2.1 IN-SITU VISUAL ANALYSIS AND PRACTICAL SAMPLING OF HISTORIC MORTARS

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1. Introduction

Sampling is a highly variable and dependant activity, subject to the ultimate aim of an investigation, and to the analyses performed on the samples in the laboratory. Sampling is defined in this context as the act of permanently removing material from building fabric for the purposes of analysis or characterisation of the material.

The sampling of historic mortar materials is usually performed as part of investigations either to assist in the conservation or restoration of a building, or an academic study into the characteristics of the materials used. Academic investigations can be pursued from many different disciplines including archaeology and material science. The quality of the conclusions reached in an investigation depends on the quality and relevance of the samples taken for answering the questions posed. Sampling, and early visual analysis is a crucial part of the *practical*, *analytical* procedure that is applied in all forms of historic mortar investigations. It should be pursued as rigorously and as carefully as are analyses performed on the samples once in the laboratory.

Appropriate sampling of historic mortars can be very difficult. Samples are often obtained in difficult physical and political circumstances and can be affected by many external issues, such as issues of conservation philosophy. This is especially so when sampling from culturally and architecturally important buildings where damage, and therefore sample quantity, must be kept to a minimum. An awareness of the interdisciplinary nature of historic mortar studies is also vital. Failure to be aware of the likely constraints placed on sampling by architects, conservators, owners and custodians of buildings can lead to considerable frustration for scientific analysts and lead them to question the validity of their investigations. Analysts must be sensitive to the needs of other professions involved in the conservation of historic buildings.

Sampling takes place through several stages which include the establishment and clarification of the objectives of an investigation, visual analysis of the structure (of its materials including building phases or stratigraphy), choice of analytical method for analysing the materials and finally sampling itself with associated information recording. The following sections deal with these stages and associated considerations in more detail.

This paper attempts to provide an overview of the considerations inherent in correct sampling methodology. It does not intend to be proscriptive, but to make the reader think carefully about what samples they need and how they must take them to answer questions relevant to their individual reasons for studying historic mortars.

2. Clarification of objectives

Two approaches are common in the analysis of historic mortars:

1. Conservation and Repair Related. Such investigations are aimed at the determination of the causes of evident problems in the deterioration of a historic building and/or the characterisation of existing materials in order to allow the practical selection of replacement materials.
2. Studies aimed at understanding the architectural, chemical and physical performance of historic mortars for the longer term development of replacement materials or the archaeological study of building technology and its associated social implications.

In both approaches mortars can be studied using a range of investigative techniques selected depending upon the information sought. Techniques ranging from visual assessment *in-situ* to high resolution chemical analysis will be chosen by investigators (Table 1).

Current ideas about the investigation of deterioration and conservation problems in buildings are moving towards considering only the minimum action required to answer investigative questions (van Hees, 1997; van Hees, 1999). No more samples should be taken than is required to answer questions using a sufficient number of analytical methods. This approach aims to be “analysis efficient” to reduce effort and costs, but is also fully consistent with mainstream conservation philosophies of minimum intervention and damage to buildings. However, it highlights the need for the very careful pre-analysis of the problem or topic of investigation to ensure that hypotheses relating to it are clearly formulated. The nature of the materials involved and their state of preservation should, ideally, be fully understood before decisions are made on the locations, numbers and type of samples to be taken. Decisions should be based on a sound understanding of the needs for authenticity, compatibility and re-treatability of materials and repair methods.

In practice many academic studies of mortars in historic buildings appear not to have such well defined aims as all that. In most, sampling is not well defined, which leaves the reader in no position to independently asses the reliability of the evidence from the analysis of the materials that stems from the quality of the samples. Only a minority of studies are clear as to their sampling context (Baronio and Binda, 1991; Schouwenberg et al, 1993). The clarification of objectives relating to damage diagnosis or academic study ties in closely with detailed knowledge of the building and its materials.

3. Visual building pre-analysis

Knowledge of the building is paramount to successful sampling and successful analysis of building material problems. Furthermore, the objectives of conservation/restoration plans should be central to sampling-analysis schemes (Knöfel and Schubert, 1993). Visual analysis with an understanding of the building construction including the types and functions of mortar and render materials is needed as is an awareness of the availability of samples and the ease of their removal. Table 2 lists the more generalised factors associated with the building, that should be considered before taking samples.

Visual analysis can achieve a great deal at minimum cost. Attention by experienced analysts can quickly clarify problems and identify materials. However it must be stressed that the correct people must be employed otherwise identifications can be erroneous. Visual analysis is a non-destructive technique and should be used as much as possible in advance of further laboratory or on-site technical investigations.

3.1 Stratigraphy of the building

The first analysis to be carried out is the visual study of the whole structure, with the focus falling second on the details of the mortar. Phases of construction (or "stratigraphy") must be clearly understood so that only the relevant materials for the investigation are sampled (Baronio and Binda, 1991; Jedrzejeweska, 1981). This is particularly important for studies where the full complexity and variability of historic materials and their temporal development are of interest. In practical and commercially based analyses, a very detailed understanding of building construction is perhaps less critical as long as the variability of materials is identified and the right materials sampled. However, cost effective and sensitive work will be facilitated by a clear understanding of the spatial and temporal distribution of materials. This knowledge is really a basic pre-requisite to correct sampling. If historic documentary sources are available concerning the different building phases, a great deal of useful information can be gained, for example the type of mortars, the types of building stones used, the provenance of the raw materials. This information is very useful and necessary for sampling relevant materials.

Where cost and time prohibits the complete understanding of a building's construction, full recording of information may not be possible. The minimum requirement for information gathering before sampling should include photographs, plans and drawings, that identify the locations of sampling points on the structure. Later, if more information is required then a full "stratigraphic" survey can be carried out. However, in the case where a particular problem arises that requires immediate attention, such information is not always obtainable. It is very important that the recording of details of the sampling, including reasons, location and the state of the materials is not ignored. Later work on the samples, especially for buildings where future sampling may be impossible, must not be compromised by a lack of information. An approach to information recording on-site is outlined in section 4.3.

Mortars can be inspected once the general visual analysis of the entire building is complete. Information on the types of binder and aggregate as well as decay forms and mechanisms can be gathered during visual analysis. Mortars that have contrasting properties can often be identified by inexpensive visual analysis prior to sampling. Binder type (if obvious), aggregate composition, texture and secondary products should be described. Examples of additional important features for inclusion in the visual analysis include the proper recording of colour (on a fresh broken surface if possible, not the weathered exterior which can be contaminated with pollution), the presence of possible biological decay and the environmental conditions. Completion of a visual analysis, or sampling form during inspection can help to focus the visual analysis and the sampling of mortars, ensuring some degree of objectivity and comparability between analyses and sampling practise (Figure 1).

Once the visual analysis and recording are complete, locations for sampling can be chosen. Visual analysis allows the specification of further, more complex, and laboratory based analyses that have specific sampling requirements. Visual analysis combined with an understanding of the building complexity is the basis of hypotheses formation about decay mechanisms and material compositions, that require testing by sampling and further analysis of mortar materials. The results of the building pre-analysis should be documented in a report. It can also be helpful to take photographs and to make sketches.

4. Practical sampling

4.1 Analytical method control on sampling

Hypothesis are tested by the analysis of materials. This requires a choice of analytical method to be made, which in turn determines the sampling requirements (see Table 3). This is because each different method of analysis requires a minimum quantity of sample and also a minimum quality of sample, either coherent lumps or powder. (A powder can be generated from a coherent lump, so coherent samples allow future changes in analytical requirements to be accommodated.) Good sampling flows from a careful visual analysis of the building and its materials and subsequently the clear definition of the aims that define which analytical techniques will provide information that will allow interpretations to be made and action taken.

Table 3, below, lists analytical methods commonly used for the characterisation of historic mortars, and the approximate *minimum* quantities of material each requires. In practice more sample is usually taken, but it is important to match possible with necessary sample quantity in order to establish which analytical techniques are possible. The sample quantities given in Table 3 are approximate and serve only to guide as specific requirements will vary. For example, Knöfel and Schubert (1993) increase the amount of sample required with increasing aggregate size and the information required from analysis. Based on Table 3 and the information given by the visual analyses, samples can be taken from the building. The visual analysis determines where the sample should be taken from and Table 3 describes *how* the samples can be taken and the minimum quantity of sample needed to perform the required analyses. It should be remembered that where sampling may be impossible in the future, and to minimise damage in the long term, a large amount of sample may be necessary, or at least as much as is possible. In addition to this, samples can also be used for more than one analysis where the earlier analysis does not result in its destruction or contamination. For example, samples tested for compressive strength through mechanical crushing could be used to determine binder : aggregate ratios or binder composition.

4.2 Representative sampling

It is not known to what extent sampling bias affects the conclusions reached in studies of historic mortars. It is certainly possible to devise strategies to take samples from a structure in a manner that will avoid bias and reflect the degree of variation in the characteristics of mortars (Cheeney, 1983). However, it is rarely possible for investigators of historic buildings to obtain enough material, due to the pressing needs of conservation philosophy which emphasise minimum intervention. Sample set sizes are small and sample locations are commonly highly constrained by concerns external to the strict characterisation of mortar materials.

Ensuring that the materials are representative of the structure is a difficult issue, especially when only a random sample of materials will return a true statistical measure. The close examination and detailed knowledge of the building construction, emphasised as essential above, may even mitigate against a representative sampling scheme, and introduce a form of bias. Nonetheless, visual non-destructive "sampling" in-situ before destructive sampling can identify the variations in macroscopic characteristics allowing choices to be made. This is still far from ideal, especially as no account can be made for the microscopic characteristics of structure and composition that are not visible. The precious nature of historic buildings is unlikely to ever allow the quantitative sampling of materials. Investigators must be aware of this and present conclusions firmly in this light, based as far as possible on the best suppositions possible with the quantity of sample permitted.

4.3 Sampling personnel

Ideally sampling should be done by those who are going to do the analysis on the samples once in the laboratory, and have been involved in the process of pre-analysis of the structure and in the selection of sampling positions. The best solution is for sampling to be done by individuals with experience of laboratory analytical techniques, even if they are not to conduct the analyses themselves. Samplers with this level of experience will understand the quality of sample required, and will generally avoid unsuitable material.

Where it is not possible to use experienced personnel, sample taking must be placed in a framework to guide inexperienced samplers in the recording of structured information to place the sample in some context. Information on location, sample characteristics, and method of sampling should be recorded. This approach attempts to ensure that future interpretations of the samples, that may be influenced by later discoveries of building complexity, can be accommodated. A standardised form can be completed during sampling, to record all the relevant information, and to prompt the sampler to seek out information when not immediately obvious (see Figure 1 for an example). The recording of in-situ sampling-relevant information like this is closely linked to stratigraphic or detailed surveys of building condition, and highlights the advantage of using the same personnel for both.

5. On-site activity

Once all the issues above have been considered sampling can begin. The variable parameters of practical on-site sampling are:

- Health and safety.
- The method (hammer and chisel, coring using power tools or by hand).
- People.
- Number of samples needed/allowed.
- Size of the samples required or allowed.
- Locations of samples and ease of access
- Information recording at the point of sampling.
- Sample labelling, storage and transport.

Despite the theoretical nature of the discussion above, visual pre-analysis and decisions about sample locations and sample quality and amount are practical on-site issues. If possible, the visual analysis of the building should be preceded by an historical investigation of the building, in co-operation with curators or historians. Working down from the visual analysis of the whole building and focussing on the mortar materials, decisions on representative sampling can be made within the constraints placed by architects and conservators.

Safe access to sampling locations is paramount. All safety regulations must be adhered to, especially regarding access on scaffolding and the use of tools if needed. Protective clothing, eye wear and face masks to protect individuals from dust generated during sampling should be available at all times.

Ample numbers of sample containers of sufficient size must be available in advance as should marker pens, tie-on labels, notebooks and prepared fill-in forms for the structured recording of information about the samples. Effort should be made to ensure the samples meet the quality and size criteria laid down in the earlier stages of hypothesis formulation and analytical method selection. Samples should be placed in bags and containers and clearly marked, if possible on the sample itself as long as this will not compromise analytical plans. Sample containers must be up to the job. Core samples can be delicate and will require suitable rigid boxes or similar, where as drill powder samples need at least a small plastic bag or bottle for storage. Sample positions should be accurately marked on a plan of the building, and photographs taken if possible of the location, before and after sampling. Collateral damage must be kept to a minimum.

The nature of the sampling method and its execution must be consistent with the intended analysis of the samples. For example, taking core samples allows the orientation of the sample on the structure to be known and the interrelationships of materials to be preserved with respect to the direction of sample taking. If the spatial distribution of soluble salts in the core was of interest then the sampling would have to be done dry, as water would wash away the materials of interest. Sampling to test moisture content must not be done with water, and the samples must be placed quickly in airtight containers to prevent drying out. These sort of practical issues should be foreseen in light of the analysis objectives and provision appropriately made.

Aside from the practical considerations of sampling, on-site difficulties are likely to include gaining the trust of colleagues from other disciplines working on the same project and entrusted with other aspects of the study or conservation of the structure. Analysts and practical samplers must work to minimise the impact of sampling and to convince others of the need for what can appear to be undue destruction of valuable cultural property. Samplers must listen carefully to their colleagues and respect their approach, but must also ensure that the samples they take are the best they can obtain for solving the problems that require answers.

6. Conclusions

The taking of samples of historic mortar for conservation and academic study appears in principle to be simple. However, the suitability of samples for the purposes of analysis is the foundation of the quality and further applicability of conclusions reached from a study. In the study of historic mortars the key issue is availability of samples. This is a problem because sampling historically important buildings goes against conservation philosophies of minimum intervention. However, the contemporary need for sampling and the power of analytical methods in many cases is no longer in question, despite continued progress in the development of non-destructive investigate techniques. The objectives of a study must be clearly stated before any sampling can take place. The building being sampled must be well understood to place samples within this context

The process of analysing mortars from an historic building can be summarised in the following steps:
1) Visual pre-analysis and inspection of building. Definition of the building construction phases or stratigraphy. Identification of materials used and their spatial and temporal interrelationships. Description of problems if any and formulation of hypotheses for diagnosis. Statement of analytical objectives and choice of analytical method/s. Selection of sampling locations and methods.
2) Practical sample taking. On site activity including information recording and removal of samples from the building, packaging and transportation to laboratory.
3) Analysis of samples in laboratory.

4) Conclusions. Interpretation of results and specification of remedial or repair actions, or statement of material characterisation and academic objectives fulfilled.

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8. References

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Table 1: Relationship between investigation aims and information required

Reason for Sampling	Possible Information Required
Conservation / Restoration: Matching, Analysis of decay (Causes: to seek solutions), Safety: Structural integrity	Kind of binder, aggregate/binder ratio, salt content, compressive strength, hardness, moisture content, mineralogy
Materials research: Composition, Durability, Mechanical/Physical behaviour	Major element and mineralogical phase conc., hardness, frost resistance, porosity, permeability, hydraulicity, compressive strength
Archaeology: Raw Material Sources, Production Technology, Construction Technology	Major and trace element conc., agg/binder, petrography/textures, All of the above…..

Table 2: Building Parameters

Significance of the building in historical, cultural or architectural terms. Structural aspects and construction technologies used. Failures and problems with materials. Intended work/conservation plan. Function/classification of mortar (original and current). Mortar composition and condition. Masonry materials. Aggregate size. Joint thickness. Previous repairs. Archaeology - building dates/phases. Construction method. Availability. Sensitivity: Cultural & architectural significance. Cost (staff needed for sampling, repair of sampling damage)

Table 3: Relationship between information sought, analytical technique and recommended quantity.

Information required	**Possible Analytical methods**	**Minimum QUANTITY and QUALITY of sample**
Appearance Qualitative structure/texture. Spatial relations of components. Binder/aggregate ratios	Optical examination Thin sections: optical microscopy Acid dissolution or thin section	In-situ or 10g (intact) ~10g (intact) ~100g (intact, crumbling or powder) depending on aggregate size.
Aggregate grading and colour.	Acid Dissolution and Sieving	~100g
Mineralogy/composition of aggregate and additives.	Thin sections (microscopy) XRD DTA TGA Scanning Electron Microscopy (SEM) IR Spectroscopy	~10g (intact, crumbling or powder.) ~2-5g (powder) ~1g (powder) ~1g (powder) ~2-5g (intact) ~1g (powder)
Mineralogy of carbonate binder.	Optical Microscopy XRD Scanning Electron Microscopy DTA TGA IR Spectroscopy	~10g (intact) ~2-5g (powder) ~2-5g (intact) ~1g (powder) ~1g (powder) ~1g (powder)
Microstructure of binder	Scanning Electron Microscopy (SEM)	~2-5g (intact)
Composition	EDX and Electron Probe analysis	5g (intact)
Organic materials	IR Spectroscopy	~1g (powder)
Major and Trace Elemental concentrations	Atomic Absorption Spectrophotometry ICP X-ray Fluorescence EDX and Electron Probe analysis	5 g (powder) 1g (powder) 10g (powder) 5g (intact)
Anions/cations concentration Salt content.	Ion Chromatography	1g (powder)
Porosity	Mercury intrusion porosimetry Density and water saturation method	10g (intact) 10g (intact)
Free/uncombined moisture content	Moisture content	10g (powder, or intact samples)
Permeability	Surface probe permeability Laboratory permeability	100g intact or in-situ Core sample intact lump
Elasticity	Indirect tensile and bending tests	>100g intact Core samples or intact joint sample.
Bond strength, tensile bending strength of mortar/masonry join.		>100g Including mortar – masonry join. Core possible.
Durability of material	Hardness/ Schmidt hammer test Freeze thaw and salt crystallisation	In-situ or intact 100g pieces. Core or joint sample Large >200g intact samples- chisel, saw or cores.
Compressive strength		100g core samples or intact joint sample.

Figure 1: Examples of possible information recording sheets to be used during sampling on site.

Building:

Address:

Sample No:
Sampled by:
Date:
Reason for sampling:

Location on building

Façade ☐ Interior ☐

Description of location:

Phase:

Feature:

Depth in wall:

Photograph:

Mortar Description

Colour:

Binder:
☐ Lime ☐ Cement
☐ Lime & Pozzolan ☐ Gypsum

Function:
☐ Bedding ☐ Pointing
☐ Grout ☐ Render
☐ Plaster ☐ Repair

Aggregate:
☐ Sand ☐ Crushed rock
☐ Shell

Condition (decay, salts etc.):

Associated building materials:

Building: Address:	Sample No: Sampled by: Date: Reason for sampling:

Aim of Study:

 Conservation Restoration
 Archaeology Material research
 Repair
 Other, please specify:

Sampling method:

 Hammer & chisel Drill core
 Other, please specify:

Analyses proposed:

 Binder/aggregate dissolution Thin section
 XRD Chemical
 Physical tests
 Other, please specify: