$  = the mass of the sample at maximum water absorption

$M_d$  = the mass of the sample after redrying at the termination of the test (see step 8).

Record the result obtained in Data Sheet 2.

DISCUSSION

Compare your numerical results and graphics with those of other lab groups.

Which materials absorbed more water?

Which materials absorbed water at a faster rate?

When was the highest percentage of water absorbed for each material?

What factors do you think influence the varying water absorption capacities of each material?

What are the possible sources of error in this experiment?

BIBLIOGRAPHY

Italian NORMAL Committee. NORMAL 7/81. Assorbimento d'acqua per immersione totale; capacità di imbibizione. Rome: CNR, ICR, 1981.

Ex. 8 (continued)

DATA SHEET 1: WATER ABSORPTION BY TOTAL IMMERSION IN POROUS MATERIALS  
/ INDIVIDUAL RESULTS

SAMPLE N°		1	2	3	4	5	6	7	8
	$M_0$								
$t_1 =$	$M_1$								
5 min	$WA_1$								
$t_2 =$	$M_2$								
15 min	$WA_2$								
$t_3 =$	$M_3$								
30 min	$WA_3$								
$t_4 =$	$M_4$								
1 hour	$WA_4$								
$t_5 =$	$M_5$								
8 hrs	$WA_5$								
$t_6 =$	$M_6$								
24 hrs	$WA_6$								
$t_7 =$	$M_7$								
48 hrs	$WA_7$								
$t_8 =$	$M_8$								
72 hrs	$WA_8$								
$t_9 =$	$M_9$								
96 hrs	$WA_9$								
	$M_d$								

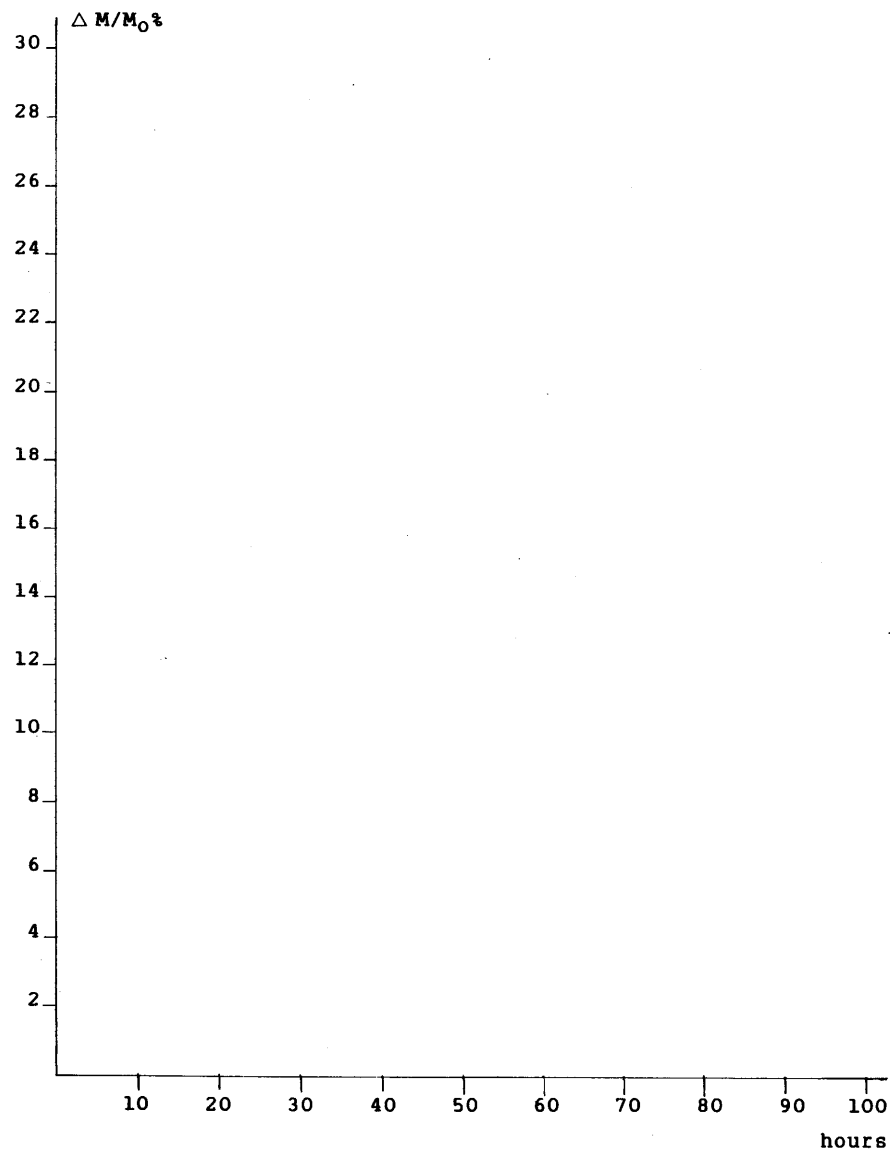
Ex. 8 (continued)

DATA SHEET 2: WATER ABSORPTION BY TOTAL IMMERSION IN POROUS MATERIALS  
/ MEAN VALUES

Material Type: _____	
Form of Sample: _____	
Dimensions: _____	
Number of Samples: _____	
TIME (t)	Mean Water Absorption ( $\Delta M/M_0$ % / number of samples)
$t_1$	
$t_2$	
$t_3$	
$t_4$	
$t_5$	
$t_6$	
$t_7$	
$t_8$	
$t_9$	
WAC = _____ achieved at t = _____	

Ex. 8 (continued)

GRAPH 1: WATER ABSORPTION CURVE / MEAN VALUES



Ex. 9 WATER DROP ABSORPTION

#### AIM

This exercise presents a simple method for the measurement of changes in the properties of stone surfaces caused either by treatment with hydrophobic (water repellent), consolidating, or hygroscopic materials, or by weathering. The measurement of water drop absorption is therefore a laboratory test for both treated and untreated stone. It is especially appropriate for determining the water repellency of stone surfaces.

#### DEFINITIONS

Water drop absorption rate: the absorption time of a limited and definite amount of water by the surface of a material.

#### EQUIPMENT

Oven, dessicator, common laboratory buret, deionized water.

#### PROCEDURE

1. Take treated and untreated samples and dry them in an oven at 60°C for 24 hours (or until the difference between two successive weighings is not more than 0.1% of the mass of the sample). Allow the samples to cool at room temperature.
2. Place the buret, filled with deionized water, at a distance of 1 cm from the sample surface.
3. Drip 1 mL of water onto the horizontal sample surface.
4. The time required for the total absorption of the water is determined as ( $t_x$ ) for treated or weathered samples, ( $t_n$ ) for untreated or unweathered reference samples.
5. Determine the evaporation time ( $t_e$ ) of the drop during the measurement procedure by dripping 1 mL of water onto an analogous rough glass surface.
6. Expression and interpretation of results:

If treated samples exhibit longer absorption times in relation to those of reference samples, this indicates a reduction of stone porosity by a consolidating treatment, or a water repellency of the surface caused by a hydrophobic impregnation.

Totally water-repellent surfaces do not absorb water; in this case, the time in which the drop disappears is equal to its evaporation time ( $t_e$ ).

Ex. 9 (continued)

If treated samples exhibit shorter absorption times in relation to those of the reference samples, this indicates an increase in the stone porosity caused by weathering or a hygroscopicity of the surface induced by hygroscopic impregnation.

In order to be compared, the absorption times measured on different samples of stone must be transformed onto a relative scale. The following equations have been found to provide a useful scale for figures:

The water drop absorption (WA) of treated and weathered surfaces is calculated as a percentage (the absorption of the reference untreated surfaces being set equal to 100%):

$$WA (\%) = \left[ 1 - \frac{t_x - t_n}{t_x} \right] \times 100$$

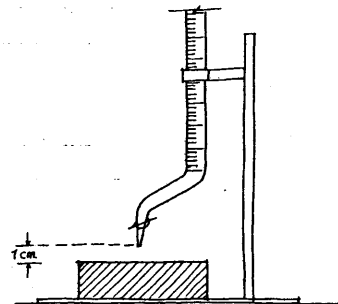
$t_x$  = absorption time into a treated or weathered surface  
 $t_n$  = absorption time into the reference untreated surface

If  $t_x > 0.05$  ( $t_e$ ), the evaporation time ( $t_e$ ) has to be taken into consideration by using this formula:

$$WA (\%) = \left[ 1 - \frac{t_x - t_n}{t_e - t_n} \cdot \frac{t_e}{t_x} \right] \times 100$$

Water repellency (WR) may be expressed as follows:

$$WR (\%) = 100 - WA$$



BIBLIOGRAPHY

Unesco, RILEM. Water drop absorption. In: Deterioration and protection of stone monuments: experimental methods. Volume V, Test II.8b. International Symposium, Unesco, Paris, 1978.

Ex. 10 PENETRATION OF WATER: CAPILLARY ACTION

AIM

Water is almost always a principal cause of the deterioration of porous materials. The movement of water is related to the physical characteristics of the material such as its structure, porosity, capillarity, and permeability. The main sources of water are rain, condensation, and capillarity. This simple experiment simulates the action of rising damp by capillarity.

EQUIPMENT

Tray, ruler, water.

PROCEDURE

1. Place a brick upright in a container as indicated in Fig. 1. Add water to the container until 1 cm of the base of the brick is immersed.

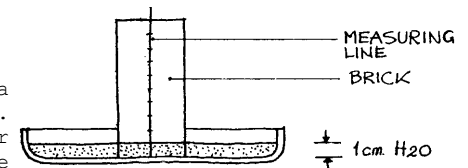


Fig. 1

2. Measure the height (H) of the rising damp every minute for the first 5 minutes, then every 5 minutes for the next 25 minutes, then every 30 minutes. Record the results below.

Time (minutes)	Height of water (cm) (Sample 1)	Height of water (cm) (Sample 2)
1		
2		
3		
4		
5		
10		
15		
20		
25		
30		
60		
90		
120		
150		
180		

Ex. 10 (continued)

3. Draw a graph with the data obtained:  
Height (H) versus (t) as in Fig. 2 and  
height (H) versus the square root of time as in Fig. 3.

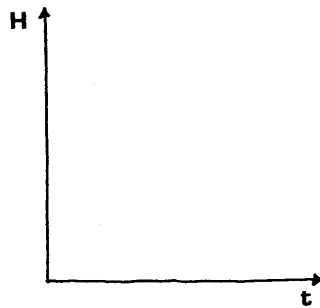


Fig. 2

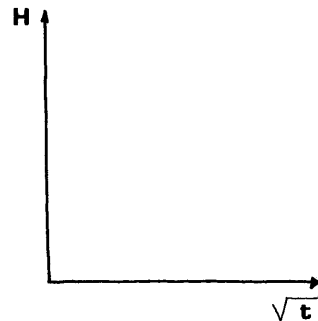


Fig. 3

#### BIBLIOGRAPHY

- Massari, G. Humidity in monuments. Rome: ICCROM, 1971.
- Mora, P. Causes of deterioration of mural paintings. Rome: ICCROM, 1974.

#### Ex. 11 POROSITY OF GRANULAR BEDS

##### AIM

The aim of this test is to measure the total porosity of a granular material by means of its apparent and real densities (the latter obtained by a fluid displacement method).

##### DEFINITIONS

Porosity E: The fraction of the total volume of a solid that is occupied by pores (more simply, the empty spaces or voids in a solid mass). The % Porosity = % Voids.

Permeability: The ability to transmit liquid or gas from one place to another.

Capillarity: The attraction between molecules, both like and unlike, which results in the rise of a liquid in small tubes or fibers or in the wetting of a solid by a liquid.

Porosimetry: The study of pore-size distribution. The volumetric distribution of the open pores, assumed to be of circular section, according to their size (radius or diameter), usually expressed as a percentage of the mass of the sample. This property is related to capillarity, which increases in inverse proportion to the pore diameter (i.e. capillarity is greater where there are smaller pores).

Apparent Volume ( $V_a$ ): The volume of a sample including the pore space.

Real Volume ( $V_r$ ): The apparent volume of the sample minus the volume of its pore space accessible to water.

Apparent Density ( $p_a$ ): The ratio of the mass to the apparent volume of the sample expressed in  $\text{kg/m}^3$ .

Real Density ( $p_r$ ): The ratio of the mass to the real (or impermeable) volume of the sample expressed in  $\text{kg/m}^3$ .

Principle of Archimedes: If a solid body is wholly or partially immersed in a liquid, the upthrust force (or buoyancy force) acting on the body is equal to the weight force of the liquid displaced by the body, and acts vertically upwards through the center of gravity of the displaced liquid. The apparent mass of a solid body immersed in water is therefore equal to the mass of the body less the mass of water displaced.

When a body floats in water, the mass of water displaced is equal to the mass of the body, whether it is totally or partially immersed.

Ex. 11 continued)

#### CALCULATIONS

Porosity  $\epsilon$  is generally calculated by means of the following formula:

$$\epsilon = 1 - \frac{\rho_a}{\rho_r} \quad \text{where:}$$

$\rho_a$  = apparent density as defined above AND

$\rho_r$  = real density as defined above.

The formula is derived in the following way:

$$\begin{aligned} \text{POROSITY} &= \frac{\text{Volume of pores}}{\text{Apparent volume of sample}} \\ &= \frac{V_a - V_r}{V_a} \\ &= \frac{V_a}{V_a} - \frac{V_r}{V_a} \\ &= 1 - \frac{V_r}{V_a} \end{aligned}$$

$$\text{since: } \rho_a = \frac{\text{mass}}{V_a}$$

$$\text{and } \rho_r = \frac{\text{mass}}{V_r}$$

$$\text{then } \frac{\rho_a}{\rho_r} = \frac{V_r}{V_a}$$

thus, substituting  $\frac{\rho_a}{\rho_r}$  for  $\frac{V_r}{V_a}$ , we arrive at:

$$\text{POROSITY } \epsilon = 1 - \frac{\rho_a}{\rho_r}$$

$$\% \text{ POROSITY} = \left[ 1 - \frac{\rho_a}{\rho_r} \right] \times 100$$

Note: Porosity has no units.

Ex. 11 (continued)

#### EQUIPMENT

Oven, balance sensitive to 0.1 g, graduated cylinder, sample containers.

#### PROCEDURE

1. Weigh the sample container ( $M_c$ ).
2. Place granular sample A (previously dried in the oven at  $60^\circ\text{C} \pm 5^\circ\text{C}$  for 24 hours) in the sample container and weigh ( $M_1$ ).

$$M_1 - M_c = \text{Mass of sample } (M_s).$$

3. Calculate the apparent volume ( $V_a$ ) of the sample by placing it in the graduated cylinder. Since for practical purposes  $1 \text{ mL} = 1 \text{ cm}^3$  the volume of the sample can be read directly from the cylinder scale. (Obviously, your reading will change depending on how closely packed the sand particles are. Shake the cylinder a few times to strike a reasonable balance between the densest and loosest compaction states.)
4. From the data obtained in Steps 2 and 3, calculate the apparent density ( $\rho_a$ ):

$$\rho_a = \frac{M_s}{V_a}$$

Record all values on the attached data sheet.

5. Next, calculate the real volume of the sample by placing it in a cylinder containing a known volume of water. The amount of water displaced is equal to the real volume ( $V_r$ ).
6. Calculate the real density ( $\rho_r$ ):

$$\rho_r = \frac{M_s}{V_r}$$

Record these values on the attached data sheet.

7. From the values obtained in Steps 4 and 6, calculate the % Porosity ( $\%, \epsilon$ ) for the sample:

$$\% \epsilon = \left[ 1 - \frac{\rho_a}{\rho_r} \right] \times 100$$

8. Repeat the above procedure for each of the granular samples.

Record all results on the data sheet.

DISCUSSION

- Can you draw any conclusions about the relationship of grain size to porosity?
- Was there any noticeable difference between the porosity of the samples of homogeneous grain size and those of mixed particle sizes? Can you draw any conclusions about the relationship between porosity and porosimetry? between porosity and permeability? between porosimetry and permeability?
- What are the possible sources of error in this experiment?

BIBLIOGRAPHY

Columbia University, Program in Historic Preservation, Conservation Science Course, Laboratory exercises, 1983.

Rossi-Doria, Paola. Some laboratory tests on stone. Course notes for the ICCROM Scientific Principles of Conservation Course, 1983.

SAMPLES	$M_s$	$V_a$	$\rho_a$	$V_r$	$\rho_r$	% porosity
	g	cm <sup>3</sup>	g/cm <sup>3</sup>	cm <sup>3</sup>	g/cm <sup>3</sup>	$\left[1 - \frac{\rho_a}{\rho_r}\right] \times 100$
A						
B						
C						
D						
E						

Ex. 12 POROSITY IN SOLIDS: INDIRECT MEASUREMENT BY  
WATER ABSORPTION

AIM

This exercise offers a simple method for the approximate measurement of porosity in solids. It can be viewed as a follow-up to Exercise 8: Water Absorption by Total Immersion.

PROCEDURE

Using the data obtained in Exercise 8, estimate the percent porosity of each sample as described below:

1. Initial mass of the sample =  $M_o$
2. Mass of the saturated sample =  $M_{max}$
3. Mass of Pores =  $M_{max} - M_o$

Since the density of water (w/v) is 1 g/cm<sup>3</sup> at 4°C, this value can also be considered the volume of the pores ( $V_p$ ).

4. Measure the apparent volume of the saturated sample by placing it in a beaker containing a known quantity of water. According to Archimedes' Principle (see definition in Ex. 11), the quantity of water displaced is equal to the apparent volume ( $V_a$ ).

5. The % Porosity = % Voids

An estimate of the % Porosity (assuming that all pores are accessible and the sample is saturated, i.e. assuming a good relationship between porosity and permeability) can, therefore, be obtained by the following:

$$\% \text{ Porosity} = \frac{V_p}{V_a} \times 100$$

Record all values in the table below.

SAMPLES	$M_o$	$M_{max}$	$M_p$ ( $V_p$ )	$V_a$	% Porosity
1					
2					
3					
4					

Ex. 12 (continued)

DISCUSSION

- What conclusions can you draw about the relationship between water absorption capacity and porosity?
- Which of the materials tested was most porous? Which was least porous? How do you think these values would affect the weatherability of the material under varying climatic conditions? How would they influence the effectiveness of certain conservation treatments?

### Ex. 13 POROSITY IN SOLIDS: HYDROSTATIC WEIGHING

#### AIM

The aim of this test is to measure the total porosity of a material by means of its apparent and real densities (a "fluid displacement" method). This can be useful for assessing the extent of some types of stone decay and for determining the extent to which pore space has been filled by an impregnation treatment.

#### DEFINITIONS

**Porosity E:** The fraction of the total volume of a solid that is occupied by pores (more simply, the empty spaces or voids in a solid mass). The % Porosity = % Voids.

**Permeability:** The ability to transmit liquid or gas from one place to another.

**Capillarity:** The attraction between molecules, both like and unlike, which results in the rise of a liquid in small tubes or fibers or in the wetting of a solid by a liquid.

**Porosimetry:** The study of pore-size distribution. The volumetric distribution of the open pores, assumed to be of circular section, according to their size (radius or diameter), usually expressed as a percentage of the mass of the sample. This property is related to capillarity, which increases in inverse proportion to the pore diameter (i.e. capillarity is greater where there are smaller pores).

**Apparent Volume ( $V_a$ ):** The volume of a sample including the pore space.

**Real Volume ( $V_r$ ):** The apparent volume of the sample minus the volume of its pore space accessible to water.

**Apparent Density ( $p_a$ ):** The ratio of the mass to the apparent volume of the sample expressed in  $\text{kg/m}^3$ .

**Real Density ( $p_r$ ):** The ratio of the mass to the real (or impermeable) volume of the sample expressed in  $\text{kg/m}^3$ .

**Principle of Archimedes:** If a solid body is wholly or partially immersed in a liquid, the upthrust force (or buoyancy force) acting on the body is equal to the weight force of the liquid displaced by the body, and acts vertically upwards through the center of gravity of the displaced liquid. The apparent mass of a solid body immersed in water is therefore

### Ex. 13 (continued)

equal to the mass of the body less the mass of water displaced.

When a body floats in water, the mass of water displaced is equal to the mass of the body, whether it is totally or partially immersed.

#### CALCULATIONS

Porosity  $\varepsilon$  is generally calculated by means of the following formula:

$$\varepsilon = 1 - \frac{P_a}{P_r} \quad \text{where:}$$

$P_a$  = apparent density as defined above AND

$P_r$  = real density as defined above.

#### EQUIPMENT

Oven, balance, tray.

#### PROCEDURE

1. Samples can have the form of a cylinder, cube, or prism. Their volume must be at least  $25 \text{ cm}^3$ .
2. Dry the samples at  $60^\circ\text{C} \pm 5^\circ\text{C}$  until constant mass. (The constant mass is reached when the difference between two successive weighings, at an interval of 24 hours, is not more than 0.1% of the mass of the sample.) The drying temperature of  $60^\circ\text{C}$  is chosen instead of a higher one to avoid deterioration of any organic materials used to treat the stone.
3. Weigh the samples:  $M_1$  (in grams).
4. Place the samples in a tray, cover with tap water at  $15\text{--}20^\circ\text{C}$ , and leave immersed for 24 hours.
5. Take each sample out of the tray and weigh it immersed in water: that is, suspended by a wire hook from the plate of the balance into a container of water (hydrostatic weighing). Let  $M_2$  (in grams) be the mass of the sample immersed in water. Quickly wipe the previously immersed sample with a damp cloth (in order to remove surface moisture) and then weigh it in air on the plate of the balance. Let  $M_3$  (in grams) be the mass of the sample saturated with water.

Ex. 13 (continued)

RESULTS

- The volume of the pores ( $V_p$ ), in  $\text{cm}^3$ , is expressed by  $M_3 - M_1$  (water density at  $20^\circ\text{C}$  considered to be 1.0).
- The apparent volume is:  $M_3 - M_2$  (In the case of stones with large pores, the apparent volume is determined by measuring the dimensions of the sample. A gauge with a precision of 0.1 mm should be used.)
- The real volume is:  $M_1 - M_2$
- Real Density ( $\rho_r$ ) =  $\frac{M_1}{M_1 - M_2} \times 10^3$
- The apparent volume is:  $M_3 - M_2$
- Apparent Density ( $\rho_a$ ) =  $\frac{M_1}{M_3 - M_2} \times 10^3$
- POROSITY  $\epsilon = 1 - \frac{\rho_a}{\rho_r}$
- % POROSITY =  $\left[1 - \frac{\rho_a}{\rho_r}\right] \times 100$
- Record your results in the data sheet on the next page.

BIBLIOGRAPHY

Rossi-Doria, Paola. Some laboratory tests on stone. Course notes for the ICCROM Scientific Principles of Conservation Course, 1983.

Unesco, RILEM. Bulk and real densities. In: Deterioration and protection of stone monuments: experimental methods. Volume V, Test I.2. International Symposium, Paris: Unesco, 1978.

## DATA SHEET: POROSITY IN SOLIDS / HYDROSTATIC WEIGHING

SAMPLE						
$M_1$						
$M_2$						
$M_3$						
$V_p (M_3 - M_1)$						
$V_a (M_3 - M_2)$						
$V_r (M_1 - M_2)$						
$\rho_r$						
$\rho_a$						
% POROSITY $\left[1 - \frac{\rho_a}{\rho_r}\right] \times 100$						

#### Ex. 14 MOVEMENT OF SALTS

##### AIM

This simple experiment illustrates how soluble salts are transported by water and how they damage porous materials. Water-soluble salts can originate from the soil, the air, or from the materials themselves. They are transported inside the materials by water capillarity or by other means. When water evaporates, the salts come toward the surface of the material and crystallize (change from liquid state to solid state) on the surface or close to it. These salts, in crystallized form, cause breaking and spalling of the surface.

##### EQUIPMENT/CHEMICALS

Oven, tray, distilled water, sodium sulfate decahydrate ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ).

##### PROCEDURE

1. Dry a sample in the oven for 24 hours at 60°C.
2. Weigh it: ( $M_0$ ).
3. Put it in a tray containing 1 cm of a 10% solution of sodium sulfate decahydrate in distilled water and let the sample soak for a minimum of two weeks.
4. Maintain the solution at constant level, and observe the crystallization of salt day by day.
5. Take the sample out of the solution. Dry it in the oven for 24 hours and then weigh it: ( $M_1$ ).
6. The quantity of salts absorbed is  $M_1 - M_0$ . Record all results below.

SAMPLES	$M_0$ (g)	$M_1$ (g)	Quantity of salts (g) $M_1 - M_0$
1			
2			
3			

##### BIBLIOGRAPHY

Massari, G. Humidity in monuments. Rome: ICCROM, 1971.

Mora, P. Causes of deterioration of mural paintings. Rome: ICCROM, 1974.

#### Ex. 15 SALT CRYSTALLIZATION

##### AIM

This test is useful to assess the success of a preservation treatment. It can also be used as an artificial weathering test.

##### EQUIPMENT/CHEMICALS

Oven, beaker, rod, tray, cover for tray, clamps, sodium sulfate decahydrate ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ), silica gel.

##### PROCEDURE

1. Use both treated and untreated samples of porous building materials.
2. Dry the samples in the oven at 60°C for 24 hours and then place them in a dessicator with silica gel to cool.
3. Weigh the samples ( $M_0$ ) when they are cool, and then totally immerse them in a 14% solution of sodium sulfate decahydrate. A saturated solution can be used for stones of greater durability or to simulate harsher weathering conditions.
4. After the samples have been immersed for 24 hours, dry them for 24 hours in the oven and then cool in the dessicator. Weigh each sample ( $M_1$ ) and then immerse again in the solution.
5. Continue the drying - immersion cycle (weighing the samples each time) until there is evidence of macroscopic deterioration or until the samples are completely destroyed.

##### BIBLIOGRAPHY

ICCROM, Scientific Principles of Conservation Course, Course exercises.

Rossi-Doria, Paola. Some laboratory tests on stone. Course notes for the ICCROM Scientific Principles of Conservation Course, 1983.

Ex. 16 QUALITATIVE ANALYSIS OF WATER-SOLUBLE SALTS AND CARBONATES

AIM/INTRODUCTION

The presence of white efflorescence on the surface of masonry is always an indication of chemical deterioration processes resulting from the reaction of three components: the materials themselves, water, and polluting compounds present in the water or the atmosphere. (A fourth component can be contributed by micro-organisms.)

The deterioration products resulting from the reaction between these three components are water-soluble salts, principally sulfates, chlorides, nitrites and nitrates. Under certain conditions, calcium carbonate (a normal component of mortars and calcareous stones), practically insoluble in water, can also be a deterioration product, usually appearing in the form of a surface incrustation.

Qualitative analysis of soluble salts (from a stone or mortar sample) furnishes information about the types of ions (sulfates, chlorides, etc.) present in the sample and gives an indication of the maximum quantity of single ions present. Such information provides some clues as to the type of deterioration in progress and its causes.

Origins of the Most Common Salts

(1) Sulfates

The sulfates most commonly found in masonry are hydrated calcium sulfate (gypsum,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) and, more rarely, magnesium sulfate ( $\text{MgSO}_4$ ).

The possible origins of such salts are the following:

- a) Sulfates are present in agricultural land and can enter a wall through capillary action.
- b) Sea water, in addition to chlorides, contains small amounts of sulfates, especially magnesium sulfate. Sea spray can thus deposit sulfates on a surface.
- c) Some materials used in the preparation of mortars and plasters can contain small quantities of sulfates as impurities. These can be dissolved in water present in the masonry wall and brought to the surface as efflorescence. Erroneous use of substances like gypsum for restoration can lead to the presence of sulfates.

Ex. 16 (continued)

- d) Another possible origin of sulfates is microbiological. In brief, there are certain types of micro-organisms capable of metabolizing reduced forms of sulfur and oxidizing it to sulfates, as well as others which produce sulfides instead. These "sulfur bacteria" are often present on exposed stone, especially calcareous types. Since there is a strict analogy between calcareous stones and mortars based on calcium carbonate, it is logical to assume that these bacteria could also develop in plasters and mortars.
- e) The most important source of sulfates, however, is atmospheric pollution. The burning of hydrocarbons leads to the transformation of the sulfur they contain into sulfur dioxide ( $\text{SO}_2$ ) emitted into the atmosphere as a gas.

Reacting with oxygen ( $\text{O}_2$ ) sulfur dioxide ( $\text{SO}_2$ ) becomes sulfur trioxide (or sulfuric anhydride,  $\text{SO}_3$ ).

This last product ( $\text{SO}_3$ ) reacts with water ( $\text{H}_2\text{O}$ ) to form sulfuric acid ( $\text{SO}_3 + \text{H}_2\text{O} = \text{H}_2\text{SO}_4$ ) which attacks the calcium carbonate ( $\text{CaCO}_3$ ) and transforms it into calcium sulfate ( $\text{CaSO}_4$ ).

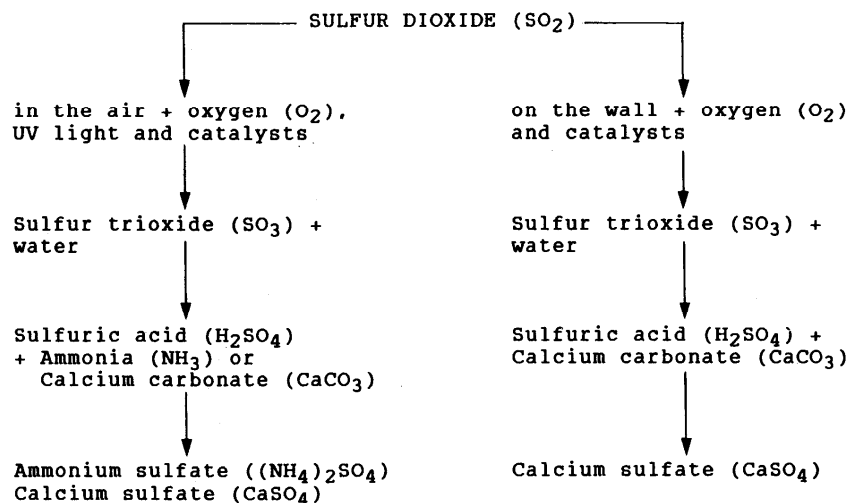
All these processes can happen in the wall. Alternately, the sulfuric acid can form in the air and then react with the calcium carbonate ( $\text{CaCO}_3$ ) of the wall.

A third possibility is that the sulfuric acid formed in the air is neutralized by basic substances such as ammonia ( $\text{NH}_3$ ), forming ammonium sulfate ( $(\text{NH}_4)_2\text{SO}_4$ ), or by calcium carbonate present as atmospheric dust, forming calcium sulfate (gypsum,  $\text{CaSO}_4$ ). In a polluted environment, these sulfate solids can constitute 20 to 30% of the dust in the atmosphere.

As a final possibility, studies have shown that sulfur dioxide ( $\text{SO}_2$ ) can be absorbed directly or as sulfurous acid ( $\text{H}_2\text{SO}_3$ ). This leads to the formation of calcium sulfite ( $\text{CaSO}_3 \cdot 2\text{H}_2\text{O}$ ) which oxidizes to sulfate.

The processes described above are summarized in the chart on the following page:

Ex. 16 (continued)



(2) Chlorides

These salts (especially sodium chloride (NaCl) and calcium chloride (CaCl<sub>2</sub>)) are principally deposited on a wall by sea spray.

Chlorides can also be the result of impurities in the materials (particularly the sand) used to prepare mortars and plasters.

Finally, some types of industrial activity, like the combustion of certain kinds of pit-coal, can result in the presence of gaseous hydrochloric acid (hydrogen chloride, anhydrous (HCl)) in the atmosphere.

(3) Nitrites and Nitrates

Nitrites are not often found in walls because they oxidize rapidly into nitrates.

The decomposition of nitrogenous organic material produces nitrites. Thus, nitrites might be present where there is infiltration of sewage water or in the vicinity of old burial sites.

In general, however, nitrates are much more frequent. They can have the same origin as nitrites, but can also come from the earth in agricultural regions.

Ex. 16 (continued)

Atmospheric pollution can also produce nitrates. The combustion of hydrocarbons, in addition to creating sulfur dioxide (SO<sub>2</sub>), also produces various organic molecules and nitrogen oxides. These nitrogen oxides are extremely dangerous to animal and vegetable life because, in the presence of ultraviolet light, they react with oxygen and the organic molecules present in the polluted atmosphere, forming ozone and organic radicals. The ozone, in turn, oxidizes these radicals to aldehydes and the sulfur dioxide (SO<sub>2</sub>) to sulfur trioxide (SO<sub>3</sub>). This particular mix of substances produces a dangerous fog called "photochemical smog" which can be found in areas having severe pollution and long periods of strong sunlight (eg. Los Angeles or Naples). The nitrogen oxides, through a series of complex reactions, form nitric acid (HNO<sub>3</sub>) which, reacting with calcium carbonate (CaCO<sub>3</sub>), leads to the formation of calcium nitrate (Ca(NO<sub>3</sub>)<sub>2</sub> · 4H<sub>2</sub>O).

(2) Carbonates

Calcium carbonate is a normal constituent of both calcareous stones and of mortars (where it is formed by the carbonization of lime).

Unlike the other salts of efflorescence, calcium carbonate is practically insoluble in water. It can, however, be dissolved as bicarbonate when the water contains a high enough quantity of carbon dioxide (CO<sub>2</sub>).

Carbon dioxide is a gas that is normally present in the atmosphere. Its concentration can increase, however, under particular conditions - such as in the case of certain industrial activity or when a large number of people occupy a closed room. If dissolved in water present in a humid wall, carbon dioxide forms carbonic acid (H<sub>2</sub>CO<sub>3</sub>) which reacts with calcium carbonate (CaCO<sub>3</sub>), forming the more soluble bicarbonate.

There is thus an equilibrium between these various substances (carbonates, CO<sub>2</sub> + water, bicarbonates) which leads to the production of soluble bicarbonates when there is a high concentration of carbon dioxide (CO<sub>2</sub>).

When a wall begins to dry, bicarbonate salts in solution come to the surface. As evaporation takes place, the previously established equilibrium shifts in favor of the formulation of calcium carbonate which, being practically insoluble, is rapidly deposited on the surface.

Ex. 16 (continued)

- (5) NOTE: It should be remembered that cement can contain several soluble alkaline salts besides sulfates, nitrites, and nitrates, which are added to give the final product particularly desired characteristics. Thus, if cement has been used in a building where there is some humidity in the walls, the soluble salts present in the cement can migrate toward the original plasters or mortars, causing destructive efflorescence or crystallization upon evaporation.

EQUIPMENT

Mortar and pestle, test tubes, funnels, filter paper.

CHEMICALS

- Dilute hydrochloric acid (2N)
- Dilute nitric acid (2N)
- Dilute acetic acid (2N)
- Sulfamic acid
- Barium chloride (10% solution in water)
- Solution of silver nitrate (0.1N)
- Zinc powder
- Griess-Ilosvay's reagent

PROCEDURE

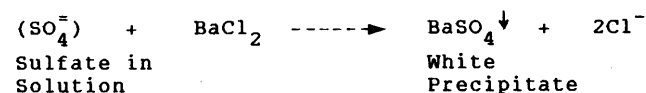
1. Grind the sample to a fine homogeneous powder in a small mortar and pestle; a few milligrams of sample are sufficient for qualitative analysis.
2. Put half of the sample so obtained in a 10 cc test tube for the following analyses and conserve the rest for an eventual control.
3. Add about 2 cc of distilled or deionized water to the test tube and shake gently to dissolve the material.
4. Wait a few minutes until the insoluble part of the sample is deposited at the bottom of the test tube. The solution must be clear; otherwise, it is necessary to filter it using fine filter paper and a small funnel.
5. Conserve the test tube containing the insoluble part of the sample for the analysis of carbonates. Split the clear solution into 4 equal parts, putting each part in a small test tube.

Ex. 16 (continued)

- \* For the best possible control of experimental results, it is necessary to execute, simultaneously, so-called "white" reactions. That is, for each of the following tests, the analysis of the actual solution should be followed by an analysis using only the water (the same water used for the preparation of the solutions). The results of the "white" reactions can then be compared with those obtained using the sample solution.

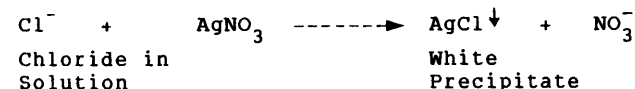
6. Analysis of Sulfates ( $\text{SO}_4$ )

- Use one of the solutions in the test tubes.
- Add 1 or 2 drops of dilute *hydrochloric acid* ( $\text{HCl}$  2N) and 1 or 2 drops of a 10% solution of *barium chloride* ( $\text{BaCl}_2$ ).
- A white precipitate of *barium sulfate* ( $\text{BaSO}_4$ ), insoluble in dilute nitric acid, indicates the presence of sulfates.
- The reaction can be summarized as follows:



7. Analysis of Chlorides ( $\text{Cl}^-$ )

- Use the solution in the second test tube.
- Add 1 or 2 drops of dilute *nitric acid* ( $\text{HNO}_3$  2N) and 1 or 2 drops of a solution (0.1N) of *silver nitrate* ( $\text{AgNO}_3$ ).
- A whitish-blue, gelatinous precipitate of *silver chloride* ( $\text{AgCl}$ ) indicates the presence of chlorides.
- The reaction can be summarized as:



8. Analysis of Nitrites ( $\text{NO}_2$ )

- Use the solution in the third test tube.
- Add 1 or 2 drops of *dilute acetic acid* ( $\text{CH}_3\text{COOH}$  2N) and 1 or 2 drops of *Griess-Hosvay's reagent*.
- A more or less intense pink color indicates the presence of nitrites.

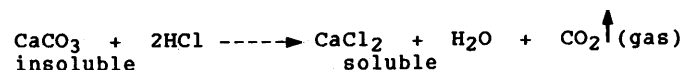
Ex. 16 (continued)

#### 9. Analysis of Nitrates (NO<sub>3</sub>)

- (a) Absence of nitrites: If there are no nitrites (i.e. if the previous reaction was negative), add to the same solution a small quantity of *zinc powder*. The zinc, in the presence of acetic acid, will reduce the nitrates (if present) to nitrites. These will then react with the Griess-Ilosvay's reagent. In this case, therefore, a more or less intense pink color indicates the presence of nitrates.
- (b) Presence of nitrites: If nitrites were present in the solution utilized for step 8, use the solution in the fourth test tube for this analysis. Add a small quantity one or two crystals of *sulfamic acid* (HSO<sub>3</sub>NH<sub>2</sub>) in order to destroy the nitrites. (Use the method employed in Step 8 on a small part of the solution to be sure that all nitrites are destroyed. If not, continue to add small quantities of sulfamic acid until they are no longer present. Avoid adding an excessive amount of sulfamic acid to the solution.)
- Having eliminated the nitrites, add to the solution 1 or 2 drops of dilute *acetic acid* (CH<sub>3</sub>COOH 2N) and 1 or 2 drops of *Griess-Hosvay's reagent*. Now the solution will not turn pink because the nitrites have been destroyed.
  - Add a small amount of *zinc powder*. A more or less intense pink color indicates the presence of nitrates.

#### 10. Analysis of Carbonates (CO<sub>3</sub>)

- Use the insoluble part of the original sample remaining at the bottom of the large test tube.
- Add 1 or 2 drops of concentrated *hydrochloric acid* (HCl).
- Bubbles of gas (CO<sub>2</sub>) in the solution indicate the presence of carbonates (CO<sub>3</sub>).
- The reaction can be summarized as follows:



Ex. 16 (continued)

#### 11. Expression of Results

The current method of expressing results is to indicate the presence of an ion with a cross (+) and its absence with a minus (-). The relative abundance of an ion is usually indicated by a proportionate number of crosses (++, +++, etc.). The following example should clarify this usage:

SAMPLE	Sulfates	Chlorides	Nitrites	Nitrates	Carbonates
1	++	±	-	±	+
2	+++	+	-	+	+
3	etc.				

- = absence of the ion

± = concentration of the ion at the limit of perceptibility

+ = presence of the ion

++ = presence of the ion in notable quantity

+++ = presence of the ion as a principal component

#### DISCUSSION

Using the example above as a model, record your experimental results in the Data Sheet.

Can you make any hypotheses about the origins of the various salts discovered in analysis?

What other analyses might you carry out to verify your hypotheses?

#### BIBLIOGRAPHY

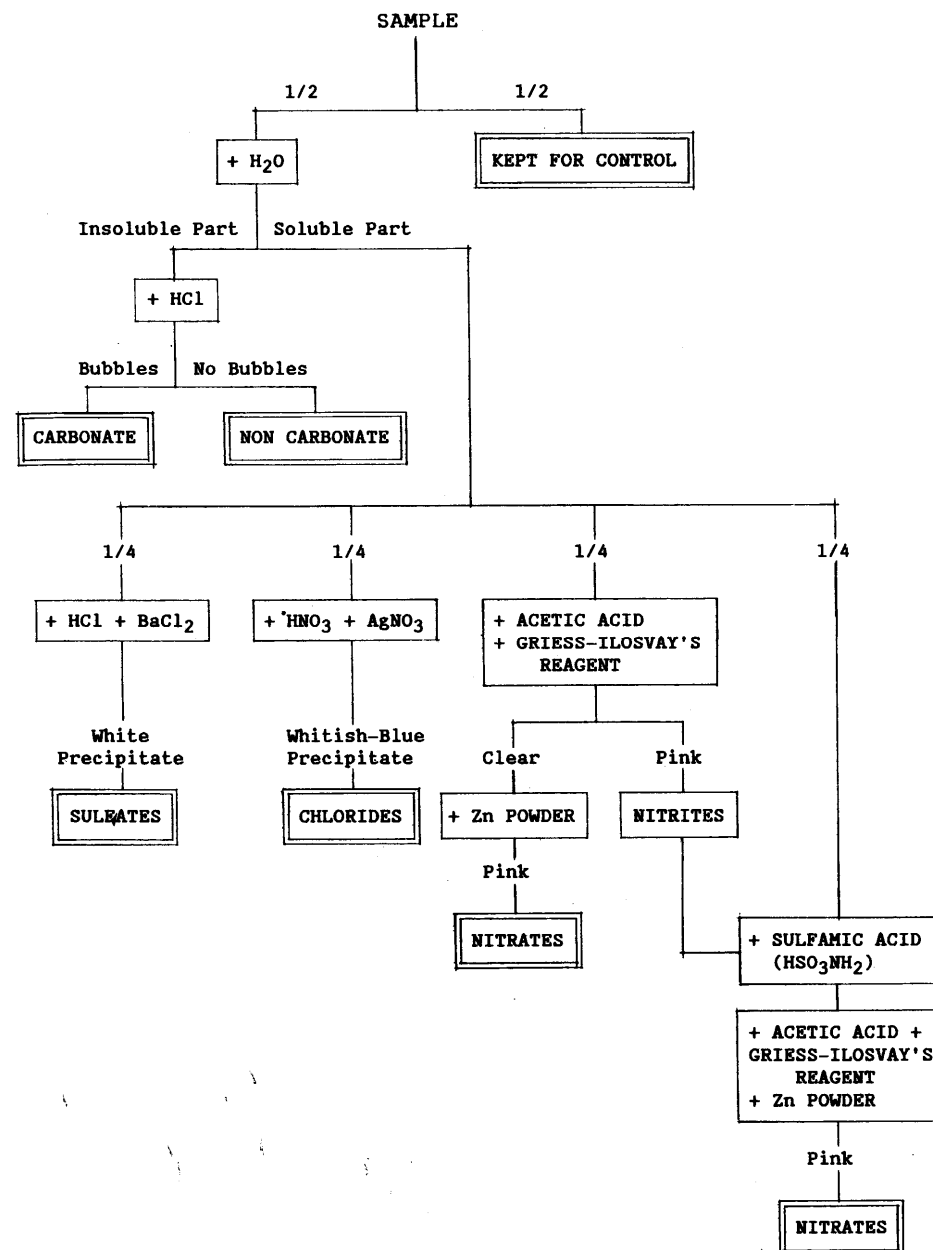
Istituto Centrale del Restauro, Corso sulla manutenzione di dipinti murali, mosaici, stucchi (DIMOS). Part II, Modulo 3. Rome: Istituto Centrale del Restauro, 1979.

Ex. 16 (continued)

DATA SHEET

SAMPLE	Sulfates	Chlorides	Nitrites	Nitrates	Carbonates

Ex. 16 (continued)



## Ex. 17 SEMIQUANTITATIVE ANALYSIS OF WATER-SOLUBLE SALTS

DATA SHEETAIM/PREMISES

Measurement is based on the principle that the intensity of color assumed by the solution after addition of specific reagents is proportional to the concentration of the ions analyzed. This exercise will provide a basic familiarity with the range of products available for the approximation of ion concentration in solution.

EQUIPMENT

Technical balance (0.01 g), flask (500 cc), pipet, small test tubes, filter paper, various test kits and paper strips.

PROCEDURE

1. Weigh 1 gram of sample powder. Put the weighed powder in a 500. cc flask and fill halfway to the mark with deionized water. Stopper the flask, shake and turn it upside down for a few minutes. Let the solution rest for a few minutes and then add deionized water to the mark.
2. Filter off the undissolved particles on dry filter paper before analysis.
3. Following the instructions in the test kits, analyze samples of the solution for the salts that are present in it. Use the filtered solution and dilute it (1:5) if the concentration of one of the salts is too high.
4. Record your results in the data sheet according to the convention established in exercise 16.

BIBLIOGRAPHY

Istituto Centrale del Restauro. Corso sulla manutenzione di dipinti murali, mosaici, stucchi (DIMOS). Part II-3: Analisi° dei prodotti di deterioramento; schede delle strumentazioni. Rome: Istituto Centrale del Restauro, 1979.

SAMPLE	Sulfates	Chlorides	Nitrites	Nitrates	Carbonates

## EARTHEN BUILDING MATERIALS

REFERENCES: ASTM D422-63; BS1377: 1975, Test 7(B)

INTRODUCTION/AIM

A soil consists of an assemblage of discrete particles of various shapes and sizes. The types and relative proportions of these particles give the soil a particular character and behavior. The aim of particle size analysis (or particle size distribution tests) is to group these particles into separate ranges of sizes and so determine the relative proportions, by dry weight, of each size range.

Particle size distribution tests can be divided into two basic groups: sieving procedures for "coarse" particles (gravel and sand) and sedimentation procedures for "fine" particles (silt and clay). The procedure described below is a composite method for the analysis of soils containing both coarse and fine particles. Part I outlines the preparation of the sample and the sieving procedure. Part II describes the sedimentation procedure for fine particles.

DEFINITIONS

Particle size is usually given in terms of equivalent particle diameter. Definitions vary according to the standard consulted. The British Standard employs the following definitions:

- Gravel: Particles from 2 mm to 60 mm.
- Sand: Particles from 60 µm to 2 mm.
- Silt: Particles from 2 µm to 60 µm.
- Clay: Particles smaller than 2 µm.

The American (ASTM) standard is slightly different. A comparison of the ASTM with the British system is shown below.

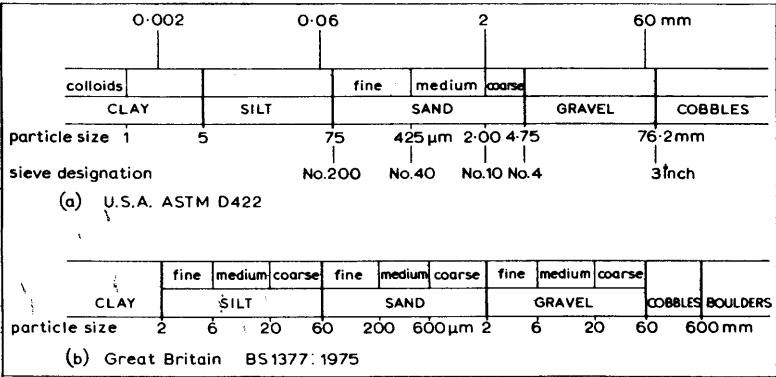


Fig. 1

## Ex. 18A (continued)

EQUIPMENT/CHEMICALS

- Mortar with rubber-covered pestle
- Balance sensitive to 0.01 gram
- Standard drying oven
- A series of sieves, of square mesh woven wire cloth, ranging in size from 20 mm to 75  $\mu$ m (see Procedure for exact sizes)
- Mechanical sieve shaker
- Sieve brushes
- Porcelain evaporation dish
- 600 ml beaker
- 250 ml cylinder
- Glass stirring rod
- Magnetic stirrer with teflon-coated stirring rod, 5 cm long
- 4% Solution of sodium hexametaphosphate (40 grams dissolved in 1 liter of distilled water)
- Data sheet

PROCEDURE

1. Sample: The most important requirement for any test sample is that it be fully representative of the material from which it is taken. Both the quantity of the sample material and the selection procedure are extremely important in this regard.

Quantity: For testing purposes, a sample smaller than the received sample is usually required. The actual quantity to be analyzed is usually determined by the size of the largest particles present. For clays, silts, and sand, a sample size of about 100 grams is sufficient; with gravel size particles, much larger quantities are required. The following table can be considered a guide to the minimum quantity of material required for particle size distribution tests. The recommendations relate to the maximum size of particle present in substantial proportion' (more than 10% of the total sample):

Maximum size of material present in substantial proportion retained on BS sieve (mm)	Minimum mass of sample to be taken for sieving	Source
Pass 2mm or smaller	100 g	Based on BS 1377:1975, Section 1.5.4.2 (5)
6.3	200 g	
10	500 g	
14	1 kg	
20	2 kg	
28	6 kg*	
37.5	15 kg	
50	35 kg	
63	50 kg	
75	70 kg	
100	150 kg	Author's suggestion
150	500 kg	
200	1000 kg	

Fig. 2

Selection: Once the desired quantity of test material has been established, select the test sample from the original material so that it is truly representative (a single scoopful from a bag of material does not, for example, truly represent the whole). With cohesive soils (silts and clays), this should be done by randomly spooning the soil and regularly remixing the remaining stock until the necessary quantity is obtained.

2. Sample Preparation: Once a suitable sample is selected, crush it lightly in a mortar with a rubber-covered pestle to break down aggregations of particles. The rubber-covered pestle prevents the crushing of individual particles which must remain intact for particle size analysis.

After crushing, place the sample in an evaporating dish and dry overnight in an oven maintained at 105-110°C. After drying to constant weight, allow the whole specimen to cool, and then weigh it to the nearest 0.01 g.\*

- \* There is some disagreement about the effects of oven drying on test results. ASTM D421 recommends only air-drying of soil samples, as does the British Standard. Nevertheless, whereas oven drying may have a deleterious effect on samples used for the plastic limit test, it is usually not a significant factor for the normal use of the particle size curve. It can therefore be safely used in the preparation of samples for particle size analysis.

3. Pre-treatment/Dispersion of Sample: Clayey and silty soils must first be pre-treated for the subsequent sedimentation test. This pre-treatment involves the use of a dispersing agent to ensure complete separation (or dispersion) of discrete particles of soil in the silt to clay range.

Numerous substances have been tried as dispersing agents, but the usual choice is sodium hexametaphosphate (1.1 Na<sub>2</sub>O : 1 P<sub>2</sub>O<sub>5</sub>) known commercially as Calgon.

This is prepared in a standard 4% solution by mixing 40 grams of dry material with enough distilled water to make 1000 mL. The solution should be freshly mixed and never over 1 month old.

After weighing, transfer the dried and crushed sample material to a 600 mL beaker and cover with about 200 mL of 4% sodium hexametaphosphate solution. Stir until the soil is completely wet and allow to stand for at least one hour (some standards recommend an overnight standing period).

After this standing period, complete the dispersion of the particles by using a magnetic stirrer. Successful results have been achieved by stirring the mixture at high speed for 15 minutes using a 5 cm Teflon-coated stirring bar.

Ex. 18A (continued)

4. Washing: Transfer the soil and solution to a 75  $\mu\text{m}$  (N°200) sieve nested on a receiver. Carefully wash the soil with a jet of distilled water until all fine material is washed through the sieve (until the water is clear). The amount of water should not exceed 500 mL. Be very careful not to lose any soil by splashing the material out of the sieve or by allowing the water to overflow.

The material collected in the receiver will be used for the appropriate sedimentation test (Particle Size Analysis: Part II). Put it aside.

5. Transfer (by backwashing) the material retained on the 75  $\mu\text{m}$  sieve to a weighed evaporating dish and let it stand for a short period of time until the top of the suspension becomes clear. Pour off as much of the clear water as possible; then place the dish with the remaining soil/water suspension in the oven for drying.
6. Sieving Procedure: When the material in the evaporating dish is dry, allow it to cool and weigh it.

Immediately thereafter, run the sample through a stack of sieves varying from larger sizes to smaller sizes from the top down. A sieve stack usually consists of 6 or 7 sieves with the sizes approximately doubling in opening from the bottom to the top sieve (as 6, 12, 24 mm, ...). Some recommended sieve stacks (see footnotes) for sandy to fine-grained soils are shown below:

Brockville<sup>1</sup>

N° 4	4.75 mm
10	2.00 mm
20	850 $\mu\text{m}$
40	425 $\mu\text{m}$
60	250 $\mu\text{m}$
140	106 $\mu\text{m}$
200	75 $\mu\text{m}$

NBS<sup>2</sup>

N°16	1.18 mm
30	600 $\mu\text{m}$
50	300 $\mu\text{m}$
100	150 $\mu\text{m}$
200	75 $\mu\text{m}$
270	53 $\mu\text{m}$
400	38 $\mu\text{m}$

ASTM<sup>3</sup>

N° 4	4.75 mm
8	2.36 mm
16	1.18 mm
30	600 $\mu\text{m}$
50	300 $\mu\text{m}$
100	150 $\mu\text{m}$
200	75 $\mu\text{m}$

Ex. 18A (continued)

Place the stack of sieves in a mechanical shaker for 5 to 10 minutes, depending on the quantity of material. If this is not available, shake by hand for about 10 minutes.

Remove the stack of sieves from the shaker and record the weight of material remaining on each sieve. Any particles lodged in the apertures of the sieve should be carefully removed with a sieve brush. Weights should be recorded to the nearest 0.01 g on the test sheet (attached).

Sum the weights and compare to the weight obtained at the beginning of Step 5. A loss of more than 2 percent by weight of the retained sample is considered unsatisfactory and the test should be repeated.

Any material passing the 75  $\mu\text{m}$  sieve should be added to the material for the sedimentation test.

7. Calculations: In order to draw up a particle size distribution curve, it is necessary to calculate the cumulative percentage (by mass) of particles finer than each sieve aperture size -- that is, passing each sieve. This can be done in the following way (see sample data sheet: Fig. 3):

First: Compute the percent retained on each sieve by dividing the weight retained on each sieve by the original weight obtained in Step 2. Record on the attached data sheet.

Second: Compute the percent passing (or percent finer) by starting with 100 percent and subtracting the percent retained on each sieve as a cumulative procedure. For example, as seen in the sample data sheet (Fig. 3), the quantity of 9.7 g was retained on the N°4 sieve; the percent retained is  $9.7/500 \text{ g} \times 100 = 1.9\%$ . The percent passing (not retained) =  $100\% - 1.9\%$  or  $98.1\%$ .

On the N°10 sieve, 39.5 g were retained; the percent retained is  $39.5/500 \text{ g} \times 100 = 7.9\%$ . The percent passing is  $98.1\%$  (that retained on the previous sieve) -  $7.9\% = 90.2\%$ . Stated simply:

$$\text{Percent passing} = \text{Percent arriving} - \text{Percent retained}$$

8. Graph: Make a semilogarithmic plot of grain size versus percent finer using the graph on the attached data sheet (see sample, Fig. 4).
9. Sedimentation Test: Using the portion of fine sample set aside in Sep 4 (plus any particles that passed the 75  $\mu\text{m}$  sieve during dry sieving), proceed to the sedimentation procedure: Exercise 18B: Particle Size Analysis/Part II.

SAMPLE DATA SHEET: GRAIN SIZE ANALYSIS-MECHANICAL

$$\% \text{ passing} = 100 - \sum \% \text{ retained.}$$

**Fig. 3**

SAMPLE DATA SHEET: GRAIN SIZE DISTRIBUTION

## System

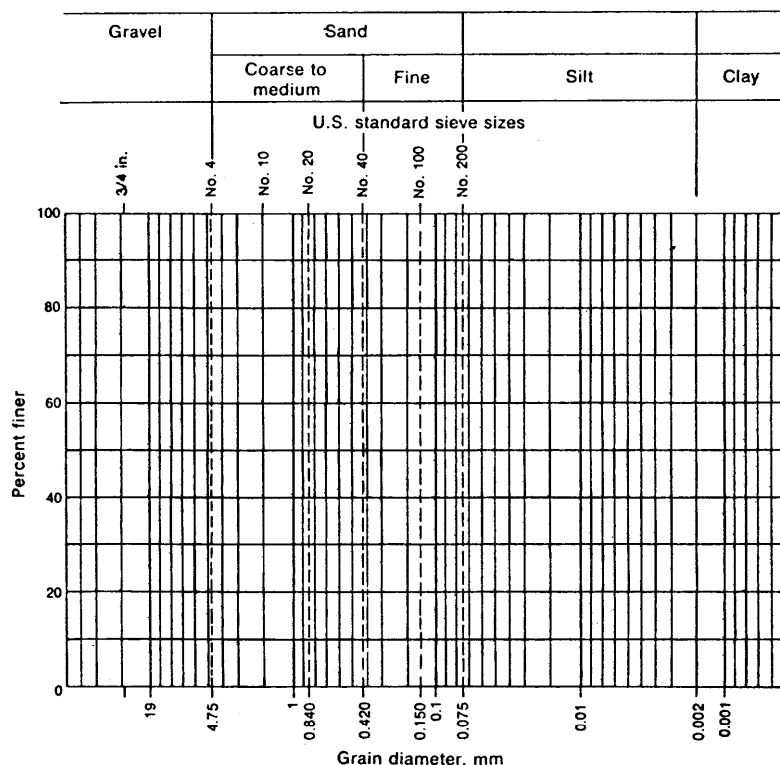
Fig. 4. Curve B constructed with data from Fig. 3.



Ex. 18A (continued)

DATA SHEET: GRAIN SIZE DISTRIBUTION

Project \_\_\_\_\_ Job No. \_\_\_\_\_  
 Location of Project \_\_\_\_\_ Boring No. \_\_\_\_\_ Sample No. \_\_\_\_\_  
 Description of Soil \_\_\_\_\_ Depth of Sample \_\_\_\_\_  
 Tested By \_\_\_\_\_ Date of testing \_\_\_\_\_



Visual soil description \_\_\_\_\_

Soil classification:

\_\_\_\_\_ System \_\_\_\_\_

Ex. 18B PARTICLE SIZE ANALYSIS: PART II SEDIMENTATION  
 PROCEDURE: HYDROMETER METHOD

INTRODUCTION / SEDIMENTATION THEORY

The theory of sedimentation is based on the fact that large particles in suspension in a liquid settle more quickly than small particles, assuming that all particles have similar densities and shapes. The velocity which a falling particle eventually reaches is known as its terminal velocity. If the particles are approximately spherical, the relationship between terminal velocity ( $V$ ) and the particle diameter ( $D$ ) is given by Stokes's Law (after the English physicist, Sir George Stokes, 1891). This law states that the terminal velocity is proportional to the square of the particle diameter or:

$$V \propto D^2$$

Though clay particles are certainly not spherical, the application of Stokes's Law provides a basis for the comparison of particle size distribution in fine soils which is sufficiently realistic for most practical purposes.<sup>1</sup>

SEDIMENTATION PROCEDURE: HYDROMETER METHOD

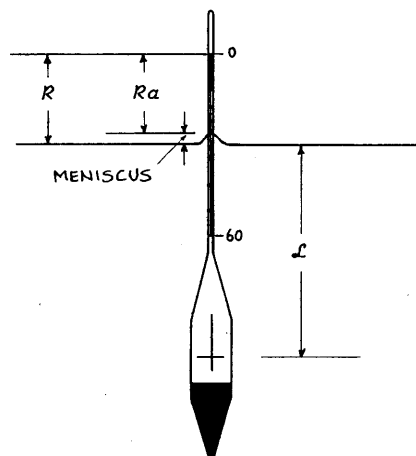
In a sedimentation procedure, a suspension of a known mass of fine soil particles of various sizes is made up in a known volume of water. The particles are allowed to settle under gravity. Since they settle at different rates, the number of each particle size in suspension will change as a function of time. Thus, by measuring the density of the suspension at a constant depth at known intervals of time and then applying Stokes's Law, we can assess the distribution of particle sizes.

A special instrument called an *hydrometer* (Fig. 1) is used to measure the specific gravity of the soil-water suspension at a particular depth (the center of the hydrometer bulb). Any soil grains larger than those still in suspension in the zone known as  $L$  (the distance from the center of the bulb to the water surface) have fallen below the center of the volume, and this decreases the specific gravity (or density) of the suspension at the center of the bulb. Also, since the hydrometer is a constant weight, the *lower* the specific gravity of the suspension, the *deeper* the hydrometer will sink into the suspension. Temperature must be carefully controlled because the density of water decreases as the temperature rises (or falls) from 4°C. ;

Ex. 18B (continued)

#### EQUIPMENT

- Soil Hydrometer calibrated to read either specific gravity of the suspension or grams per liter of suspension. The latter type (specifically Model 152H) is recommended by ASTM:



$L$  = Effective depth

$Ra$  = Actual hydrometer reading

$R$  = Hydrometer reading corrected for meniscus

Fig. 1

- Two 1000 mL glass measuring cylinders about 330 mm high marked at 1000 mL, with stoppers or rubber bungs (also called hydrometer jars).
- Constant temperature bath (optional)
- Glass rod, about 12 mm in diameter and 400 mm in length
- Thermometer
- Stopwatch
- Plastic wash bottle

#### PROCEDURE

1. Preparation of the Suspension: Take the suspension of pretreated soil which passed the 75  $\mu$ m sieve in Part I (plus any material which passed the 75  $\mu$ m sieve in dry sieving) and transfer to one of the 1000 mL sedimentation cylinders. Add distilled water<sup>2</sup> to the 1000 mL mark.

Fill the second cylinder (the control cylinder for<sup>3</sup> holding the hydrometer between readings) with distilled water.

Ex. 18b (continued)

Place both cylinders in the constant temperature bath set at 25°C, until they have reached the bath temperature. If a bath is not available, make sure the distilled water temperature is adjusted so that both the sedimentation and control cylinders are at the same temperature.

Take a rubber stopper and cap the sedimentation cylinder to obtain a water-tight fit. Carefully shake the cylinder to obtain a uniform suspension. If necessary, stir the sediment with a glass rod to make sure it all goes into suspension. Invert the cylinder for a few seconds and then place it in an upright position (in the constant temperature bath, if used).

Start the stopwatch.

2. Hydrometer Readings: Remove the rubber stopper from the sedimentation cylinder and steadily insert the hydrometer so that it floats freely in the suspension. It must not be allowed to bob up and down or to rotate when let go.

Take readings at the top of the meniscus level (see Fig. 2) at the following times from zero: 1/2, 1, 2, and 4 minutes. Also take a thermometer reading.

Slowly remove the hydrometer, rinse in distilled water, and place it in the control cylinder (which should be within 1°C of the soil-water suspension). Do the same with the thermometer. Take a meniscus reading in the control cylinder on the hydrometer.

Replace the rubber stopper in the sedimentation cylinder, reagitrate the suspension, and take another set of 1/2, 1, 2, and 4 minute readings. Repeat until you obtain two sets of readings that agree within one unit of each other for all four readings. (Insert and withdraw the hydrometer carefully to avoid unnecessary agitation. Each operation should take about 10 seconds).

When agreement between readings is reached, take additional readings at elapsed times of 8, 15, 30, 60 minutes and 1, 2, 4, 8, 16, 32, 64, 96 hours. It is not necessary to keep rigidly to these times as long as the time of each reading is carefully recorded on the data sheet (attached).

Record the temperature of the soil-water suspension to the nearest 1°C for each hydrometer reading.

After each reading, remove both the hydrometer and the thermometer and place in the control cylinder (which should be at the same temperature).

3. Correction of Hydrometer Readings: Each hydrometer reading ( $R_a$ ) must be subjected to four corrections. These are:

- (a) Meniscus correction ( $C_m$ )
- (b) Temperature correction ( $C_t$ )
- (c) Dispersing agent correction ( $x$ )
- (d) Correction for unit weight of solids (a)

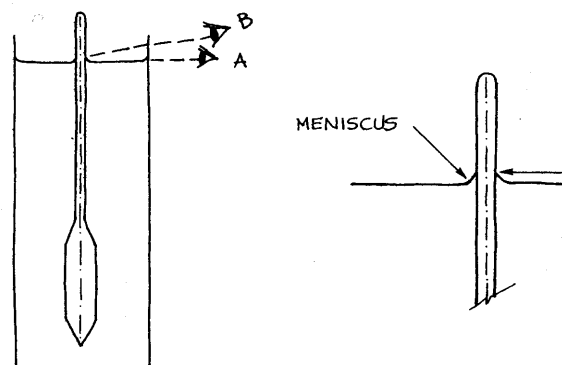
These are obtained as described below.

(a) Meniscus Correction: A hydrometer is calibrated to read correctly at the surface of the liquid in which it is immersed (bottom of the meniscus). Soil suspensions, however, are not clear enough to allow a reading at this level, so the scale has to be read at the upper rim of the meniscus. A meniscus correction ( $C_m$ ) must therefore be added to each actual' reading ( $R_a$ ) in order to obtain the true reading. The  $C_m$  is a constant for each hydrometer and is calculated as follows:

Insert the hydrometer in a 1000 mL cylinder three quarters full of distilled water. The plane of the surface of the liquid is seen as an ellipse from just below the surface. Raise the eye until the surface is seen as a straight line and take a reading (A) (see Fig. 2). Take a second reading at the upper level of the meniscus (B). The meniscus correction is the difference between the two readings multiplied by 1000.

$$C_m = (B - A) \times 1000$$

The true hydrometer reading ( $R$ ) =  $R_a$  (actual reading) -  $C_m$



(b) Temperature correction ( $C_t$ ): Hydrometers are calibrated at 20°C. If a test is carried out at a different temperature, both the density of the water and the density of the hydrometer (owing to thermal expansion of the glass) will be different. These factors are corrected by constant values given in Table 1. The value of  $C_t$  is always added to the true hydrometer reading ( $R$ ).

(c) Dispersing agent correction ( $x$ ): The addition of the dispersing agent results in the density of the liquid in which sedimentation takes place being greater than that of water. The correction ( $x$ ) can be determined in two ways:

1. To determine the correction ( $x$ ), a volume of exactly 50 mL of the standard dispersing solution is placed in a weighed evaporating dish. The water is evaporated by oven drying at 105-110°C and the mass of the remaining dispersing agent ( $M_d$ ) is determined.

The correction ( $x$ ) =  $2M_d$

This is subtracted from the true hydrometer reading ( $R$ ).

2. Alternatively, the correction ( $x$ ) is determined by using a sedimentation cylinder of water from the same source, at the same temperature, and with the same quantity of dispersing agent as used in the soil-water suspension. A reading of less than zero in this standard cylinder of water is recorded as a (-) value; a reading between 0 and 60 is recorded as a (+) value. All readings are taken at the top of the meniscus. Again, the correction ( $x$ ) is subtracted from ( $R$ ). This is also called the "zero correction".

(d) Correction for unit weight of solids (a): The hydrometer is calibrated to read grams of soil of a value of  $G_s = 2.65$  in 1000 cm<sup>3</sup> of suspension at a temperature of 20°C. If the specific gravity ( $G_s$ ) of the soil grains is not 2.65, a correction factor (a) must be computed. Typical values are given in Table 2.

To facilitate calculations, a combined correction factor ( $K$ ) which combines both temperature and unit weight of solids is usually adopted. ( $K$ ) values are shown in Table 3.

Ex. 18B (continued)

4. Calculations:

(a) Apply the meniscus correction to the hydrometer readings and use Table 4 to obtain values of L (effective depth). Record these on the attached data sheet.

(b) If  $G_s$  is not known, assume a reasonable value between 2.68 and 2.74. With  $G_s$  and the test temperature for any hydrometer reading, use Table 3 to obtain the K value.

(c) With the values of K, L and the elapsed time (t), compute the values of D (particle diameter) using the following equation:

$$D = K \sqrt{\frac{L}{t}} \text{ mm}$$

Use sample computations (Fig. 3) and sample data sheet (Fig. 4) as a guide.

Record values for each (t) on the data sheet.

(d) Calculate a corrected value of  $R_c$  using the equation:

$$R_c = R_{\text{actual}} - \text{zero correction (x)} + C_t \text{ (Table 1)}$$

(e) Calculate the percent finer for the corresponding particle diameters (D) by using the formula:

$$\text{Percent finer} = \frac{R_c(a)}{W_s} \times 100$$

where  $R_c$  = corrected hydrometer reading  
 $W_s$  = weight of original soil sample in suspension  
 $a$  = unit weight of solids correction (Table 2)

Record values on the data sheet.

(f) Using the data from steps (a) to (e) above, plot the percent finer versus grain size either on the graph used for the dry sieve analysis or on a new graph.

Ex. 18B (continued)

Fig. 3 SAMPLE COMPUTATIONS

$$R_c = R_a - \text{zero correction} + C_t \text{ (} C_t \text{ from Table 1)}$$

$$R_c = 49 - 3 + 0.40 = 46.4 \quad a = 0.99 \text{ (from data sheet)}$$

$$\text{Percent finer} = \frac{R_c a}{W_s} \times 100 = \frac{46.4(0.99)(100)}{50.0} = 91.9\%$$

$$\text{Hydrometer corrected for meniscus only} = 49 + 1 = 50$$

$$L = 8.1 \text{ (from Table 4)}$$

$$v = \frac{L}{t} = \frac{8.1}{1} = 8.1$$

$$K = 0.0131 \text{ (from Table 3)}$$

$$D = K \sqrt{L/t} = 0.0131 \sqrt{8.1} = 0.037 \text{ mm} \quad (\text{Do not compute more digits than you can plot on the graph using log scale})$$

From line 3 of data sheet

$$R_c = 43 - 3 + 0.4 = 40.4$$

$$\text{Percent finer} = \frac{40.4(0.99)(100)}{50.0} = 80.0\%$$

$$\text{Hydrometer corrected for meniscus} = 43 + 1 = 44$$

$$L = 9.1 \text{ (from Table 4)}$$

$$K = 0.0131 \text{ (from Table 3)}$$

$$D = 0.0131 \sqrt{9.1/3} = 0.023 \text{ mm}$$

From last line of data sheet

$$R_c = 10 - 3 + 1.0 = 8.0$$

$$\text{Percent finer} = \frac{8.0(0.99)(100)}{50.0} = 15.8\%$$

$$\text{Hydrometer corrected for meniscus} = 10 + 1 = 11$$

$$L = 14.5 \text{ (from Table 4)}$$

$$K = 0.0127 \text{ (from Table 3)}$$

$$D = 0.0127 \sqrt{14.5/2850} = 0.0009 \text{ mm}$$

Ex. 18B (continued)

Fig. 4 SAMPLE DATA SHEET

Project Hydrometer Analysis Job No.       

Location of Project Bradley University Boring No.        Sample No.       

Description of Soil Brown Silty Clay Depth of Sample       

Tested By JEB Date of Testing 3/4/76

Hydrometer analysis

Hydrometer no. 152H  $G_s$  of solids = 2.72  $a =$  0.99

Dispersing agent NaPO<sub>3</sub> (Calgon) Amount 4.2 in 125 ml Wt. of soil,  $W_s$  50.0 g

Zero correction +3.0 Meniscus correction 1.0

Date	Time of reading	Elapsed time, min	Temp., °C	Actual Hyd. reading $R_u$	Corr. Hyd. reading $R_c$	% Finer	Hyd. Corr. only for meniscus, $R$	$L$ from Table 6-5	$L$	$K$ from Table 6-4	$D$ , mm
3-4	3:30 P.M.	1	22°	49	46.4	91.9	50	8.1	8.1	0.0131	0.037
		2		47	44.4	87.9	48	8.4	4.2		0.027
		3		43	40.4	80.0	44	9.1	3.03		0.023
		4		42	39.4	78.0	43	9.2	2.3		0.020
		8		37	34.4	68.1	38	10.1	1.26		0.015
		16		31	28.4	56.2	32	11.1	0.69		0.011
		30		26	23.4	46.3	27	11.9	0.597		0.0082
	4:30	60		24	21.4	42.4	25	12.2	0.203		0.0059
	5:35	125		21	18.4	36.4	22	12.7	0.102	0.0131	0.0042
	9:00	330	23°	18	15.7	31.1	19	13.2	0.040	0.0129	0.0026
3-5	8:00 A.M.	990	23°	16	13.7	27.1	17	13.5	0.014	0.0129	0.0015
	3:00 P.M.	1410	24°	14	12.0	23.8	15	13.8	0.0098	0.0127	0.0013
3-6	3:00 P.M.	2850	24°	10	8.0	15.8	11	14.5	0.0051	0.0127	0.0009
Line 2: % Finer = $\frac{44.4(0.99)(100)}{50} = 87.9\%$											
$D = 0.0131 \sqrt{\frac{87.9}{2}} = 0.027 \text{ mm}$											
** Compute $D$ to 2 significant digits for plotting											

$$R_c = R_{\text{actual}} - \text{zero correction} + C_T$$

$$\% \text{ finer} = R_c(a)/W_s$$

$$D = K\sqrt{L/H}$$

Ex. 18B (continued)

CORRECTION TABLES 1,2,3

Table 1: Temperature Correction Factors  $C_T$

Temp. (°C)	$C_T$
15	-1.10
16	-0.90
17	-0.70
18	-0.50
19	-0.30
20	0.00
21	+0.20
22	+0.40
23	+0.70
24	+1.00
25	+1.30
26	+1.65
27	+2.00
28	+2.50
29	+3.05
30	+3.80

Table 2: Correction Factors (a) for Unit Weight of Solids

Unit weight of soil solids (g/cm <sup>3</sup> )	Correction factor (a)
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.04

Table 3: Values of K for Several Unit Weights of Soil Solids and Temperature Combinations

UNIT WEIGHT OF SOIL SOLIDS (g/cm <sup>3</sup> )								
Temp. (°C)	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.0151	0.0148	0.0146	0.0144	0.0141	0.0139	0.0137	0.0136
17	0.0149	0.0146	0.0144	0.0142	0.0140	0.0138	0.0136	0.0134
18	0.0148	0.0144	0.0142	0.0140	0.0138	0.0136	0.0134	0.0132
19	0.0145	0.0143	0.0140	0.0138	0.0136	0.0134	0.0132	0.0131
20	0.0143	0.0141	0.0139	0.0137	0.0134	0.0133	0.0131	0.0129
21	0.0141	0.0139	0.0137	0.0135	0.0133	0.0131	0.0129	0.0127
22	0.0140	0.0137	0.0135	0.0133	0.0131	0.0129	0.0128	0.0126
23	0.0138	0.0136	0.0134	0.0132	0.0130	0.0128	0.0126	0.0124
24	0.0137	0.0134	0.0132	0.0130	0.0128	0.0126	0.0125	0.0123
25	0.0135	0.0133	0.0131	0.0129	0.0127	0.0125	0.0123	0.0122
26	0.0133	0.0131	0.0129	0.0127	0.0125	0.0124	0.0122	0.0120
27	0.0132	0.0130	0.0128	0.0126	0.0124	0.0122	0.0120	0.0119
28	0.0130	0.0128	0.0126	0.0124	0.0123	0.0121	0.0119	0.0117
29	0.0129	0.0127	0.0125	0.0123	0.0121	0.0120	0.0118	0.0116
30	0.0128	0.0126	0.0124	0.0122	0.0120	0.0118	0.0117	0.0115

CORRECTION TABLE 4

Table 4: Values of L (Effective Depth) for Use in Stokes's Formula for Diameters of Particles for ASTM Soil Hydrometer 152H

Original hydrometer reading (corrected for meniscus only)	Effective depth L (cm)	Original hydrometer reading (corrected for meniscus only)	Effective depth L (cm)	Original hydrometer reading (corrected for meniscus only)	Effective depth L (cm)
0	16.3	21	12.9	42	9.4
1	16.1	22	12.7	43	9.2
2	16.0	23	12.5	44	9.1
3	15.8	24	12.4	45	8.9
4	15.6	25	12.2	46	8.8
5	15.5	26	12.0	47	8.6
6	15.3	27	11.9	48	8.4
7	15.2	28	11.7	49	8.3
8	15.0	29	11.5	50	8.1
9	14.8	30	11.4	51	7.9
10	14.7	31	11.2	52	7.8
11	14.5	32	11.1	53	7.6
12	14.3	33	10.9	54	7.4
13	14.2	34	10.7	55	7.3
14	14.0	35	10.5	56	7.1
15	13.8	36	10.4	57	7.0
16	13.7	37	10.2	58	6.8
17	13.5	38	10.1	59	6.6
18	13.3	39	9.9	60	6.5
19	13.2	40	9.7		
20	13.0	41	9.6		

FOOTNOTES (see Bibliography for complete references)

1. Head, K.H., pp. 182-183.
2. ASTM recommends the use of distilled water, as does the British Standard. Other authors (see Brockville laboratory exercises) feel that tap water is satisfactory for most laboratory purposes.
3. Some authors recommend using the dispersant solution in the control cylinder as well as the sedimentation cylinder (see Brockville laboratory exercises).

FIGURE CREDITS (see Bibliography for complete references)

- Fig. 2 Head, K.H., p. 202.
- Fig. 3 Brockville laboratory exercises, p. 56.
- Fig. 4 Brockville laboratory exercises, p. 57.
- Table 1. Brockville laboratory exercises, p. 58.
- Table 2. Brockville laboratory exercises, p. 58.
- Table 3. Brockville laboratory exercises, p. 59.
- Table 4. Brockville laboratory exercises, p. 59.

BIBLIOGRAPHY

ASTM. Annual Book of ASTM Standards 1985. Volume 04.08: Soil and Rock; Building Stones. Philadelphia, Pennsylvania: ASTM, 1985.

Clifton, James R. - Brown, Paul W. - Robbins, Carl R. Methods for characterizing adobe building materials. NBS Technical Note N°977. Washington, D.C.: U.S. Department of Commerce, 1978.

Head, K.H. Manual of soil laboratory testing. Volume 1: soil classification and compaction tests. London: Pentech Press, 1980.

St. Lawrence College. Laboratory exercises. St. Lawrence College, Brockville, Ontario, Canada.

## DATA SHEET: GRAIN SIZE ANALYSIS-HYDROMETER METHOD

Project \_\_\_\_\_ Job No. \_\_\_\_\_

Location of Project \_\_\_\_\_ Boring No. \_\_\_\_\_ Sample No. \_\_\_\_\_

Description of Soil \_\_\_\_\_ Depth of Sample \_\_\_\_\_

Tested By \_\_\_\_\_ Date of Testing \_\_\_\_\_

### Hydrometer analysis

Hydrometer no. \_\_\_\_\_  $G_s$  of solids = \_\_\_\_\_  $u =$  \_\_\_\_\_Dispersing agent \_\_\_\_\_ Amount \_\_\_\_\_ Wt. of soil,  $W_s$  \_\_\_\_\_

Zero correction (x) \_\_\_\_\_ Meniscus correction \_\_\_\_\_

[illegible]

$$R_c = R_{\text{actual}} - \text{zero correction} + C_T$$

$$\% \text{ finer} = R_r(a)/W_s$$

$$D = K\sqrt{L/t}$$

Ex. 18B (continued)

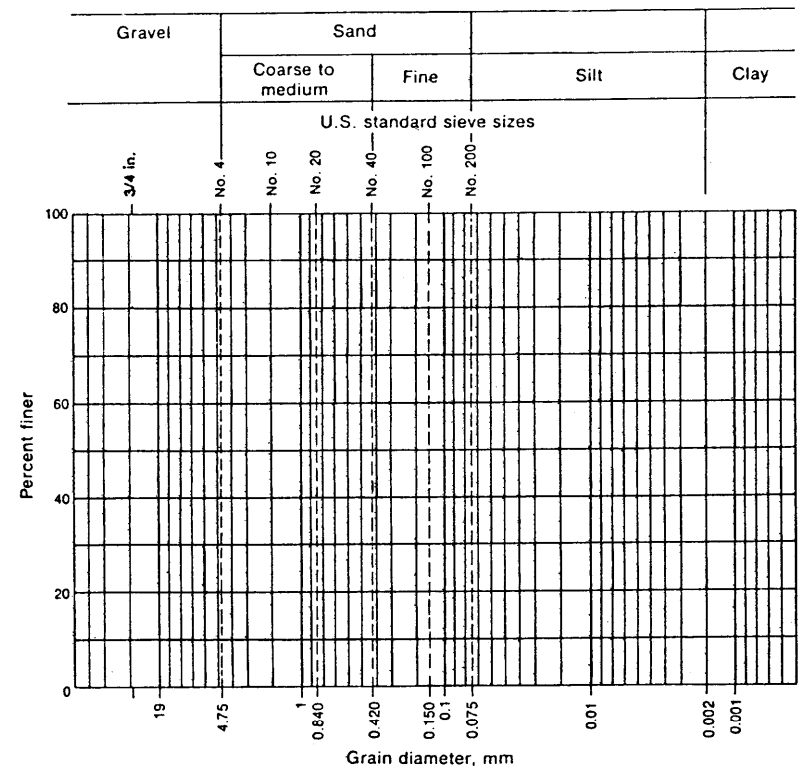
## DATA SHEET: GRAIN SIZE DISTRIBUTION

Project \_\_\_\_\_ Job No. \_\_\_\_\_

Location of Project \_\_\_\_\_ Boring No. \_\_\_\_\_ Sample No. \_\_\_\_\_

Description of Soil \_\_\_\_\_ Depth of Sample \_\_\_\_\_

Tested By \_\_\_\_\_ Date of testing \_\_\_\_\_



Visual soil description \_\_\_\_\_

**Soil classification:**

System \_\_\_\_\_

## EX. 19 PLASTIC LIMIT OF SOILS

REFERENCES: ASTM D4318-84; BS1377: 1975, Test

### 3 INTRODUCTION

The behavior of soil is related to the amount of water in its structure. At high water contents, soils are suspensions with the flow properties of liquids. As the water content decreases, the soil becomes consecutively pastelike, sticky, then plastic; at a low water content, the soil has the properties of a solid. The physical state of a soil at a given water content is termed *consistency*, which is a measure of the resistance of a soil to flow.

In 1911, the Swedish scientist Dr. A. Atterberg defined the boundaries in a soil's condition as "limits". The liquid limit, plastic limit, and shrinkage limit are the standard tests for determining these boundaries. The following exercise outlines the procedures for the plastic limit test.

(The plastic limit test can be performed on cohesive and semi-cohesive soils such as organic and inorganic clays, tuff or fine grain materials, adobe, etc. It is not possible to make a plastic limit test on sand or gravel, peat or similar materials.)

### DEFINITIONS

**Plastic Limit:** The plastic limit of a soil is the water content, expressed as a percentage of the mass of the oven-dried soil, at the boundary between the plastic and semi-solid states. The water content at this boundary is arbitrarily defined as the lowest water content at which the soil can be rolled into threads 3 mm (1/8") in diameter without the threads' breaking into pieces.

### EQUIPMENT

- The most important piece of apparatus is the HAND of the operator which should be clean and free from grease
- Spatula (blade about 75 mm x 20 mm)
- Surface for rolling: ground glass plate or non-absorbent paper
- Soil sample containers
- Evaporating dish
- Balance sensitive to 0.01 g
- A short length (about 100 mm) of 3 mm diameter metal rod
- Distilled water.

## Ex. 19 (continued) PROCEDURE

1. Preparation of the sample: Take about 15-20 grams of the soil sample that has passed the N°40 (425 µm) sieve, obtained according to the Sample Preparation, Method in Appendix A.
2. Rolling into a ball: Place the air-dried soil in an evaporation dish and thoroughly mix with distilled water until the mass becomes plastic enough to be easily shaped into a ball (without sticking to the fingers excessively when squeezed). Take a portion of this ball weighing about 8 g (or about half of the sample) for the test sample.
3. Rolling into threads: Squeeze and form the 8 g test sample into a round ellipsoidal-shaped mass.

Roll the sample between the fingers of one hand and the ground glass plate (or piece of non-absorbent paper) which should be lying on a smooth horizontal surface. Use sufficient pressure to roll the mass into a thread of uniform diameter across its length. Move your hand (with fingers outstretched) across the sample to develop this uniformity in diameter.

The rate of rolling should be between 80 and 90 strokes per minute. A stroke is counted as one complete motion of the hand forward and back to starting position again.

The pressure should reduce the diameter of the thread to about 3 mm after between 5 and 10 strokes. It is important to maintain a uniform rolling pressure as the thread approaches 3 mm.

When the diameter of the thread becomes 3 mm (use the metal rod as a guide), break the thread into 6 or 8 pieces. Squeeze these pieces together between the thumbs and fingers of both hands into a rough ball and repeat the entire process again. You will note that water from the sample is evaporating into the air; the sample is thus becoming progressively drier.

Continue this alternate rolling to a 3 mm (1/8") diameter thread, gathering together, kneading and rerolling, until the thread crumbles under the pressure required for rolling and the soil can no longer be rolled into a thread. The first crumbling point is the plastic limit.

The crumbling may occur when the thread has a diameter greater than 3<sup>R</sup> mm. This should be considered a satisfactory end point, provided the soil has been previously rolled into a 3 mm diameter thread. (If the soil thread breaks before it has initially been rolled down to the 3 mm

Ex. 19 (continued)

diameter, the moisture content is less than that for the plastic limit and more water should be added to the sample.) Crumbling will manifest itself differently in different types of soil.

Some soils will fall apart in very small particles.

Other soils form an outside tubular layer that starts splitting at both ends. The splitting progresses toward the middle and finally the thread falls apart in many small plate-like particles.

Heavy clay soils will require considerable pressure to deform the threads, particularly as they approach the plastic limit. This type of soil usually breaks into a series of barrel-shaped segments each about 6 mm (1/4") to 9 mm (3/8") in length.

4. Moisture Content Measurement: When the plastic limit has been reached, place the crumbled soil samples in a numbered and weighed moisture content container ( $M_1$ ). Cover the container immediately to avoid change in weight of the sample by evaporation.

Weigh the container and the soil and record the combined mass ( $M_2$ ) on the attached data sheet. Place the container, with the cover removed, in an oven (the vents of the oven must be open to allow the moisture to escape to the outside air).

Dry the soil to constant mass at  $110^\circ\text{C} \pm 5^\circ\text{C}$ . Normally, a drying period of 24 hours is satisfactory.

Remove the sample from the oven, replace the cover on the container and allow it to cool at room temperature. Weigh the container and oven-dried sample on the same balance previously used. Record the mass ( $M_3$ ) on the data sheet. Record the loss in mass as the mass of water ( $M_2 - M_3$ ).

5. Calculations: Calculate the plastic limit, expressed as the water content in percentage of the mass of the oven-dry soil, as follows:

$$\text{Plastic limit} = \frac{\text{mass of water}}{\text{mass of oven-dry soil}} \times 100$$

Record the plastic limit to the nearest whole number on the data sheet.

6. Repeat Tests: It is usually desirable to obtain three determinations which can be averaged to give the plastic limit. Thus, repeat steps 2 through 5 for three other samples of the subject soil. The standard deviations should be within 10 percent of the average values.

Ex. 19 (continued)

BIBLIOGRAPHY

ASTM. Annual Book of ASTM Standards 1985. Volume 04.08: Soil and Rock; Building Stones. Philadelphia, Pennsylvania: ASTM, 1985.

Clifton, James R. - Brown, Paul W. - Robbins, Carl R. Methods for characterizing adobe building materials. NBS Technical Note N°977. Washington, D.C.: U.S. Department of Commerce, 1978.

Head, K.H. Manual of soil laboratory testing. Volume 1: soil classification and compaction tests. London: Pentech Press, 1980.

Soiltest, Inc. Test for plastic limit and plasticity index of soils (slide/tape set). Chicago, Illinois: Soiltest, Inc., 1963.

Ex. 19 (continued)

DATA SHEET: PLASTIC LIMIT OF SOILS

Name \_\_\_\_\_ Sample Number \_\_\_\_\_  
Date \_\_\_\_\_ Sample Type \_\_\_\_\_

Test Number					Mean
Container Number					
Wt. Wet Soil + Cont. ( $M_2$ )					
Wt. Dry Soil + Cont. ( $M_3$ )					
Water Loss ( $M_2 - M_3$ )					
Wt. of Container ( $M_1$ )					
Wt. Dry Soil ( $M_3 - M_1$ )					
PLASTIC LIMIT $\frac{(M_2 - M_3)}{(M_3 - M_1)} \times 100$					

Mean/ Plastic Limit \_\_\_\_\_

Standard Deviation \_\_\_\_\_

Ex. 19 (continued)

APPENDIX A: PREPARATION OF SAMPLE FOR LIQUID/PLASTIC LIMIT TESTS

1. Expose the soil sample from the field to the air at room temperature until it is thoroughly dry. (Samples should not be oven-dried prior to testing. Even controlled temperatures can alter the soil in various ways.)
2. If necessary, break up the aggregations of soil in a mortar with a rubber-covered pestle.
3. Sieve the pestled soil by hand through a 425 pm sieve nested on a receiving pan. About 250 grams of sieved dry soil is necessary for the two tests.

\* If the soil is predominantly clay, it is preferable to take the soil in its natural state (without drying) and to remove coarse particles by hand during the mixing process. organic soils, and most tropical soils, should always be tested in their natural condition.

Natural soil can be cut up into smali pieces with a cheese grater.

Ex. 20 LIQUID LIMIT OF SOILS

REFERENCES: ASTM D4318-84; BS1377: 1975, Test 2

DEFINITIONS

**Liquid Limit:** The water content, expressed as a percentage of the oven-dried soil, at the boundary between the liquid and plastic states. The water content at this boundary is arbitrarily defined as the water content at which two halves of a soil cake placed in a Casagrande device flow together for a distance of 1/2 inch (12.7 mm) along the bottom of the groove separating the two halves, when the cup is dropped 25 times for a distance of 1 cm (0.3937 inch) at the rate of 2 drops/second.

EQUIPMENT

- Porcelain evaporating dish (about 115 mm in diameter)
- Mixing spatulas (blade about 100 mm x 30 mm)
- Casagrande device and grooving tool (see below)
- Aluminum soil sample containers
- Balance sensitive to 0.01 gram
- Stopwatch
- Distilled water.

PROCEDURE:

1. Adjustment of the Apparatus: The Casagrande Device (named after A. Casagrande who designed it in 1932) must be clean and the cup must be dry and oil-free. Check that the cup moves freely but without too much side-play. Make sure the screws connecting the hanger arm are tight and that a groove has not been worn into the bowl through long usage.

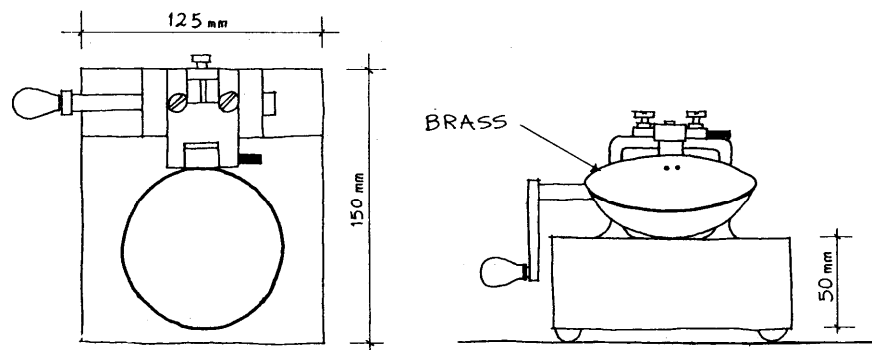


Fig. 1 CASAGRANDE DEVICE

Ex. 20 (continued)

Check the grooving tool to be sure that it conforms to the dimensions shown in Fig. 2 below.

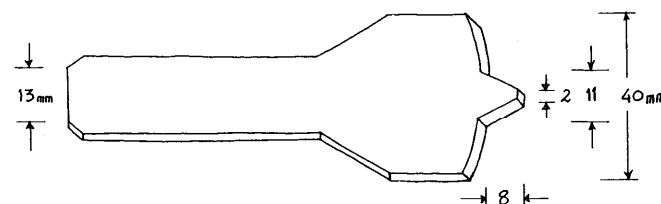


Fig. 2

Place the machine on a firm solid base so that it will not wobble. Using the rectangular element on the handle of the gauging tool and the adjustment plate, adjust the height to which the cup is lifted so that it is exactly 1 cm above the base (Fig. 3). Check the adjustment by revolving the crank rapidly several times. If the adjustment is correct, a slight ringing sound will be heard when the cam strikes the cam follower.

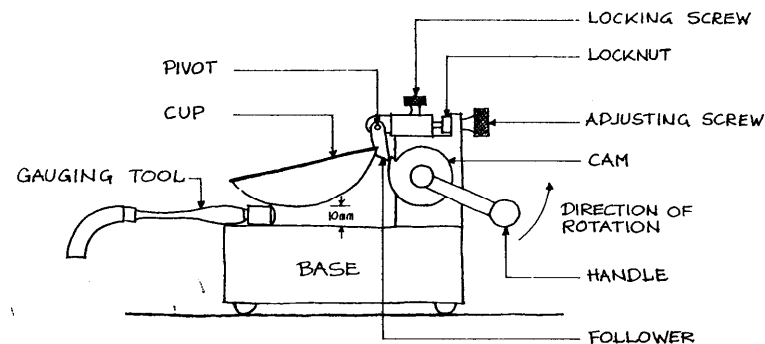


Fig. 3

Ex. 20 (continued)

2. Sample: Take about 100 grams of the sample material that has passed the N<sup>o</sup>40 (425  $\mu$ m) sieve, obtained according to the Sample Preparation Method in Appendix A.
3. Mixing: In the evaporating dish, thoroughly mix the soil sample with 15-20 mL of distilled water by alternately stirring, kneading, and chopping with a spatula. Add more water, in increments of 1-3 mL, until the sample is a thick homogeneous paste.
4. Placing: Make sure the cup of the Casagrande device rests on the base. Place a portion of the mixed sample in the cup. Using the spatula, press it from the middle outwards into the position shown in Fig. 4 to prevent trapping any air bubbles in the mass. Use as few strokes of the spatula as possible. Level the surface of the soil paste (again using the spatula) and trim it to a depth of 1 cm at the point of maximum thickness. Return excess soil to the evaporating dish.

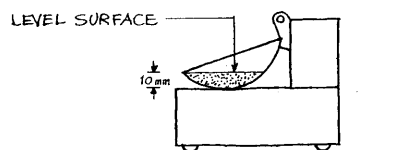


Fig. 4

5. Cutting Groove: Divide the soil in the cup into two equal halves by firm strokes of the grooving tool along the centerline of the cam follower. Starting near the hinge, draw the grooving tool toward the front in a continuous circular motion, always keeping the tool normal to the surface of the cup (Fig. 5).

Each stroke should penetrate a little deeper until the last stroke gently scrapes the bottom of the cup clean. Make the groove with as few strokes as possible (no more than 6). The completed groove is shown in Fig. 6.

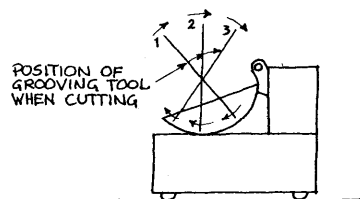


Fig. 5

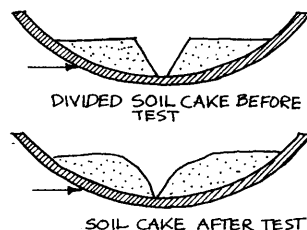


Fig. 6

Ex. 20 (continued)

6. Dropping the Cup: Lift and drop the cup by turning the crank handle at a rate of two revolutions per second (practice with a stopwatch to obtain the correct speed). Continue turning until the groove is closed (i.e. until the two halves of the soil sample come into contact at the bottom of the groove) along a distance of 13 mm. The back end of the standard grooving tool serves as a gauge length. Record the number of drops (1) required to reach this condition. If this exceeds 50, mix in a little more water and repeat steps 4 to 6.

Note: The groove should close because of the plastic flow of the soil, not as a result of sliding on the surface of the cup. If sliding occurs, this should be recorded and the result discarded.

7. Moisture Content Measurement: Remove a slice of soil approximately the width of the spatula, extending from edge to edge of the soil cake at a right angle to the groove and including that zone of the groove where the two portions flowed together. Place in a suitable tared container ( $M_1$ ). Weigh and record the combined mass ( $M_2$ ) on the attached data sheet. Oven dry the soil to a constant mass at  $110 \pm 5^\circ\text{C}$  (overnight drying is usually sufficient); reweigh as soon as it has cooled. Record this mass ( $M_3$ ) on the data sheet. Record the loss in mass due to drying as the mass of water ( $M_2 - M_3$ ). Refer to Fig. 7: Sample Data Sheet.
8. Repeat Tests: Transfer the soil still in the cup to the remaining soil paste in the evaporating dish. Wash and dry the cup, grooving tool, and spatula. Reattach and adjust the cup for the next trial.

Repeat steps 4 to 7 at least three more times with the soil collected in the evaporating dish, adding a little more water each time. The object of this procedure is to obtain samples of such consistency that the number of drops required to close the groove will be roughly evenly spaced over the range from 50 to 10. Ideally, there should be at least two results on either side of 25. The test should always proceed from the drier (50 drops) to the wetter (10 drops) condition.

9. Calculations: Calculate the water content of the soil ( $W_n$ ) for each, drop count, expressed as a percentage of the weight of the oven-dried soil:

$$W_n = \frac{\text{mass of water}}{\text{mass of oven-dried soil}} \times 100 = \frac{(M_2 - M_3)}{(M_3 - M_1)} \times 100$$

Ex. 20 (continued)

10. Flow Curve: On a semi-logarithmic chart, plot the moisture content as ordinates (linear scale) against the corresponding number of drops as abscissae (logarithmic scale). The "flow curve" is the best straight line that can be drawn through the plotted points (see Fig. 7).
11. Liquid Limit: The liquid limit is read as the moisture content corresponding to the intersection of the flow curve with the 25-drop ordinate.
12. Plasticity Index\*: The difference between the liquid limit and the plastic limit (see Ex. 19) is calculated to give the plasticity index of the soil:

Plasticity index = Liquid limit - Plastic limit

The plasticity index is also reported to the nearest whole number.

When the liquid limit or plastic limit cannot be determined, report the plasticity index as NP (nonplastic).

When the plastic limit is equal to, or greater than, the liquid limit (e.g. in soils with a high mica content), report the plasticity index as NP.

\* The plasticity index depends largely on the amount of clay present. The strength of the soil increases as the plasticity index increases. However, the tendency of a clay to expand when wet and shrink when dried also increases as the plasticity index increases.

FIGURE CREDITS (see Bibliography for complete references)

1. ASTM, p. 126 (1979 edition).
2. Head, K.H., p. 78.
3. Head, K.H., p. 77.
4. Head, K.H., p. 79.
5. Head, K.H., p. 79.
6. ASTM, p. 126 (1979 edition).
7. Head, K.H., p. 82.

FOOTNOTES

1. ASTM uses the word "drop" to indicate one falling motion of the Casagrande cup. Other standards use the word "blow" or "bump".

Ex. 20 (continued)

BIBLIOGRAPHY

ASTM. Annual Book of ASTM Standards 1985. Volume 04.08: Soil and Rock; Building Stones. Philadelphia, Pennsylvania: ASTM, 1985.

Clifton, James R. - Brown, Paul W. - Robbins, Carl R. Methods for characterizing adobe building materials. NBS Technical Note N°977. Washington, D.C.: U.S. Department of Commerce, 1978.

Head, K.H. Manual of soil laboratory testing. Volume 1: soil classification and compaction tests. London: Pentech Press, 1980.

Soiltest, Inc. Test for plastic limit and plasticity index of soils (slide/tape set). Chicago, Illinois: Soiltest, Inc., 1963.

Ex. 20 (continued)

APPENDIX A: PREPARATION OF SAMPLE FOR LIQUID/PLASTIC LIMIT TESTS

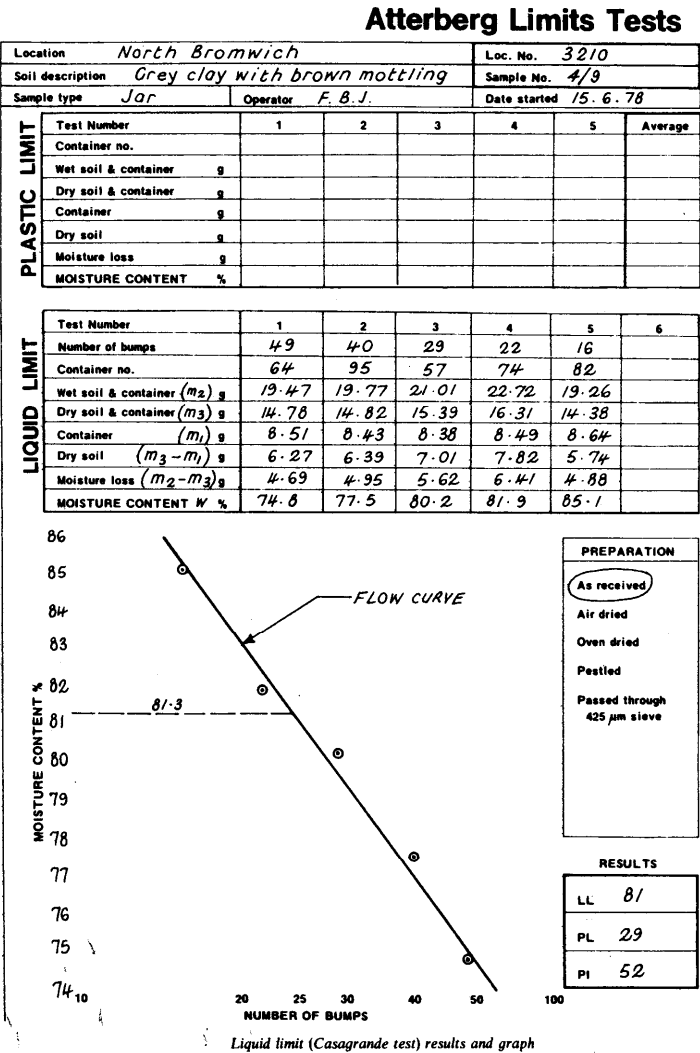
1. Expose the soil sample from the field to the air at room temperature until dried thoroughly. (Samples should not be oven dried prior to testing. Even controlled temperature can alter the soil in various ways.)
2. If necessary, break up the aggregations of soil in a mortar with a rubber-covered pestle.
3. Sieve the pestled soil by hand through a 425 µm sieve nested on a receiving pan. About 250 grams of sieved dry soil is necessary for the two tests.

\* If the soil is predominantly clay, it is preferable to take the soil in its natural state (without drying) and to remove coarse particles by hand during the mixing process. Organic soils, and most tropical soils, should always be tested in their natural condition.

Natural soil can be cut up into small pieces with a cheese grater.

Ex. 20 (continued)

Fig. 7: SAMPLE DATA SHEET



Ex. 20 (continued)

DATA SHEET: LIQUID LIMIT OF SOILS

Name \_\_\_\_\_ Sample Number \_\_\_\_\_  
Date \_\_\_\_\_ Sample Type \_\_\_\_\_

Test Number						
Container Number						
Number of drops						
Wt. Wet Soil + Cont. ( $M_2$ )						
Wt. Dry Soil + Cont. ( $M_3$ )						
Water Loss ( $M_2 - M_3$ )						
Wt. Container ( $M_1$ )						
Wt. Dry Soil ( $M_3 - M_1$ )						
Moisture Content % $\frac{(M_2 - M_3)}{(M_3 - M_1)} \times 100$						

STONE. BRICK AND MORTARS

Moisture  
Content %

45  
44  
43  
42  
41  
40  
39  
38  
37  
36  
35  
34  
33

10 20 25 30 40 50 100  
Number of drops

RESULTS
Liquid Limit (LL) _____
Plastic Limit (PL) _____
Plasticity Index (LL - PL) _____

## Ex. 21 MORTAR ANALYSIS: SIMPLE METHOD

### INTRODUCTION/AIM

The following test is a simple method for determining the proportions of the three principal components of an historic mortar: (1) the binder (basically calcium carbonate ( $\text{CaCO}_3$ ), soluble in acid); (2) the fines (finely-textured. impurities such as clays); and (3) the sand or aggregate. The analysis gives approximate information and should be carried out together with other analyses (i.e. mineralogical, microscopic, etc.) in order to derive the maximum possible information from the mortar sample, either for the purposes of historical research or to prepare a restoration mortar compatible with the original.

As regards the latter, it should be remembered that several important factors that affect the condition and performance of a mortar are not revealed in mortar analysis. These include the original water: binder ratio, the mixing and placing method, the rate of drying, and the cleanliness and condition of the aggregates. In some ways, the most useful aspect of mortar analysis is the identification of aggregates for matching.

### EQUIPMENT/CHEMICALS

Oven, balance, mortar and pestle, beakers, funnel, wash bottle, filter paper, glass rods, heat lamp, sieve set, 14% solution of hydrochloric acid, deionized water.

### PROCEDURE

#### A. Collection/ Examination/ Dissolution of Binder

1. Collect a sample of adequate size (about 40-50 grams).
2. Examine the sample and record the following characteristics: color, texture, inclusions, hardness.
3. Powder half of the sample (20-25 grams) with a mortar and pestle; leave the other half for further analysis.
4. Dry the powdered sample in the oven at  $110^\circ\text{C}$  for 24 hours and then weigh it ( $W_1$ ) with a balance (0.01 g precision). Record the weight on the attached data sheet.
5. Place the sample in a 600 mL beaker and moisten it with water,
6. Immerse the moistened sample in a 14% solution of hydrochloric acid in order to dissolve the binder.

Observe the reaction and record observations.

Ex. 21 (continued)

B. Separation/ Filtration/ Sieving

1. Label the filter paper to be used, weigh it ( $W_2$ ), and record the weight on the data sheet.
2. Fold the paper into quarters and place it in a funnel. Position the funnel so that it will drain into a large flask.
3. Add a few drops of hydrochloric acid (HCl) to the sample to verify complete acid digestion of the binder (i.e. that the reaction initiated in Step 6 above is complete).
4. Slowly add water to the remaining sample material.
5. Swirl with a glass rod to suspend the fines.
6. Slowly pour the liquid with suspended material through the filter, being careful to keep solid particles (sand) at the bottom of the beaker.
7. Repeat process (steps 4-6) until the water added to the beaker remains clear.
8. Dry the fines collected on the filter paper with a heat lamp.
9. Wash the sand with water several times and leave it to dry for 24 hours.
10. Weigh the filter paper with the dry fines ( $W_3$ ). Subtract the weight of the paper ( $W_2$ ) to determine the weight of the fines ( $W_3 - W_2$ ). Record both values on the data sheet.
11. Weigh the dry sand ( $W_4$ ) and record the weight on the data sheet.
12. Express the amount of sand as a w/w percentage of the whole sample. Express the amount of fines in the same manner. The amount of dissolved binder is calculated by summing up the percentages of sand and fines and subtracting from 100%.\*

C. Characterization of Sand

1. Examine the sand with a binocular microscope. Record characteristics: color, particle shape and size, etc.

Ex. 21 (continued)

2. Sieve the sand in a standard sieve set to determine the particle size distribution. Express the amount of each particle size as a percentage of the whole (see Ex. 18A).

\* The limitations of mortar analysis should always be considered. For example, calcareous aggregates will be dissolved with calcareous binder material and present misleading binder :aggregate proportions. Similarly, clay minerals present as impurities may not be readily distinguishable from the silicates present in hydraulic cement (see Ashurst, p. 20).

BIBLIOGRAPHY

Ashurst, John. Mortars, plasters and renders in conservation. London: Ecclesiastical Architects' and Surveyors' Association, 1983.

Columbia University, Historic Preservation Program. Laboratory exercises.

National Park Service, North Atlantic Preservation Center. Mortar analysis methodology.

MORTAR ANALYSIS: DATA SHEET

Name \_\_\_\_\_ Sample N° \_\_\_\_\_

Date \_\_\_\_\_ Origin of sample \_\_\_\_\_

Visual description of sample (color, texture, hardness, inclusions, etc.): \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_Mortar Analysis:Original weight of powdered sample ( $W_1$ ) = \_\_\_\_\_Weight of filter paper ( $W_2$ ) = \_\_\_\_\_Weight of filter paper + dry fines ( $W_3$ ) = \_\_\_\_\_Weight of dry fines ( $W_3 - W_2$ ) = \_\_\_\_\_Weight of dry sand ( $W_4$ ) = \_\_\_\_\_% of sand ( $(W_4/W_1) \times 100$ ) = \_\_\_\_\_% of fines ( $(W_3 - W_2)/W_1 \times 100$ ) = \_\_\_\_\_

% of dissolved binder = \_\_\_\_\_

Observations: dissolution of binder, color of liquid: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_Characterization of Sand:

Microscopic Examination	% Finer than	
_____	4.75 mm	_____
_____	2.36 mm	_____
_____	1.18 mm	_____
_____	600 $\mu$ m	_____
_____	300 $\mu$ m	_____
_____	150 $\mu$ m	_____
_____	75 $\mu$ m	_____
_____	53 $\mu$ m	_____
_____	38 $\mu$ m	_____

## Ex. 22 ANALYSIS OF CALCIUM CARBONATE CONTENT IN MORTARS: CALCIMETER METHOD

AIM

Somewhat different from the previous test, this method defines the 3 principal components of a mortar as *carbonates*, *solubles* (those substances soluble in acid without producing carbon dioxide) and *sand*. The proportions of each are determined with the use of a special instrument called a calcimeter.

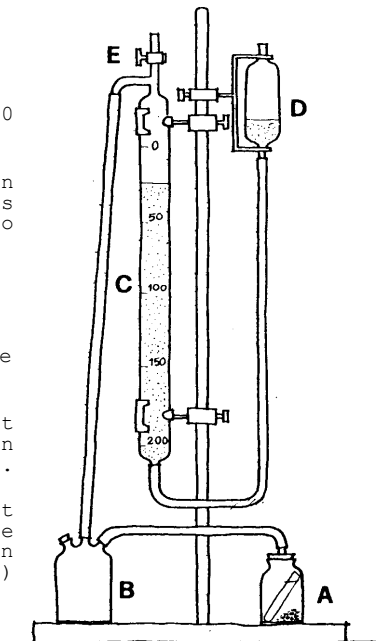
In actual practice at ICCROM, the test has proved less reliable and consistent than simple mortar analysis. This may be due to problems with the instrument or with the formula and constant used for calculations. Nevertheless, the procedure has been included to illustrate alternative methodologies and to promote further experimentation with the technique.

EQUIPMENT/CHEMICALS

Oven, dessicator, calcimeter, balance (0.01 g precision), aluminum cups, filter paper, funnel, flask, plastic gloves, 17% solution of hydrochloric acid, silica gel, distilled water.

PROCEDURE

1. Take a well-preserved and representative sample.
2. Powder part of the sample, about 20 g.
3. Dry the powdered sample in the oven for 48 hours at 60°C. After this period, put it in the dessicator to cool and then weigh it (W).
4. Measure the room pressure and temperature.
5. Close "E" (see figure); record the volume in "C".
6. Pour the sample in "A"; carefully put the hydrochloric acid solution in the test tube (use plastic gloves).
7. Put the test, tube in "A" without dripping any HCl on the sample. Close "A", then shake it up and down in order to dissolve the binder ( $\text{CaCO}_3$ ) with the HCl.



Calcimeter

Ex. 22 (continued)

8. The carbon dioxide (CO<sub>2</sub>) of the reaction will pass through "B" into "C".
9. When the reaction is complete, record the new volume in "C", i.e. the height reached by the water on the cc scale.
10. Carefully remove the small test tube from "A". Stir the remaining solution with a glass rod and then carefully transfer the solution together with light suspensions and precipitates to a dry beaker. The sand should remain at the bottom of vessel "A".
11. Wash the sand with water several times (the water is discarded) and leave it to dry for 24 hours.
12. Weigh the dry sand.
13. Calculate the % of CaCO<sub>3</sub> with the following formula:

$$\% \text{ CaCO}_3 = \frac{P+VKc}{760} \times \frac{273.16}{273.16+t} \times \frac{V(cc)}{1000} \times \frac{100.29}{22.414} \times \frac{100}{\text{matg}}$$

where:

P = room pressure  
V = volume change in "C"  
t = room temperature  
Kc = constant of instrument (0.221)\*\*  
matg = grams of material in sample

\*\* This constant must be calculated for each instrument. A control test using pure CaCO<sub>3</sub> (in which the formula can be set equal to 100%) should permit the necessary adjustment of the formula.

14. The amount of sand is expressed as a simple w/w percentage of the whole sample. The "solubles" are calculated by summing up the percentage of carbonates and sands, and subtracting them from 100 percent.

BIBLIOGRAPHY

Jedrzejewska, Hanna. Old mortars in Poland: a new method of investigation. In: Studies in Conservation, Vol. 5, N°4, 1960, pp. 132-138.

Ex. 23 MIXING MORTARS FOR CONSERVATION

AIM

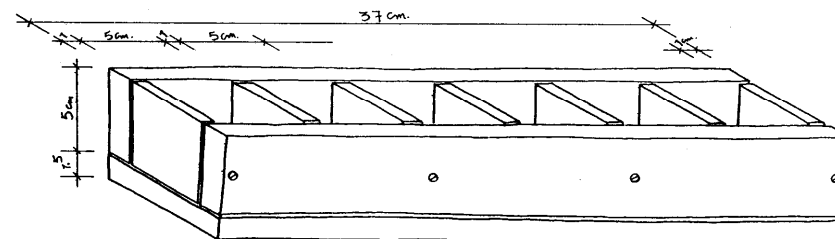
This exercise indicates the potential range of mixes to be used in the conservation of ancient masonry. Factors such as consistency, workability and shrinkage of mortars will be examined.

EQUIPMENT

Small vessels and tools for mixing mortars, paper cups for measuring volume, wooden molds, adhesive labels.

MATERIALS

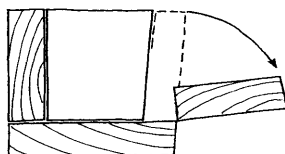
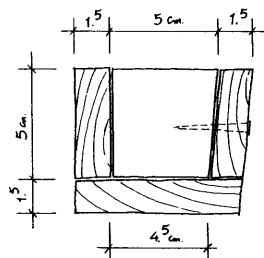
Vaseline, lime putty, hydraulic lime, hydrated lime, white cement, portland cement, sand, pozzolana, brick dust, organic additives (eggs, urea, sugar, horse dung, yoghurt, powdered skim milk), water.



1. Ensure that the wooden mold is clean. Grease it with vaseline to prevent adhesion of the sample mortars.
2. Examine the list of mortars on page 121. Choose one sample mortar from each of the six categories (A-F).
3. For each sample mortar chosen, mix the components, adding only the amount of water necessary to achieve a homogeneous paste and good workability. Do not add water to mixes made with lime putty. Record your observations regarding work-ability in the data sheet.
4. Pour the mix into one of the greased mold compartments and label it, indicating your name and the type of mortar.

Ex. 23 (continued)

5. Wash out the mixing vessel and proceed to the next mortar sample.
6. When the six sample mortars have been prepared, leave the mold to set at 20°C and about 50% relative humidity for a period of 48 to 72 hours.
7. After this initial setting period, evaluate and compare the mortar samples noting characteristics such as shrinkage, setting time, degree of carbonation, hardness (abrasion resistance) and cohesion. Record your observations in the data sheet.



8. Open the wooden molds. If possible, remove the samples and allow them to set for another period of 5 to 7 days Re-evaluate and record your observations.

BIBLIOGRAPHY

Ashurst, John. Mortars for the repair of historic masonry surfaces. London: Department of the Environment.

Ex. 23 (continued)

SAMPLE MORTARS (parts by volume)

Non-Hydraulic Mortars

<b>A. Lime Putty (LP):</b>		<b>Aggregate</b>	
1		1	
1		2	
1		3	
1		6	
<b>B. Hydrated Lime (HL):</b>		<b>Aggregate</b>	
1		1	
1		2	
1		3	
1		6	
<b>C. Hydrated Lime (HL):</b>		<b>Aggregate</b>	<b>: Organic Additive</b>
1		3	horse dung
1		3	sugared water
1		3	skim milk + water
1		3	egg + water
1		3	yoghurt
1		3	urine

Artificial Hydraulic Mortars

<b>D. Lime Putty (LP) :</b>		<b>Aggregate</b>	<b>: Additive</b>
1		3	1/2 pozzolana
1		3	1/2 brick dust
1		3	1/2 white cement
1		3	1/2 portland cement

Hydraulic Mortars

<b>E. Hydraulic Lime (HCL):</b>		<b>Aggregate</b>	
1		1	
1		2	
1		3	
1		6	
<b>F. White Cement (WHC):</b>		<b>Aggregate</b>	
1		3	
1		6	
<b>Portland Cement (PC):</b>		<b>Aggregate</b>	<b>: Additive</b>
1		3	
1		6	
1		3	soap
1		6	soap

Ex. 23 (continued)

SAMPLE COMPOSITION	A	B	C
WORKABILITY			
SETTING TIME			
SHRINKAGE			
CARBONATION			
HARDNESS (Abrasion Resistance)			
COHESION			
OTHER (Color, Texture, etc.)			

SAMPLE COMPOSITION	D	E	F
WORKABILITY			
SETTING TIME			
SHRINKAGE			
CARBONATION			
HARDNESS (Abrasion Resistance)			
COHESION			
OTHER (Color, Texture, etc.)			

Ex. 24 CLEANING BUILDING STONE

AIM

This exercise will introduce various materials and methods for cleaning building stone. It aims to provide a general overview of the options available when it has been determined that cleaning is advisable.

EQUIPMENT/MATERIALS

Paper pulp, attapulgate, "AB 57", plastic wrap, air-abrasive equipment, water nebulizer.

PROCEDURE

After reading the following descriptions of the various cleaning methods, practice applying them to the stone objects and fragments given to you. Compare ease of use, cleaning results, and collateral effects of each treatment on the stone.

1. ATTAPULGITE: A hydrated aluminum-magnesium silicate, having the typical structure of a clay mineral; the chief ingredient of fuller's earth.

Cleaning principle: Because of its crystalline structure, this clay has a high surface area and absorption potential. Salt solutions can thus be absorbed by the clay and extracted from the stone.

Use: Mix clay and distilled water (1:1) into a paste. Apply packs of wet clay to the stone and cover with plastic film. After a few hours, remove the plastic wrap to permit evaporation of water from the clay. (It is possible to add some paper pulp to the clay to prevent cracking of the compress during drying.) When the pack has dried, carefully remove it from the subject area. Repeat if necessary.

Recommended: (a) To extract soluble salts. (b) To remove stains caused by organic materials such as oils. In the latter cases, it is necessary to use an organic solvent (carbon tetrachloride, dichloromethane, white spirit, etc.) instead of water.

Inadvisable: When the stone surface is in bad condition. Application of the clay compresses could lead to detachment of loose fragments.

2. "AB 57": A solution of slightly basic salts with chelating agents added to surfactants, fungicides and thixotropic substances to form a paste (developed by P. and L. Mora; see Bibliography). The ratio of the water to thixotropic agents always remains the same, while the quantity of the other components can be varied according to necessity. A typical mixture is obtained by mixing the ingredients in the following proportions:

Water	1000 mL
Ammonium bicarbonate	30 g
Sodium bicarbonate	50 g
Disodium salt of EDTA	25 g
Desogen (10% solution)	10 mL
Carboxymethyl cellulose	60 g

Cleaning principle: The ammonium and sodium bicarbonates and the EDTA (ethylene diamine tetra acetic acid, a chelating agent) facilitate the dissolution of calcium salts (especially gypsum); Desogen (Ciba-Geigy) is a surfactant (reduces surface tension) with a disinfecting action. The carboxymethyl cellulose gives the paste the needed bulk (to prevent flow on vertical surfaces) and prevents the solution from penetrating too deeply into the stone.\*\*

Use: Make a solution of the bicarbonates and the EDTA in water. Add the Desogen and then, slowly, the cellulose. Mix constantly to form a homogeneous paste.

Apply packs of the paste, covered with plastic wrap, to the surface to be cleaned. Leave in place for 1 to 12 hours (depending on the condition of the stone), then remove. Removal should be accomplished by delicate scraping (using scalpels) of the subject area followed by careful washing with water to eliminate sodium and ammonium salts. As a final phase, damp, absorbent compresses can be applied to completely eliminate the salt residues.

Recommended: When the stone surface is covered with a layer of soot rich in gypsum and other salts.

Inadvisable: When the stone surface is in bad condition and fragments might be detached.

\*\* It is possible to use micronized silica instead of carboxymethyl cellulose, thus creating a paste that can be more easily removed at the end of the treatment. It should be noted, however, that the adhesive strength of micronized silica is lower than that of carboxymethyl cellulose, so its cleaning action on surface crusts is slower and often less effective.

3. NEBULOUS SPRAYS or NEBULIZED WATER: a very fine water spray, or mist.

Cleaning principle: The solvent action of water (with respect to water-soluble materials) is enhanced by the high surface area of the droplets. Cleaning is thus accomplished without mechanical action.

Use: Using a nozzle which creates a fine mist, spray water in such a way that it wets the subject stone surfaces but is not aimed directly at them.

Recommended: To remove dust and relatively soft layers of soot, rich in water-soluble materials.

Inadvisable: When the stone is so heavily damaged by the presence of water-soluble salts that the hydration process (triggered by the penetration of water into the stone) could produce spalling and cracking.

4. AIR ABRASIVE CLEANING: Micro-sandblasting using glass microspheres (diameter 0.001 mm) or a fine powder of aluminum oxide.

Cleaning principle: Cleaning is accomplished by the mechanical action of a compressed air jet containing an abrasive. Because of the small size of the spray and the low hardness of the powders used, the cleaning action is slow and fairly easy to control.

Use: Turn the air-abrasive equipment to the lowest pressure setting, aim the pencil at the subject surface and begin cleaning. Adjust the pressure of flow and the distance between the pencil and surface based on the response of the material to the abrasion.

Recommended: To clean rather thick crusts from sound stone or to clean consolidated stone.

Inadvisable: When the Stone surface is in bad condition or the operator unskilled in the use of the air-abrasive apparatus.

Ex. 24 (continued)

BIBLIOGRAPHY

Ashurst, J. Cleaning stone and brick. (Technical Pamphlet N°4). London: Society for the Protection of Ancient Buildings, 1977.

ICCROM, Scientific Principles of Conservation Course. Course exercises, 1977.

Mora, P. and Mora-Sbordoni, L. Metodo per la rimozione di incrostazione su pietre calcaree e dipinti murali. In: Problemi di Conservazione, Bologna: Editrice Compositori, 1975, pp. 339-344.

Ex. 25 REPOINTING OF STONE AND BRICKWORK

AIM

Mortar joints in masonry have the function of bonding the units together and levelling any irregularity. The finishing of joints at surface level is known as pointing. The pointing condition of a building is an important factor in its appearance and maintenance. Before beginning repointing work, it is important to determine the cause of mortar decay, as repointing alone will not cure the fault. Previous to any repointing, the original mortar should be analyzed in order to replace it with a compatible new mortar (see Exercises 21 and 22).

EQUIPMENT

Chisels, hammers, bristle brushes, trowels, pointing tools.

MATERIALS

Binders (lime putty, hydrated lime, hydraulic lime, etc.), sand, tap water.

PROCEDURE

1. Remove the defective or decayed mortar from the joints until sound material is reached. For rubble stone work, the areas to be repointed must be raked out to a depth of at least 40 to 100 mm. For brickwork, the corresponding depths are 12 to 25 mm. If the joint cannot easily be raked back to 10 mm, then repointing is premature. Raking out of old mortars should be carried out with a suitable tool, such as an old wood chisel, so as not to damage the adjacent masonry.
2. Once raking out is complete, thoroughly wash out the joints (using bristle brushes) to remove any remaining loose and dusty material. At the same time, wet the surfaces of the stone or brick which are to receive the new mortar, thus reducing the absorption of moisture from the pointing when applied.
3. Mix the appropriate mortar to be used for repointing.
4. Apply the mortar to the joints which have been washed out and thoroughly wetted. The mortar should be rammed well into the joint with a suitable pointing tool (not a trowel), so that it fills the joint and adheres firmly to the sides, leaving no voids.
5. The joint should be finished just sufficiently far back from the surface of the stone or brick to avoid encroachment of the mortar over worn arises, which will result in

Ex. 25 (continued)

weak and vulnerable feather edges to the joint. A bristle brush or a water spray may be used to roughen the surface of the joint.

6. Repointed areas should be protected from the drying effect of strong sun or wind by wet sacking or a similar damp material. If frosts are expected, the work should be covered overnight.

BIBLIOGRAPHY

Hawkins, Bob. Repointing of stone and brickwork. Matlock, Derbyshire, England, Design and Conservation Section, County Planning Department.

Ex. 26 INVESTIGATION OF THE CARBONATION PROCESS IN LIME MORTARS BY MEANS OF PHENOLPHTHALEIN

AIM

Knowledge about the carbonation process in lime mortar can only be achieved by practice. Literature and technology today deal mostly with cement and hydraulic binders, which behave quite differently. This exercise will show how temperature, relative humidity, air circulation, and water can influence the carbonation of lime mortar samples placed in different climatic conditions. It should be pointed out that it would be easy to expand this simple investigation to test other mixtures, other surface treatments, etc. It is also important to remember that though this test does not have the accuracy of a scientific investigation, it could be very useful as part of a preliminary survey, as a comparative work site test (see appendix: modified procedure for in-situ investigation), or as an educational exercise.

EQUIPMENT

Thermohygrograph, room or chamber with controlled climate, thermometer, hair hygrometer, fan, plastic ring molds, pipets, small vessels for mixing, felt-tip pen, absorbent paper.

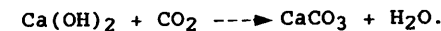
MATERIALS

Lime putty, sand, vaseline, distilled water, acetic acid, 1% solution of phenolphthalein (in 95% alcohol).

PREMISES

Lime mortar hardens in two major steps:

1. The mortar dries as the excess water soaks into the substrate and the rest evaporates. This dry lime mortar is hard enough to take a little stress. Like dry clay, it will be dissolved by water.
2. The dry lime mortar (calcium hydroxide) reacts with the carbon dioxide in the air and *carbonates*, forming calcium carbonate or limestone:



This produces a mortar that is almost insoluble in water. There is also a considerable rise in strength.

The problem is that the carbonation process is rather delicate and varies according to the temperature, moisture, presence of CO<sub>2</sub>, etc., as well as the physical characteristics of the material, such as pore structure, thickness, and so on.

ex. 26 (continued)

Thus it can easily happen that carbonation does not take place within a reasonable period of time. It can take years (tens and even hundreds of years!) instead of days or weeks. Another problem is that you cannot see if a lime mortar is carbonated or not, unless you use some kind of chemical indicator.

Phenolphthalein can be used for this purpose because it reacts with a sharp red color to alkaline materials and is colorless in a neutral or acid environment. Since calcium hydroxide is highly alkaline (basic) and calcium carbonate is almost neutral, phenolphthalein can be used to check the progress of the carbonation process.

Slaked lime (calcium hydroxide) is somewhat soluble in water and thus gives a clear alkaline reaction: phenolphthalein turns sharp red.

The carbonated lime (calcium carbonate) is almost insoluble in water which results in a neutral reaction.

The carbonation process starts from the surface of the mortar as this is the first place reached by the CO<sub>2</sub> in the air. The same process also starts from the surface of the lime crystals and works inward. This means that until the crystals are completely carbonated, you will have an alkaline reaction in a mortar that is not completely dry. If the mortar is dry, you will have no reaction in those parts of the sample having partly carbonated lime (i.e. where the crystals are transformed to calcium carbonate on the surface). If these crystals still contain calcium hydroxide in their inner parts, you will observe an alkaline reaction from those internal parts after a few minutes. The reason is that even the purest alcohol (in which the phenolphthalein is dissolved) contains water which, after a while, dissolves the alkaline calcium hydroxide in the uncarbonated parts of the crystals.

In a mortar that is not completely dry, the alkalinity is high enough even in the partly carbonated parts to give an immediate sharp red reaction.

A fully carbonated mortar will give no red reaction, regardless of whether it is wet or dry.

With phenolphthalein and distilled water, it is thus possible to test if a lime-bound mortar is carbonated or not, or if it is in an intermediary stage.

Ex. 26 (continued)

#### PROCEDURE

1. Prepare a mortar of lime putty and sand, 1:3 parts by volume.
2. Grease the plastic ring molds with vaseline to prevent the mortar from sticking.
3. Place the molds on at least 4 layers of absorbent paper to soak excess water from the specimens.
4. Fill the molds with lime mortar. Check for complete filling also at the bottom. Remove excess mortar, leaving the surface of all samples in the same grade of smoothness.
5. Leave the samples in the laboratory at approximately 50% relative humidity and 20°C for 24 hours. When the mortar has set, carefully lift away the molds and put a number from 1 to 9 on the top surface of all samples. Use a felt-tip pen for the numbering.
6. Place the samples (with the number upwards) on flat movable wooden boards with a minimum distance of 2 cm between samples.
7. Place samples 1-7 in the internal climatic conditions specified below. Place samples 8 and 9 outside, one in the sun and the other in the shade, using a thermohygrograph to record external temperature and humidity conditions.

SAMPLE	1	2	3	4	5	6	7	8	9
Room temp. °C	20	20	50	20	20	20	20	Sun	Shade
Room R.H. %	100	75	50	50	50	50	50	-	-
Air circulation	-	-	-	-	Fan	Fan	Fan	-	-
Wetting cycles	-	-	-	-	2-3x	-	-	-	-

8. The extent of carbonation is indicated by the reduction of alkalinity observed by dripping indicator solutions (i.e. Phenolphthalein) on freshly broken surfaces of the mortar samples. Break all: samples after 4 days as shown in Fig. 1a. Test with phenolphthalein on piece 1 and compare. If a bright red color appears, the sample is not yet carbonated.

Ex. 26 (continued)

9. After 3 more days, break the samples as shown in Fig. 1b; test again on piece 2 and compare. If the sample is not yet carbonated, wait a week and then repeat the procedure on piece 3.

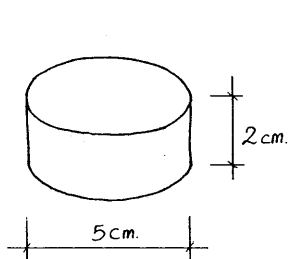


Fig. 1

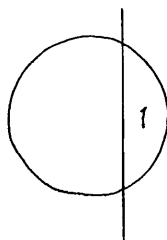


Fig. 1a

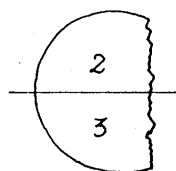


Fig. 1b

10. Note the effect of temperature, humidity (in the specimen and in the surrounding air), wind (natural or by fan) and CO<sub>2</sub> content on the carbonation process. Note the difference between exposed and hidden surfaces (top and bottom of samples, etc.).

#### BIBLIOGRAPHY

British Research Establishment. Carbonation of concrete made with dense natural aggregates.. Watford: B.R.E., 1978.

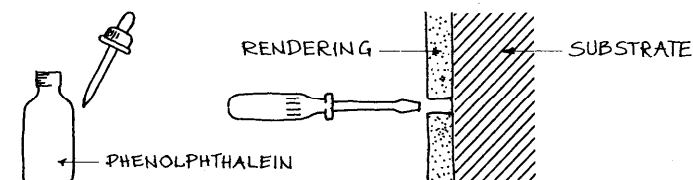
Holmstom, Ingmar. Lecture notes. ICCROM International Architectural Conservation Course, 1981.

Ex. 26 (continued)

#### APPENDIX: MODIFIED PROCEDURE FOR IN-SITU INVESTIGATION

On a building site, proceed as follows (see diagram below):

1. Drill a hole in the dry lime mortar (rendering, joint, etc.) down to the substrate (e.g. brick wall) using a nail or small screwdriver. If possible, choose a hidden place for the drilling. The drill site, however, should be representative of the total subject area in terms of climate, drying conditions, etc.
2. Clean the hole of grains and dust by blowing. This is most easily done with a drinking straw.
3. Apply phenolphthalein in the hole. A few drops are sufficient; application is most easily accomplished with a pipet. Be careful not to contaminate the pipet with drill dust, etc.



4. Observe the reaction. If the hole turns completely red, there is no carbonation or the carbonation process has started but the mortar is too rich in moisture.

In many cases, you will see no reaction near the surface and a sharp red reaction behind it. This tells you that the carbonation process has started in the outer parts but has not yet reached the inner parts.

5. If there is no reaction, apply a few drops of distilled water. Still no reaction means that the mortar is fully carbonated; a red reaction with the water means that carbonation has started all over but is not yet completed.
6. To remove the red discoloration immediately, apply acetic acid. If you leave the discoloration, it will disappear in time.
7. Mend the drill hole with an appropriate mortar.

Ex. 26 (continued)

If the observed carbonation process is not satisfactory,  
investigate the possible reasons for the problem:

Were the conditions too wet, too dry, or too cold? Adjust the  
conditions by heating, by ventilation, or by creating shade.

Test again in new holes and compare results.

ARCHITECTURAL SURFACES:

RENDERS. PLASTER (INTONACO). PAINT

## Ex. 27 SAMPLING OF ARCHITECTURAL SURFACE MATERIALS

### AIM

The aim of this exercise is to provide general guidelines for the sampling of architectural surface materials such as renders, plasters (intonaco), and paints.

### EQUIPMENT

Drawings, photographs or blueprints of the subject building, camera, soft bristle brushes, scalpel, chisel, mallet, sample containers, labels.

### PROCEDURE

#### A. Fundamental Principles:

1. Complete documentary research should be carried out before sampling is begun.
2. The objectives of the sampling should be clearly defined from the outset.
3. The number and size of the samples should be kept to the minimum that will provide the desired information. Samples must be representative but should be taken, where possible, from areas where they cause least damage to the structure. In the case of paints and renders, undisturbed areas (such as just under the cornice) yield the most information.

Remember -- you need to know what you are looking for!!!

#### B. Description/Documentation:

1. Prior to sampling, the subject building must be carefully documented. This should include a thorough description, both written and visual, of:
  - a. The building's *environment*.
  - b. The building *form* and *structure*.
  - c. The *types of building materials* and their characteristics: color, texture, particle size, etc.
  - d. The *condition* of the various materials; zones of obvious deterioration due to weathering, biological attack, or other causes.
  - e. Previous restoration, repair, substitution, interventions.
2. The areas to be sampled should be photographed before, during, and after sampling.

Ex. 27 (continued)

### C. Sampling Methodology:

1. A sample of surface material generally consists of a series of strata representing various finish layers, subsequent interventions, and surface deposits. A truly representative sample should include all the existing layers in addition to the substrate. The thickness of the sample will thus differ in each case, depending on the type of material and, the building history. No sample, however, should be larger than 3 cm<sup>3</sup>.
2. Samples should be taken with appropriate tools, chosen according to the consistency of the surface materials. In general, the most commonly employed sampling instruments are:
  - a. Soft bristle brushes for powdery material.
  - b. Scalpels or exacto-blades for paints and more coherent finishes.
  - c. Chisels and mallets for harder renders and plasters.
3. Samples should be collected in small containers of appropriately inert materials which can be labelled and sealed tightly. Plastic test tubes, measuring about 6 cm in length and having a fitted stopper have proved useful for this purpose. Avoid sheets of paper, cardboard, paper envelopes ... in short, any fragile material that might deteriorate. Put only one sample in each container.

The sample label should include the name of the sampler, the date, the name of the building, and the number of the sample. It is best to number samples sequentially; each number should also be indicated on a corresponding photograph or drawing of the subject building. In this way, it is not only possible to locate the area from which a sample was taken but also to retrace the chronology of the sampling procedure.

Conserve the samples in their containers until they can be appropriately prepared for laboratory analysis.

### BIBLIOGRAPHY

Istituto Centrale del Restauro. Corso sulla manutenzione di dipinti murali, mosaici, stucchi (DIMOS), Part I, Modulo 4: Tecnica delle sezioni stratigrafiche. Rome: Istituto Centrale del Restauro, 1978.

Italian Normal Committee. NORMAL - 3/80: Materiali lapidei: campionamento.

Ex. 28 IMBEDDING A SAMPLE FOR CROSS SECTION

### AIM

In order to be subject to microscopic analysis, a sample of plaster or paint must be:

1. Imbedded in a cold-setting resin
2. Cut in cross section
3. Polished and
4. Inserted in a container for observation and conservation.

The following two exercises will outline the process of sample preparation.

### EQUIPMENT

Molds, plastic cups, mixing rod, plastic gloves, vacuum pump, dessicator.

### MATERIALS

Polyester resin\*, catalyst for resin, release agent for mold. \*

The chosen resin must:

1. Be perfectly transparent
2. Have a low index of refraction (ideal value 1.54)
3. Not yellow or darken as it ages
4. Not "depolymerize" in time
5. Not react with the sample material (i.e. must not liberate acidic substances)
6. Be cold-setting.

The polyester resin "SNIATRON 5119", a product of SNIA, has been found to meet all these requirements. It is particularly adapted to use at ambient temperatures utilizing cobalt octoate (cobalt 2 - ethylhexoate) as the catalyst and methyl ethyl ketone peroxide as the hardener. However, any other polyester resin that meets the stated requirements could be used to imbed the samples.

### PROCEDURE (see illustrations which follows)

1. Work on a sheet of paper

#### 2 Preparation of Mold:

- a. Prepare a mold to retain the liquid resin until it has set into a solid: Molds that have been used successfully are:
  - 35 mm film containers (type without screw lids)
  - polythene ice cube trays (cubes about 2 cm<sup>2</sup>).

Ex. 28 (continued)

- b. Use a release agent\* for all types of molds except polythene to prevent the resin from sticking. Possible release agents are:

- vaseline
- thick oil
- grease
- polyester release agent.

\* Preliminary testing is required to assure that the chosen release agent does not react with the resin.

- c. Make sure that the sample and mold are completely prepared before mixing the resin. The mold should have a label indicating the provenance and number of the sample, the date, and the name of the experimenter.

3. Mixing the resin: For each sample, use about 12-25 cc of polyester resin, depending on the sample size. Put the resin in a plastic cup. Measure and add the correct amount of catalyst. Mix well and the resin is ready for use.

Table of Catalyst Quantities for Polyester 5119

Resin	Maximum	Normal	Minimum
50 cc	67 drops	23 drops	15 drops
25 cc	34 drops	12 drops	8 drops
12 cc	17 drops	6 drops	4 drops
	cold weather	normal weather	hot weather

Quantity of catalyst in weight: Maximum 3%, Normal 1%, Minimum 0.5%; 45 drops = 1 cc.

4. Imbedding Samples:

- a. Put a 3 to 4 mm layer of resin at the bottom of the mold. When this layer has partially set to a gel state, place the sample with the painted side up in the center of the partly-set resin. Press lightly to make it adhere. Pour another layer of resin on top to cover the sample.
- b. Experience will assure the proper timing. If the first layer of resin is allowed to set too long, the two layers of resin will not stick together. If the first layer of resin is not left long enough, the sample will sink to the bottom of the mold.

Note: If desired, extra catalyst may be added to increase the setting speed. About three extra drops of catalyst are usually sufficient. Again, experience will dictate the optimum proportions.

Ex. 28 (continued)

5. Creating a Vacuum: To assure penetration of the resin into the pores of the sample and to remove any air bubbles from the resin, put the filled mold for about ten minutes in a dessicator that is connected to an hydraulic vacuum pump.

6. Removing Imbedded Samples: When the resin is hard\*, remove the imbedded sample from the mold by a sharp tap with the palm of the hand. For the polythene molds, simply press gently on the bottom of the mold to eject the imbedded sample.

\* Setting time is normally about 1/2 hour to the gel state and 24 hours to harden.

7. Label the sample immediately.
8. Clean your hands and the working area with a solvent (such as acetone). The sample is ready to be cut in cross section.

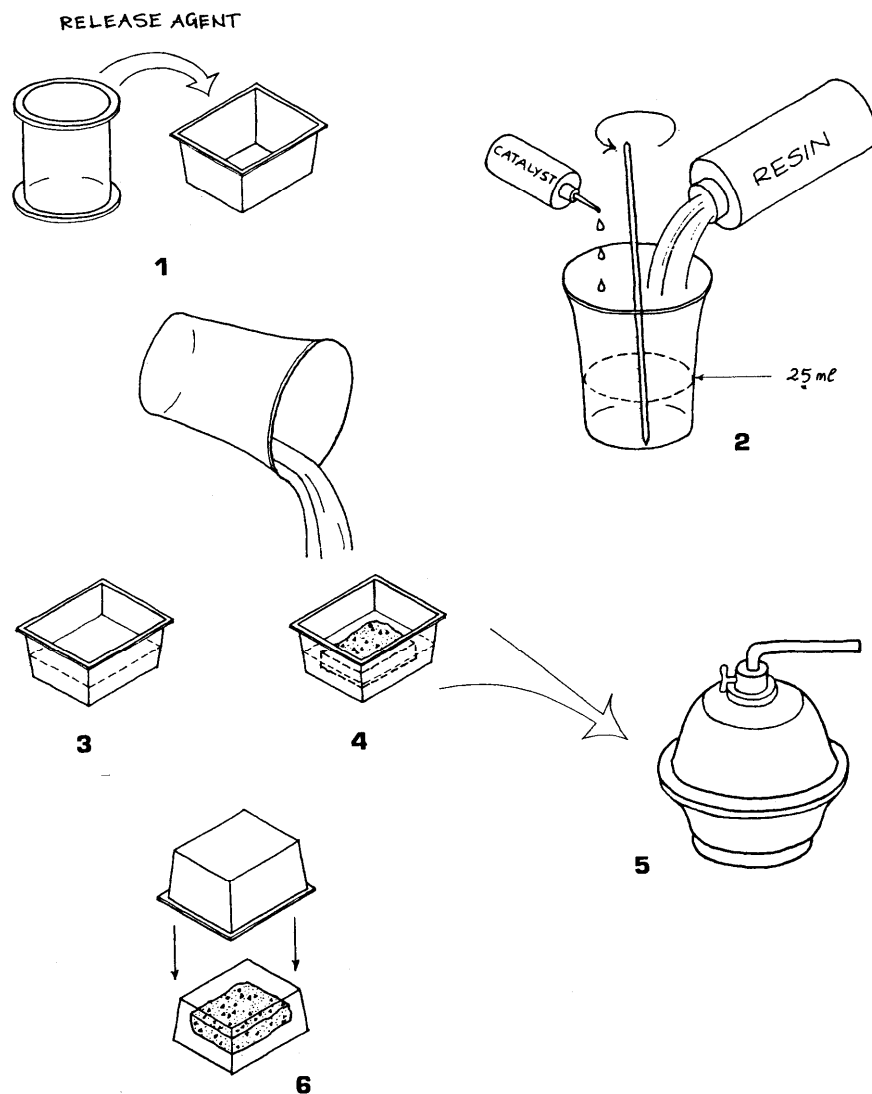
BIBLIOGRAPHY

ICCROM, Scientific Principles of Conservation Course, Course exercises.

Istituto Centrale del Restauro. Corso sulla manutenzione di dipinti murali, mosaici, stucchi (DIMOS), Part I, Modulo 4: Tecnica. delle sezioni stratigrafiche. Rome: Istituto Centrale del Restauro, 1978.

Ex. 28 (continued)

# IMBEDDING A SAMPLE FOR CROSS SECTION



## Ex. 29 CUTTING AND POLISHING A CROSS SECTION OF SURFACE MATERIAL: OBSERVATION OF THE SAMPLE

### AIM

Once imbedded in the synthetic resin, a sample must be cut in cross section and then polished with various grades of abrasive paper of diminishing coarseness. It can then be examined under a microscope. This exercise outlines the cutting and polishing procedure.

### EQUIPMENT/MATERIALS

Vice, hand saw, rough file, mineral oil (a liquid petroleum derivative), emery papers (grades 240, 320, 400, 600), microscope slide covers, Canada balsam, stereo binocular microscope (with photographic attachment if possible).

### PROCEDURE (see illustrations)

1. Work on a sheet of paper.
2. Cutting:\*
  - a. Clasp the embedded sample firmly in a vice.
  - b. With the metal hand-saw, cut it to reveal the cross section (obtaining a parallelepiped). The cut should be made along an oblique plane with respect to the horizontal surface of the sample in order to obtain a good legible stratigraphy.
  - c. With a file, roughly flatten the cut surface. The sample is ready for polishing.

\* The cutting procedure can also be done with a mechanical low-speed saw with diamond blade, if available.

3. Polishing:
  - a. This process requires a series of 3 to 4 grades of emery paper mounted on a flat board which can be inserted in a bath of mineral oil. The grades most commonly used are 240, 320, 400, and 600 (see Fig. 4).
  - b. Using the apparatus described above (or a grinder, if available), polish the sample with the varying grades of wet emery paper (starting from 240) using mineral oil as the lubricant. It is important to exert a uniform pressure on the sample block in order to avoid deformation of the sample surface.

A good method is to move the sample in one direction on the first emery paper, then to turn it 90° between each successive paper. Do not use the next finer paper until

Ex. 29 (continued)

all the scratches of the previous paper have disappeared. (This can be periodically checked under a stereo-binocular microscope with a magnification of about 10x). Wash the sample with mineral oil between each successive grade of paper.

- c. The polishing operation is complete when microscopic observation reveals that all scratches and surface imperfections have disappeared. The point of this procedure is, in fact, to obtain a plane surface adapted to observation under reflected light.

4. Mounting the cross section in a container for conservation:

- a. Mounting a cross section serves several purposes. It:
  - protects the sample surface from dust
  - facilitates the handling of the section
  - facilitates observation of the section with an optical microscope
  - permits the application of reference marks
  - simplifies storage and recording.

The container illustrated in Fig. 4 has been used successfully for the storage of cross sections. It consists of a plexiglass block (8 x 3 x 1 cm) having a circular cavity (roughly 2-3 cm in diameter and 7-8 mm in height) at its center.

- b. To prepare the sample for mounting, glue an ordinary glass cover slide to the polished surface of the cross section using a thin layer of Canada Balsam.\* To avoid the formation of air bubbles, rest the cover slide first on one edge of the cross section and then gently press it against the layer of balsam. Once it is in place, press again on the cover slide with a rectangular block to eliminate any bubbles and excess balsam. Allow it to harden.

\* Canada Balsam, obtained from the North American balsam fir, is commonly used because it has an index of refraction (1.52 - 1.54) close to that of both the cover slide and of the lens. Thus it does not interfere appreciably with microscopic observation of the sample.

- c. When the cross section is dry, fit it with the attached cover slide into the cavity of the plastic container described above in (a). Fix the edges of the cover slide to the container to avoid the accumulation of dust in the cavity. Normal adhesive tape is sufficient as a fixative.
- d. Label the plastic container, including the sample number, date, and provenance of the sample.

Ex. 29 (continued)

- e. So mounted, the cross section is ready for observation with a stereo-binocular microscope.

5. Observation of the Cross Section:

- a. Observe the cross section with a stereo-binocular microscope under reflected normal light. Note the sequence of layers. For each individual layer, note:
  - thickness
  - color
  - distribution of particles (uniformity, density, etc.)
  - discontinuity of adhesion or cohesion
  - penetration of superimposed substances; inclusions.
- b. Record your observations on the attached sample form.
- c. If the required equipment is available, make a photo-micrograph of the magnified cross section to accompany your written data. In the absence of photographic equipment, the cross section should be drawn to scale on millimeter paper to provide an indication of the thickness and relative position of the layers.

BIBLIOGRAPHY

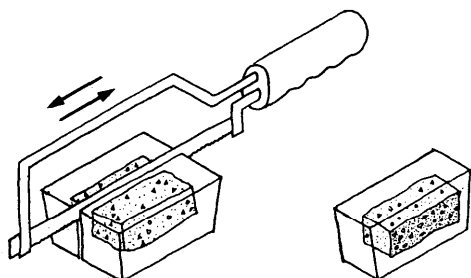
ICCROM, Scientific Principles of Conservation Course, Course exercises.

Istituto Centrale del Restauro. Corso sulla manutenzione di dipinti murali, mosaici, stucchi (DIMOS), Part I, Modulo 4: Tecnica delle sezioni stratigrafiche. Rome: Istituto Centrale del Restauro, 1978.

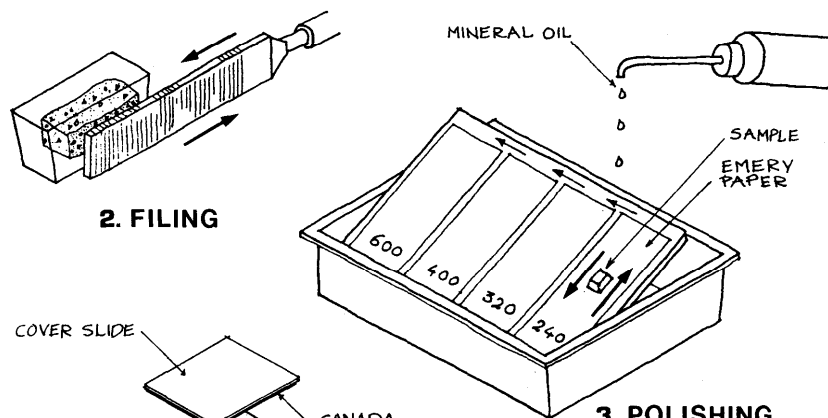
Perrault, Carole L. "Techniques employed at the North Atlantic Historic Preservation Center for the sampling and analysis of historical architectural paints and finishes." Bulletin of the Association for Preservation Technology, Vol. X, N°2, 1978, pp. 6-46.

Ex. 29 (continued)

CUTTING AND POLISHING A CROSS SECTION

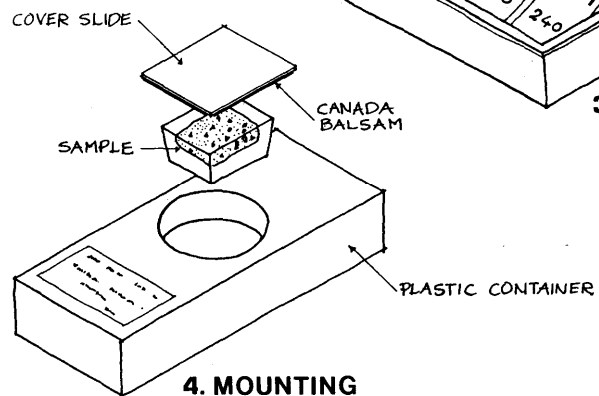


**1. CUTTING**



**2. FILING**

**3. POLISHING**



**4. MOUNTING**

Ex. 29 (continued)

SAMPLE OBSERVATION SHEET

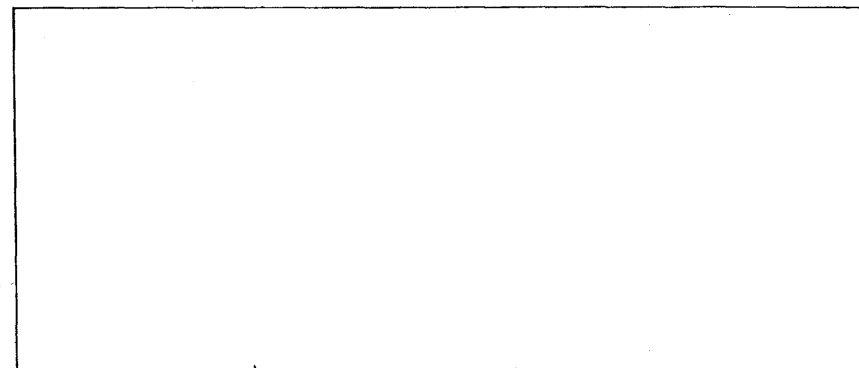
Name of Sampler:

Site:

Location:

Date:

Photograph or Drawing of the Cross Section



Microscopic Examination

Sample N°:

Magnification:

Location:

Type of Illumination:

Approximate number of layers:

Substrate:

Chronology/Description of layers (starting from substrate):

1.	9.
2.	10.
3.	11.
4.	12.
5.	13.
6.	14.
7.	15.
8.	16.

Ex. 30 SIMPLIFIED METHOD FOR IMBEDDING AND POLISHING  
A CROSS SECTION OF PLASTER OR PAINT

AIM

Though not as sophisticated as the procedure described in Exercises 28 and 29, the following simplified method (developed by Laura and Paolo Mora) is perfectly adequate for the preparation of most cross sections of architectural surface materials. It is quick, requires a much smaller quantity of resin, eliminates the need for cutting the sample, and allows all procedures to be carried out with only the simplest equipment. In addition, this method facilitates storage, making it possible to keep several samples from the same building or site in one container.

EQUIPMENT

Mixing vessels for resin, mixing rods, plastic 'stick' molds, files, varying grades of emery paper (240, 320, 400, 600), vice, microscope slide covers.

MATERIALS

Modelling clay, polyester resin, catalyst for resin, Canada balsam.

PROCEDURE (see illustrations below)

1. Imbedding the Sample

- a. Work on a sheet of paper.
- b. Put a layer of modelling clay (gray or white is best) in the bottom of the plastic 'stick' mold to serve as a support for the samples.
- c. Stand the samples on edge, in the modelling clay, making sure:
  - to leave a sufficient amount of space between samples;
  - that a portion of the sample protrudes above the lip of the plastic stick.
- d. Using adhesive labels on the plastic stick, indicate the date, number and provenance of each sample.
- e. Mix the polyester resin and catalyst in proper proportions (see Ex. 28).

Ex. 30 (continued)

- f. Pour the well-mixed resin into the groove in the plastic stick so as to completely fill it (thus surrounding the samples with resin). Be sure that the resin is level with the top edge of the groove.

g. Allow the resin to harden overnight.

2. Polishing

- a. Clasp the plastic stick containing the hardened resin firmly in the vice.
- b. Using the file, flatten the surface of the sample until it is roughly level with the top edge of the plastic stick.
- c. Starting with the coarsest emery paper (240), polish the sample surface with increasingly fine grades of emery paper until all scratches and surface imperfections have disappeared. Again, it is important to exert a uniform pressure on the sample block in order to avoid deformation of the sample surface. Similarly, a finer paper should not be used until all the scratches of the previous one have disappeared.

3. Mounting

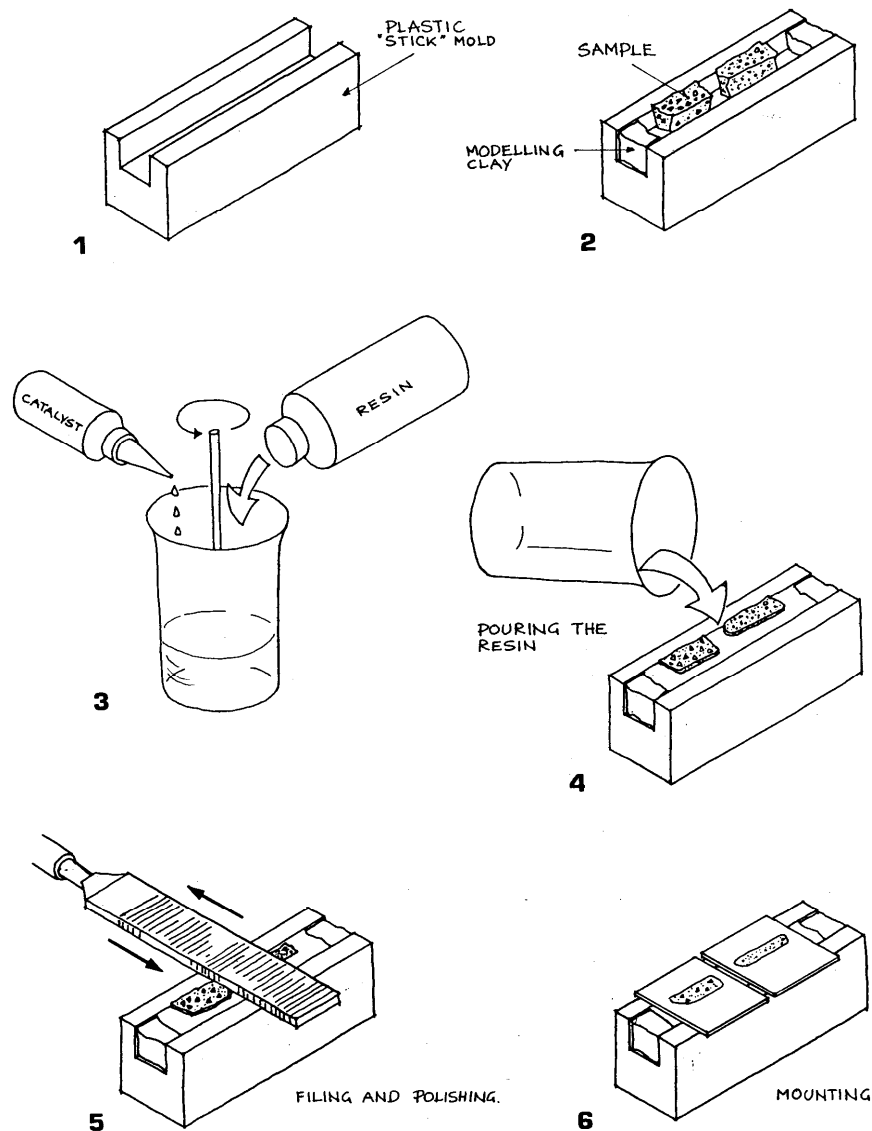
- a. When the polishing operation is complete, glue an ordinary glass cover slide to the surface of each cross section using a thin layer of Canada balsam. Gently press on the cover slide with a piece of soft cloth to eliminate any air bubbles and excess balsam. Allow it to harden.
- b. When the balsam has dried, the cross sections are ready for observation with a stereo-binocular microscope.
- c. Upon completion of the investigation, the samples can be easily stored. A simple archival system, such as a number code attached to each plastic stick, is recommended.

BIBLIOGRAPHY

Mora, L. and Pi. Unpublished methodology for the imbedding and polishing of cross sections, 1985.

Ex. 30 (continued)

IMBEDDING AND POLISHING A SAMPLE FOR CROSS SECTION:  
SIMPLIFIED METHOD



Ex. 30 (continued)

SAMPLE OBSERVATION SHEET

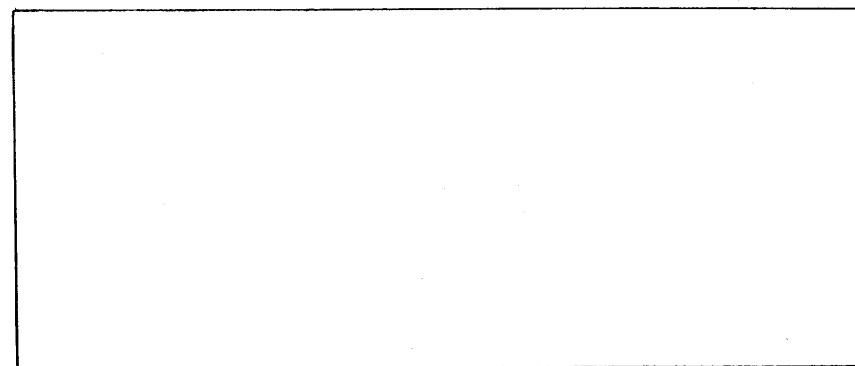
Name of Sampler:

Site:

Location:

Date:

Photograph or Drawing of the Cross Section



Microscopic Examination

Sample N°:

Magnification:

Location:

Type of Illumination:

Approximate number of layers:

Substrate:

Chronology/Description of layers (starting from substrate):

1.	9.
2.	10.
3.	11.
4.	12.
5.	13.
6.	14.
7.	15.
8.	16.

WOOD

## Ex. 31 WOOD STRUCTURE

### AIM

Wood is a heterogeneous material characterized by specific elements and having different properties in each of its three directions. This exercise should provide an introduction to the typical structures of hard and soft woods as viewed under the microscope.

### EQUIPMENT/ MATERIALS

Stage microscope, wood samples.

### PROCEDURE

#### 1. The three directions of wood:

Three sections of the same species of wood will be given to you. Identify under a microscope (40x and 100x magnification) the cross, tangential, and radial sections. Mark them C, T, and R.

Observe:

- cross section: annual growth ring
- tangential section: longitudinal cells
- radial section: radial rays (cross pattern)
- tangential section: cross section of radial rays.

#### 2. Soft wood (coniferous) and hard wood (deciduous):

Three prepared sections of soft wood and three analogous sections of hard wood will be given to you.

Note the difference between:

- the two cross sections
- the two tangential sections
- the two radial sections.

Refer to figures 1 and 2.

What are the typical characteristics of hard and soft woods in each of their three dimensions?

### BIBLIOGRAPHY

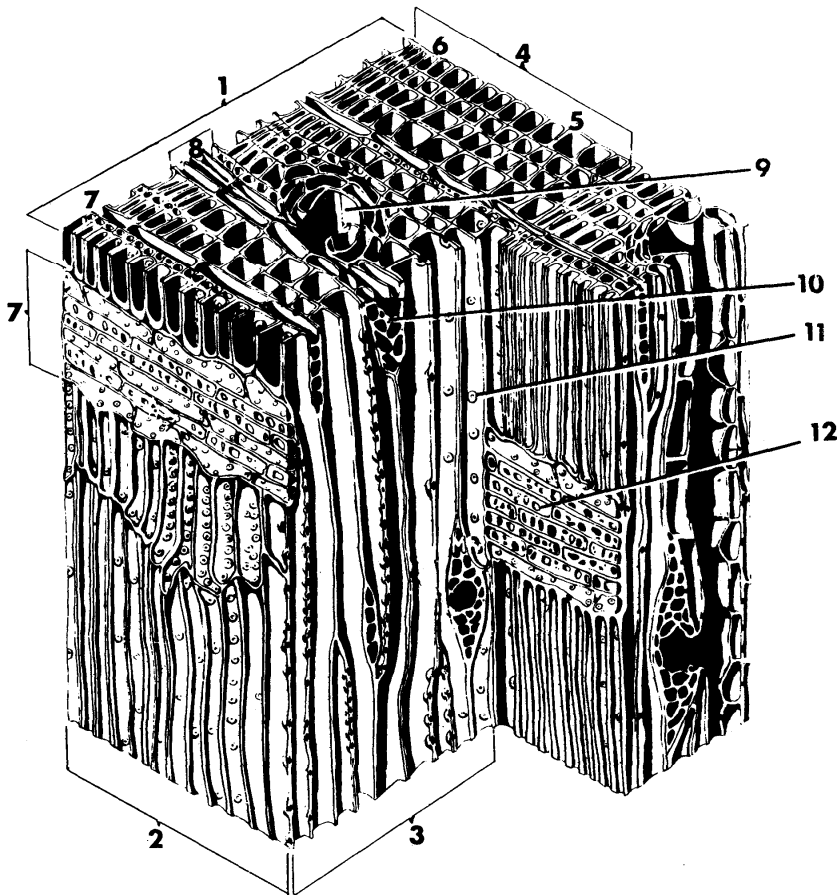
Foulger, A.N Classroom demonstrations of wood properties. ,Washington, D.C.: U.S. Department of Agriculture, Forest Products Laboratory, 1969.

Jane, F.W. The structure of wood. London: Adam and Charles Black, 1970.

Meylan, B.A. Three dimensional structure of wood. A scanning electron microscope study. London: Chapman and Hall, 1972.

Ex. 31 (continued)

Fig. 1: SOFTWOOD



Softwood Key—

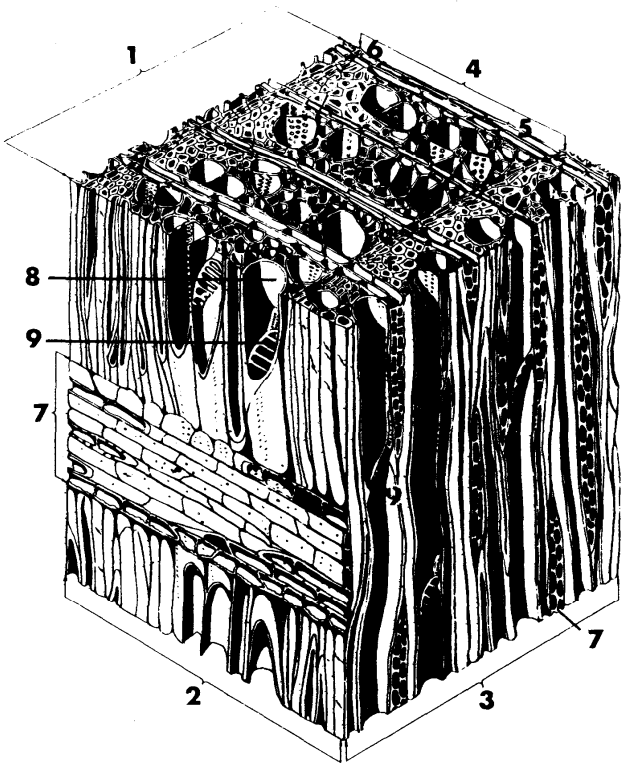
- 1. CROSS-SECTIONAL FACE
- 2. RADIAL FACE
- 3. TANGENTIAL FACE
- 4. ANNUAL RING
- 5. EARLYWOOD
- 6. LATEWOOD
- 7. WOOD RAY
- 8. FUSIFORM RAY
- 9. VERTICAL RESIN DUCT
- 10. HORIZONTAL RESIN DUCT
- 11. BORDERED PIT
- 12. SIMPLE PIT

M-134,267

From Foulger, p. 2.

Ex. 31 (continued)

Fig. 2: HARDWOOD



Hardwood Key—

- 1. CROSS-SECTIONAL FACE
- 2. RADIAL FACE
- 3. TANGENTIAL FACE
- 4. ANNUAL RING
- 5. EARLYWOOD
- 6. LATEWOOD
- 7. WOOD RAY
- 8. VESSEL
- 9. PERFORATION PLATE

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From Foulger, p. 4.

## Ex. 32 SWELLING AND SHRINKAGE OF WOOD

### AIM

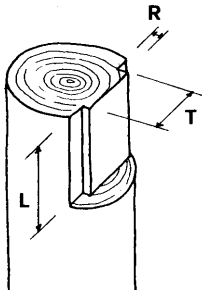
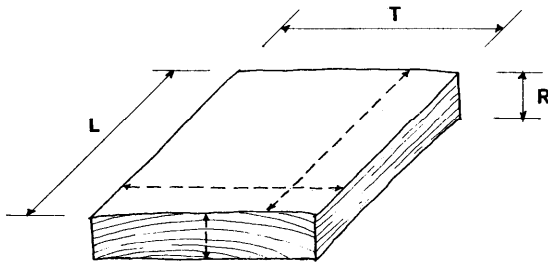
This exercise studies the swelling and shrinkage of wood as a variable of its moisture content. Special attention is paid to the effect of restrained swelling, when the tendency to swell is greater than the possibility of the material to do so. Restrained swelling has practical applications to the behavior of exposed wooden structures.

### EQUIPMENT/ MATERIALS

Ruler, clamps, clamping plates, hygrometer, wood samples.

### PROCEDURE

1. Take two pieces of wood (samples 1 and 2), approximately 150 x 150 x 20 mm. Measure the dimensions of each piece accurately. Draw a pencil line on the wood where you took the measurements, so as to locate the same place -again. Record the measurements in Data Sheets 1 and 2.
2. Measure the wood moisture content (WMC) of each piece with the hygrometer. Record the values in Data Sheets 1 and 2.



### THREE DIMENSIONS OF WOOD

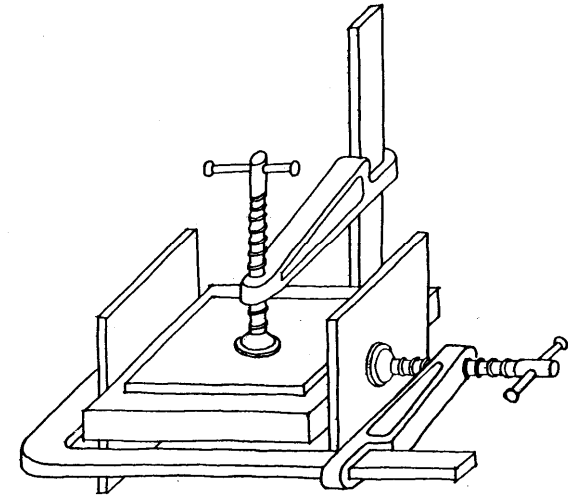
T = tangential dimension. It should be noted, however, that all values said to be tangential are not tangential but circumferential. On a micro-scale, circumferential and tangential are the same.

R = radial dimension. The radial dimension of a wooden board is radial only in one part, often in the center as illustrated. At the sides, this dimension is usually tangential.

L = longitudinal dimension.

### Ex. 32 (continued)

3. Clamp one of the pieces of wood as illustrated below so that it cannot swell in the tangential direction (where the tendency to swell is greatest). Do not tighten the clamp to the point that you crush the wood. Use clamping plates to ensure an even stress. Prevent the piece of wood from buckling with another clamp and plates, as illustrated.



4. Immerse both the clamped and unclamped pieces of wood in water, making sure that they are completely covered. Measure their dimensions and moisture content after 1 hour and after 24 hours, and record the values.
5. Allow both pieces of wood to dry, preferably in the sun. Measure the dimensions of each sample after 1 hour. Remove the restraining clamps after 24 hours and measure the samples again. Measure the wood moisture content at each interval with the hygrometer. Record all values in Data Sheets 1 and 2.

### DISCUSSION

- What general statements can you make about the swelling and shrinkage of wood?
- In which direction was swelling most pronounced?
- Did swelling occur immediately or only after prolonged wetting?
- What effect did the imposition of restraint have upon the behavior of swelling wood?

Ex. 32 (continued)

- What observations can you make about the capacity of wood to regain its original dimensions when dry (after a period of prolonged wetting)? What were the differences between the restrained and unrestrained wooden pieces in this regard?
- How would the properties observed in this exercise affect the behavior of wooden members subject to periodic wetting and drying? What types of deformation might you expect? What types of solution might you propose to correct situations in which such deformations occur?

**DATA SHEET 1: Unrestrained Swelling**

SAMPLE N°1	DRY	WET	1 hr	WET 24 hrs	DRY 1 hr	DRY 24 hrs	
	mm	mm	%Δ	mm	%Δ	mm	%Δ
T							
R							
L							
WMC%							

**DATA SHEET 2: Restrained Swelling**

SAMPLE N° 2	DRY	WET	1 hr	WET 24 hrs	DRY 1 hr	DRY 24 hrs
	mm	mm	%Δ	mm	%Δ	mm
T						
R						
L						
WMC%						

BIBLIOGRAPHY

Kaila, P. Lecture Notes, ICCROM International Architectural Conservation Course, 1985.

Ex. 33 CROSS SECTIONS AND IDENTIFICATION OF WOOD

AIM

It is sometimes necessary to identify a species of wood before deciding upon a treatment. This exercise outlines the basic procedure for the preparation and interpretation of wood samples. Often, in complicated cases, accurate identification will require the assistance of an expert or a specialized institution. A basic understanding of the analytic process, however, will better enable the architectural conservator to request the information required.

EQUIPMENT/CHEMICALS

Hot plate, stage microscope, hand microtome, beakers, watch glasses, spoon, tweezers, razor blade, microscope slides, cover slides, fine brush, sodium hydroxide, alcohol, distilled water.

PROCEDURE

1. Sampling:

Cut a cube measuring approximately 10 x 10 x 10 mm from the wood to be sampled. Try to orient the cube so that the faces are almost parallel to the three-dimensional directions of the wood.

2. Preparation:

Boil the cube in a 2-4% aqueous solution of sodium or potassium hydroxide. Softwoods require 1/2 to 2 hours; hardwoods require longer. The boiling process can be considered complete when the wood cube sinks to the bottom of the solution. Transfer the cube into pure alcohol for several hours (preferably overnight).

3. Cutting and Mounting:

Take a razor blade or sharp knife and trim the cube so that its faces are exactly parallel to the three-dimensional directions of the wood.

Clamp the cube into the hand microtome.

Cut very thin laminae (thin sections) from the cube. (If a hand microtome is not available, this can also be done with a razor blade.) Begin with the radial plane; then do the same in the; tangential plane and, finally, across the grain.

Transfer each thin' section to a microscope slide with the help of a fine brush and wet with a drop of alcohol/water solution (2:1). Put a cover slide on top of the section. Label the slide.

Ex. 33 (continued) 4.

Identification:

Examine the prepared thin section with a stage microscope utilizing transmitted illumination (magnification of at least 100x is desirable).

Each species of wood has characteristic elements (pits, cells, etc.) in each of its directions. By comparison with known cross sections, it is possible to identify the species of the sample. The more damaged the sample, the more damaged the specific elements will be, making identification more difficult. Very often, in such cases, it will be necessary to consult a specialist.

BIBLIOGRAPHY

ICCROM, Scientific Principles of Conservation Course. Course exercises, 1977.

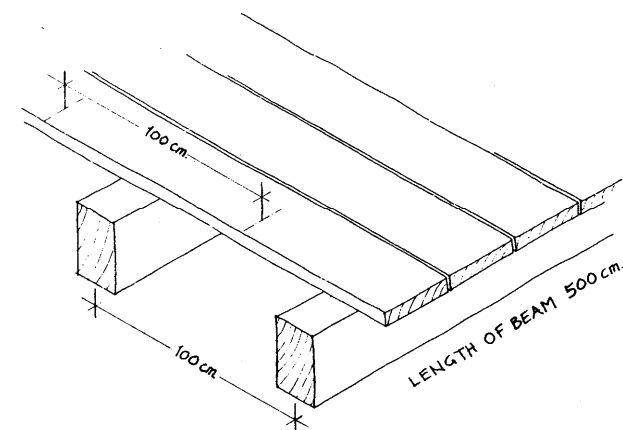
Schweingruber, Fritz. Microscopic wood anatomy. Zug: Zuercher A.G., 1978.

Ex. 34 TEST LOADING OF WOOD

AIM

The aim of this exercise is to simulate, on a small scale, a bending test to evaluate the structural strength of a wooden beam. A beam may be large enough to support a given load safely, but if its deflection is too large, a floor will vibrate or a plaster ceiling will crack. In practice, a beam is designed for bending and then investigated for deflection. The maximum allowable deflection therefore determines the load-bearing capacity of a wooden beam.

SAMPLE CALCULATIONS/ TESTING PRINCIPLES



Simplified Example of Load Test for a Floor Beam (see above): One beam carries a floor area of  $100 \text{ cm} \times 500 \text{ cm} = 5 \text{ m}^2$  Required live load (e.g. building code):  $250 \text{ kg/m}^2$  Required load on this beam:  $1250 \text{ kg}$   
Maximum allowable deflection:  $L/360 = 500/360 = 1.4 \text{ cm}$

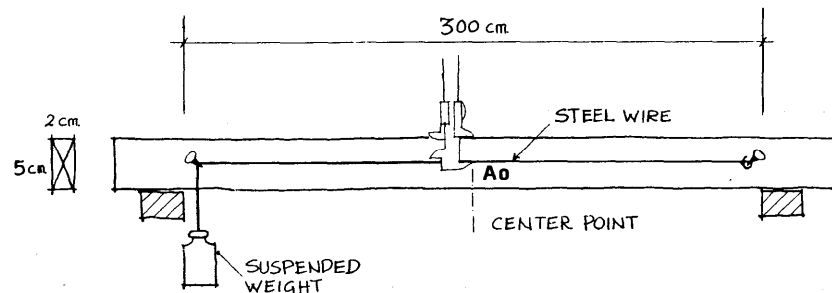
The test load is placed on the beam as illustrated on the following page. The load is applied gradually, increasing by increments of 10% of the total load every 24 hours. The deflection is read at each interval from a scale placed at the center of the beam. If the deflection does not exceed the maximum allowable (i.e. 1.4 cm) at 1250 kg, the test loads are removed. After another period of 24 hours, the recovery on the deflection should be at least 90%.

Ex. 34 (continued)

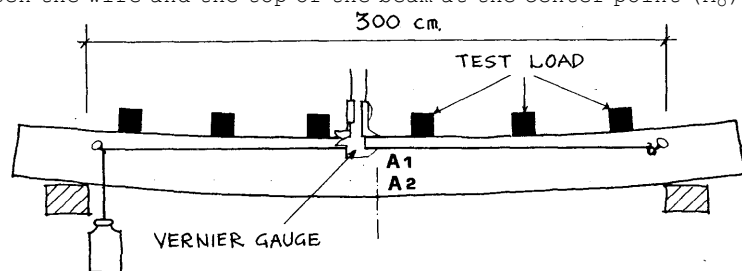
# EQUIPMENT/MATERIALS

2 large bricks, wooden beam, steel wire, test loads, vernier caliper.

# PROCEDURE

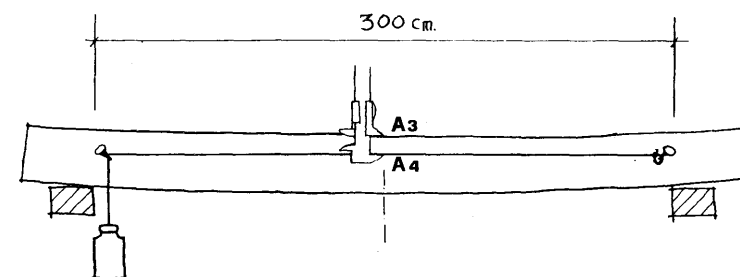


1. Support a wooden test beam on two large bricks. Record all dimensions of the beam (breadth, height, and length between bricks) and mark the center point between the two bricks.
2. Attach a steel wire to the beam along the center line, with a suspended weight at one end to keep it taut. Measure the distance between the wire and the top of the beam at the center point ( $A_0$ ).



3. Load the beam along its length with a weighed test load. Record the weight of the test load in the data sheet.
4. Using a vernier gauge, measure the deflection at the center of the beam immediately after loading and record the value ( $A_1$ ).
5. After 24 hours, measure the deflection again and record the value ( $A_2$ ).

Ex. 34 (continued)



6. Remove the test load. Record the deflection immediately after removal ( $A_3$ ).
7. 24 hours after removing the test load, again measure the deflection of the beam and record ( $A_4$ ).

DATA SHEET

	Measure A	Deflection ( $A_0 - A_1, A_2, A_3$ )
Unloaded	$A_0 =$	
Immediately after loading	$A_1 =$	
24 hours after loading	$A_2 =$	
Immediately after removal of load	$A_3 =$	
24 hours after removal of load	$A_4 =$	

Weight of test load \_\_\_\_\_

Calculated Weight \_\_\_\_\_

8. Using the formula below, calculate the load-bearing capacity of the test beam at the maximum allowable deflection. Compare the calculations with the experimental results in the data sheet to see if the beam behaved within the calculated limits during the 24-hour test period.

Ex. 34 (continued)

The maximum allowable deflection is usually calculated with formula:

$$W = \frac{384 \cdot E \cdot I \cdot d}{5 L^3}$$

L = length of beam  
b = breadth  
h = height

Where: W = Total weight (weight of beam + load) kg  
E = modulus of elasticity of beam kg/cm<sup>2</sup>

$$I = \text{Moment of inertia} = \frac{(b \cdot h^3)}{12} \text{ cm}^4$$

d = maximum allowable deflection  
= L/360 in new timber  
= L/180 in somewhat decayed timber  
= L/100 in bent old timber

Sample Calculations

Given a spruce beam of L = 300 cm, b = 2 cm, and h = 5 cm.

Specific gravity of spruce = 460 kg/m<sup>3</sup> so:  
Weight of beam of 0.03 m<sup>3</sup> = 1.4 kg  
Weight of load at maximum deflection = x  
Modulus of elasticity = 0.07 · 10<sup>6</sup> kg/cm<sup>2</sup>

$$\text{Moment of inertia} = \frac{(b \cdot h^3)}{12} \text{ cm}^4 = \frac{2 \cdot 5^3}{12} \text{ cm}^4 = 21 \text{ cm}^4$$

$$d = L/360 = 300/360 = 0.8 \text{ cm}$$

$$L^3 = 27 \cdot 10^6 \text{ cm}^3$$

Substituting in the formula:

$$W = \frac{384 \cdot E \cdot I \cdot d}{5 L^3}$$

L = length of beam  
b = breadth  
h = height

$$1.4 \text{ kg} + x = \frac{384 \cdot 0.07 \cdot 10^6 \text{ kg} \cdot 21 \text{ cm}^4 \cdot 0.8 \text{ cm}}{5 \cdot 27 \cdot 10^6 \text{ cm}^3}$$

$$\begin{aligned} 1.4 \text{ kg} + x &= 3.3 \text{ kg} \\ x &= 3.3 \text{ kg} - 1.4 \text{ kg} \\ x &= 1.9 \text{ kg} \end{aligned}$$

BIBLIOGRAPHY

Kaila, P. Lecture Notes, ICCROM International Architectural Conservation Course, 1985.

Ex. 35 PREPARATION AND APPLICATION OF TRADITIONAL OIL HOUSE PAINTS

AIM

The following exercise provides an introduction to the proper preparation and application of traditional oil-based paints to wooden surfaces.

EQUIPMENT

A wooden panel or door, a methylene chloride paint stripper, sand paper, linseed oil, zinc white paste, a powdered earth pigment (e.g. yellow or red ochre), turpentine, paint brushes, spatulas, scrapers.

PROCEDURE

1. Preparation of the Wooden Surface

- a) Using a brush, apply a thick layer of the methylene chloride paint stripper to the wooden surface (to be repainted). Wait 3 to 5 minutes and then scrape off the old paint layer with the scraping tool.
- b) Repeat this procedure until all layers of undesirable paint are removed. In general, the panel should be stripped of all synthetic paint. Any linseed oil-based paint which is strongly adhered to the surface can be allowed to remain.
- c) Once all layers of undesirable paint have been removed with the paint stripper, prepare the surface for the new paint by sanding. (In addition to methylene chloride, the paint stripper contains paraffin wax as a medium which must be removed from the surface before repainting.)

2. Mixing and Application of the Priming Layer

- a) Prepare the priming paint by mixing boiled linseed oil with the zinc white paste.
- b) Dilute the mixture by about 10% with a thinning agent (50% linseed oil and 50% turpentine).  
\* The principle in painting with traditional oil paints is to progress from less fatty (oily) paint layers to more fatty ones. If the priming paint is diluted only with oil, it becomes too fatty for a first layer. Turpentine is therefore used to create a mix with the desired characteristics.
- c) Cover the surface with a very thin layer of primer. The layer should not be opaquely white, but should rather have a veil-like appearance on the wood.

Ex. 35 (continued)

d) In actual practice, let the priming layer and each successive layer dry for one week. Remember that:

- Oxygen makes oil paint dry (oxidation).
- Light is necessary for the drying process. Warmth quickens the drying process, but too rapid a drying of the surface prevents oxygen from penetrating deeper, and thus has a negative effect. It is best not to paint with oils in direct sunshine or at temperatures below 5°C.
- The surface to be painted must be thoroughly dry before application of the oil paint. Water or water vapor does not penetrate a fresh linseed oil finish. Rain is thus less of a problem if it falls after application of the paint.

### 3. Mixing and Application of Principal Coats

- a) Since a 7-day drying period is not practical for the purposes of an exercise, sand down another part of the same wooden surface to which the primer was applied and brush off the dust.
- b) Mix a small amount of the earth pigment with a little oil to form a paste. Mix this pigmented paste into the priming paint prepared in step 2a (zinc white paste and boiled linseed oil, *undiluted* by turpentine).
- c) Paint the dry, sanded surface with this pigmented paint. (In actual practice, this layer would be applied on top of the priming layer after a 7-day drying period.) Notice how much thicker and more opaque this layer is, as compared to the priming layer. Note, too, the typical marks left by the brushes in the painted surface.

### 4. Cleaning, Care of Tools, Maintenance

- a) Wash your hands and the paint brushes with a thinning agent (oil and turpentine 1:1). Note:
  - The turpentine has a tendency to make your hands dry. It is advisable to apply a moisturizing cream afterwards.
  - Uncleaned, the paint brushes can be kept soft by storing them immersed in water. Paint cannot take oxygen from the water and therefore does not dry on the brushes.
- b) An exterior facade painted with a traditional linseed oil paint will require repainting after about 30 years.

### BIBLIOGRAPHY

Kaila, P. Lecture Notes, ICCROM International Architectural Conservation Course, 1985.

