

Final Report for the Research and Development Project

Non-Destructive Field Tests in Stone Conservation

Literature Study

Rapport från Riksantikvarieämbetet 2006:3



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1. Objectives and Project Description

1.1 Objectives of the Project

For several years the National Heritage Board (NHB) in Sweden has striven to monitor and evaluate stone conservation treatments. One of the reasons is that conservation methods that are often used in Sweden sometimes fail. Another reason is that such evaluations will make it possible to recognize when the stone needs treating again. Above all, the evaluations may help to reject poor and disputable methods and thus improve conservation.

In previous evaluations conducted by the NHB, the methodology was first and foremost based on visual and tactile methods. Quantitative methods were also used, such as the Drill Hardness Meter and the Karsten pipes. Observations and measurements were compared with information from conservation reports. This methodology does have certain weaknesses, however. For example, information and data relating to the condition of the stone during and shortly after conservation is often lacking. That information is necessary to an understanding of *whether* and *why* conservation has failed.

The purpose of this project is thus to improve the evaluation of stone conservation in Sweden using NDT methods that provide comparable quantitative data. In order to achieve this goal it will be necessary to adjust the conservation process by putting a greater emphasis on pre-investigations and documentation. The pre-investigation phase should include a selection of areas of "zero values" using NDT methods. Each NDT method naturally has advantages as well as drawbacks, but these are not always explained in the literature. The NDT methods used in this project have been evaluated in an attempt to discover more about their possible uses.

The project is divided in two stages, or steps;

1. *A literature study* based on conference articles and conservation literature and complemented by interviews.
2. *Laboratory and field-studies*. A number of chosen NDT methods were tested and evaluated. These methods are available in Sweden and easy to use *in-situ*. The tests were conducted on Gotland sandstone – one of the most frequently conserved stones in Sweden.

The ultimate goal of this work is to create a manual with instructions as to what kind of information is required before, during and after conservation (and how it should be collected). It is hoped that such a methodological manual will be used as a matter of routine within the conservation

process, particularly as it will facilitate an assessment as to whether the conservation was both successful and durable. This ultimate ambition is, unfortunately, beyond the scope of this project as both more research and an implementation strategy managed by the NHB are necessary. However, a preliminary manual has been created.

1.2 Summary of Literature Study

The overall aim of Step 1 – the literature study – is to assess which NDT field methods can be used in stone conservation in Sweden. The main goal is to provide a preliminary overview of NDT methods available to the stone conservator-restorer [1]. In this study it is not possible to provide a complete and comprehensive technical and scientific explanation of all the methods. Such information can be found in conference publications, conservation and scientific literature and by consulting scientists and stone conservators. The literature study provided here is quite separate from the field-test programme and the laboratory test programme of selected NDT methods (see Step 2, below). The advantages and disadvantages of the various methods are discussed – especially from the Swedish perspective. It should be pointed out that drawbacks of the methods have not always been discussed in the study. This doesn't necessarily imply that the method is faultless, only that the literature (within this study) either hasn't yielded such information or that the information provided is not sufficient. A specific example of this concerns the question of moisture measurement. While some of the NDT methods for measuring moisture are described as being very successful, the articles give conflicting information about the methods. A few methods do seem to have potential, however, such as the portable NMR instrument, the infrared camera and the microwave instruments.

The first chapter describes the project and its background, the second outlines the current situation – in terms of dealing with evaluations of stone conservation – and the third indicates the current state of the art of NDT conservation methods. The fourth chapter, which forms the core of the report, focuses on each specific NDT method. The sections in the fourth chapter are divided according to specific conservation questions, such as the measurement of surface roughness and weathering of the stone, and measurement of water absorption. Each question is briefly discussed and each NDT method described. Examples of each method's use in conservation are also included. At the end of each

section a table has been included to help the reader orient him/herself. In order to encourage further reading, a bibliography has been included (Appendix 2).

It has been difficult to find sufficient information about several of the NDT methods in the conference articles. The methods are rarely explained in detail in the articles and the advantages and problems associated with the method are seldom discussed. It is therefore difficult to assess the value of the method. Many articles are very positive about the results – even though there might be serious drawbacks with the method. For example, the methods may be labour intensive or inaccurate and difficult to use *in-situ*. An illustrious example is the measurement of surface roughness, where profilometric methods have often been used and the results presented as mere facts. However, when the methods were tested by Grissom and co-workers, they found that simple visual and tactile examinations gave more accurate results than the instruments (see below). One might therefore wonder whether the profilometric methods are of any value at all in stone conservation. The lack of clarification regarding the limitations of the methods thus makes it difficult to achieve a complete overview of the situation through the literature. In order to decide which method is best suited to a particular situation it is necessary to evaluate the methods practically and make comparisons.

1.3 Project Background

Between 1989 and 1995 the Swedish NHB managed and directed the ambitious "Air Pollution and the Cultural Heritage" programme. This programme resulted in a nationwide inventory of building stones from early medieval times until the 1940s (published in the Series of Swedish Building Stones) and in a database containing information about architect, the building's age, stone type and state of preservation. Moreover, the programme led to a greater awareness of stone deterioration which resulted in a tremendous amount of new research projects and further development of stone conservation methods in Sweden. As a consequence of this upsurge of activity and interest the stone conservation field improved and a lot of stone treatments were carried out. Since the programme came to an end in 1995, few attempts have been made to evaluate the result of all this effort. This particular study project is a small part of this important work.

1.4 Description of the Project

The project is divided in two parts; *Step 1* and *Step 2*.

2.3.1 Step 1

A literature study of NDT methods used in stone conservation and discussions with experienced scientists and conservator-restorers in Sweden and abroad. The aim is to learn

more about NDT methods and to establish their key advantages and disadvantages.

The methods studied include:

- Methods for measuring the relief/roughness of the stone's surface, such as profilometric stylus measurements and laser scanner methods.
- Methods for measuring the water-soluble salt content in the stone, e.g. the Löfvendahl method.
- Methods for measuring water absorption, e.g. the Karsten pipe, the Mirowski pipe and the Italian contact sponge test.
- Methods for investigating the stone's inner structure, e.g. the tomographic methods.
- Methods for analyzing substances on the surface of the materials, e.g. a portable FT-IR.
- Methods for measuring the strength and mechanical properties of the stone, i.e. a method that gives information about the condition of the stone, e.g. ultrasonic measurements and micro drill resistance (which is destructive).
- Methods for measuring the stone's moisture content, e.g. the microwave method.
- Methods for measuring colour changes, e.g. the Spectrophotometer.

In addition to these methods, aspects of stone conservation theory, practice and use are also discussed.

2.3.2 Step 2

The *in-situ* testing of four NDT methods on the Gotland sandstone of fourteen buildings in Stockholm. Gotland sandstone was also tested in the laboratory. This step included a visit to Gotland and some of the open and closed quarries. The individual parts are:

- 1 *Variation of Ultrasound Pulse Velocity (UPV) due to changes in the relative humidity of Gotland sandstone.* A laboratory test programme designed to analyse how the UPV changes in relation to the relative humidity and water saturation of Gotland sandstone. The aim of this study is to analyse the stone material in laboratory conditions and compare this with observations made in the field. [5] The tests were conducted by Dr. Katarina Malaga at SP; the Swedish National Testing and Research Institute located in Borås. This programme was initiated in July 2005 and concluded in June 2006.
- 2 *Variation in colorimetric measurements on Gotland sandstone taken with a Minolta Spectrophotometer due to heat, cold and moisture content.* Testing of the variations of colorimetric measurements with the Minolta camera was undertaken in different climatic conditions. The tests were conducted in the NHB's stone atelier in February 2006.
- 3 *Measurement of the w- and B-value of Gotland sandstone from the Valar quarry.* The aim was to learn more about the properties of Gotland sandstone. The tests were made

with the German capillary suction standard test DIN 52 617 and conducted March 2006 in the NHB's stone atelier.

4 *Field test programme of four NDT methods used in stone conservation on Gotland sandstone objects in Stockholm.* The methods were:

- a. Measurement of water absorption using the Karsten pipe,
- b. Ultrasonic Pulse Velocity (UPV) measurements (conducted by Dr. Katarina Malaga, SP),
- c. A granular disintegration test with a "tape test" using Herma labels (invented by the NHB).
- d. Measurements of the colour of the stone with a Minolta camera.

The methods were tested on three occasions over the period of a year and during different climatic conditions (in August and October 2005 and in May 2006) in order to register variations in temperature and moisture. Nineteen stone objects on fourteen buildings in Stockholm were chosen (see Chapter 7.3). The criteria for choosing the objects were:

- Building accessibility
- The age of the stone (ranging from the 16th to the 20th centuries)
- Knowledge of the conservation history of the stone (for this purpose several art historians, conservators and architects were consulted).

5 A literature study of the geology, deterioration and conservation of Gotland sandstone has been undertaken. Furthermore, archives, reports and articles have been scrutinised in order to find out more about the history of stone conservation – especially on Gotland sandstone – in Sweden. The aim of this study has been to understand what may have happened to the stone.

1.5 Scientists Consulted in this Study

Several well-known conservation scientists were contacted using email and the telephone in an attempt to find out more about NDT practice in stone conservation in other European countries, (see appendix 1 for the list of people contacted). Moreover, a visit was made to one of the three governmental research institutes on the conservation and restoration of

works of art in Florence, Italy (ICVBC-CNR). The contact at the institute was Dr. Susanna Bracci, who kindly handed over the new Sponge test elaborated by Dr. Piero Tiano.

1.6 Description of the Literature Study

The literature study comprises a review of periodicals, conference publications and database searches. As all the necessary publications were not available in Sweden, the greatly appreciated assistance of the Library of the Royal Academy of Letters, History and Antiquities has ensured that the search has been as systematic as possible. The used media are:

- 1 Databases and Internet resources: AATA and the BCIN.
- 2 Evaluations and reports conducted by the Stone Department at NHB.
- 3 Periodicals: *Studies in Conservation*, *Bautenschutz & Bausanierung*, *APT Bulletin*, *Restauro*, *Journal of Cultural Heritage*, *Reviews in Conservation*, and *JAIC Journal of American Institute of Conservation*. [3]
- 4 Conference publications: *International Congress of the Deterioration and Conservation of Stone*, *ICOM Committee for Conservation's Triennial Meetings*, *Compatible Materials Recommendations for the Preservation of European Cultural Heritage (PACT)*, *International Symposium on the Conservation of Monuments in the Mediterranean* (only the 1994 and 1997 Conferences are available in Sweden), *International Conference Non Destructive Test on Works of Art* (the library at NHB has only one proceeding from 1994), *Structural Studies, Repairs and Maintenance of Heritage Architecture* (not available in Sweden; some articles were ordered from abroad), *Programme Franco-Allemand de Recherche pour la Conservation des Monuments Historiques*, the SWAPNET's (*Stone Weathering and Atmospheric Pollution Network*) and *Euromarble's* publications. Other publications from Conferences and stone conservation in general such as the *RILEM* publications (for example the *Conservation of Stone and other Materials* from 1993) and the *Getty Conservation Institute's* publications, *Bayrisches Landesamt für Denkmalpflege's* publications, and the *Conference on Science, Technology and the European Heritage* (1991).

2. Evaluation and Stone Conservation

2.1 Evaluation of Stone Conservation in an International Context

In his report on the state of the art in stone conservation from 1996, the scientific conservator Clifford A. Price has divided the evaluation of stone conservation into two categories: 1) those that characterize the stone shortly after treatment has taken place, and 2) those that are concerned primarily with the monitoring of long-term performance. [4] These test methods usually measure those properties in the stone that change when it deteriorates. Such properties include the surface hardness, the strength, the ultrasonic pulse velocity and the acoustic emission. In most of the examples found in the articles, the evaluations have been conducted in the laboratory and analyse how conservation reacts on fresh stone. One typical example where problems are discussed is in Villegas, Vale and Bello's evaluation of consolidants and surface hardness with ultrasonic measurements on stones used in Andalusian cathedrals in Spain. [5] One important factor is that fresh stone is different to old stone that has been subjected to weathering and conservation treatments.

Evaluations are sometimes performed on old stone that has been conserved using destructive methods. An interesting example of this is the study of the durability of the hydrophobic treatment of sandstone facades of the Alte Pinakotek and Schillingfürst Castle in Bavaria on different occasions between 1984 and 2001. The evaluation was conducted by the Bavarian State Department's Historic Building's laboratory. The methodology was found to be satisfactory – even though the methodology was both destructive (core samples were taken and tested in the laboratory), and non destructive (Karsten pipes were used *in-situ*). The results of the programme proved that the laboratory and field evaluations could be correlated. [6] It therefore appears that the Karsten measurements are good indicators of the durability of the treatment.

Another interesting example of an extensive evaluation is the evaluation of stone sculptures and monuments treated over the last twenty years in Austria (evaluated between 2000 and 2002 by Nimmrichter and co-workers). The evaluation consists of both oral information and testing the objects; both using NDT methods (Karsten, UPV, drill resistance, electrical conductivity, knocking by hand, colour description and so forth) and some sampling. The results were quantified: in 55 percent of the conservations the long-

term effect of the conservation was good, and in 10 percent of the cases the conservation treatments had actually caused new deterioration. Moreover, Nimmrichter and co-workers pointed out that the previous conservation reports had not been sufficiently systematic and lacked the necessary data. Finally they concluded that more scientific pre-work and controlling with special analyses (such as UPV) during the conservation is important. [7]

Of late more *in-situ* evaluations of previous conservations have been included in the publications. This is quite a natural development since the field of stone conservation has grown considerably during the second half of the 20th century. An evaluation of what has been done is therefore timely. As we have seen in the Austrian case, the evaluations expose certain difficulties as all the parameters that can cause damage are not known. The reasons for this are that the conservation documentation has not always provided sufficient data and also that the conservator-restorers very seldom leave untreated reference surfaces. Despite this there are recent examples of successful evaluations, such as the evaluation of the "Bologna Cocktail" [8] and the conservation of the four Virtues in Porta dell Carta in the Ducal Palace in Venice. [9] One other particularly interesting example is the testing of the consolidation of Brethane™ in Great Britain, where untreated samples were evaluated together with the treated samples (see below). [10]

The conservation scientist Marisa Laurenzi Tabasso, frequently involved with the evaluation of stone conservation, has compiled a list of some of the stone conservation evaluations. The list includes various monuments in Bologna (1985), Clemenswerth Castle in Lower Saxony (1996) Lausanne Cathedral (2000), the Lunette of St. John the Evangelist in Venice (2000), the Loggia Cornaro in Padua (2002), Chartres Cathedral in France (2004), and Seven Baroque Churches in Lecce (2004). She concludes that these evaluations demonstrate the difficulty of estimating the actual durability of the treatments. The assessment would be easier if the conservator-restorers had also made provision for a reference area after the conservation; *a zero point*, that could be regularly monitored to detect any changes. She suggests a methodology for this purpose – using NDT methods – that measures "surface colour by reflectance spectrophotometry, water absorption under low pressure (Karsten pipe), amount of deposited dust per unit surface, amount of water-soluble salts (extracted using Japanese paper poultices

wetted with deionized water), surface roughness (using a portable rugosimeter), and biological contamination.” [11] This methodology is a good starting-point for this project. Tabasso moreover noted that despite considerable development in the field of conservation the crucial question in conservation is still whether “the material parameters currently in use are suitable for judging conservation treatments, and is it possible to determine treatment durability?” [11] This was the question she posed during the Dalhem workshop in 1996 and was still valid in 2004. All the participants at the Dalhem conference maintained that “there was a lack of professionalism within the context in which conservation measures and treatments are planned and implemented and that there was no defined quality control.” [11]

2.2 Previous Evaluations Conducted by the NHB in Sweden

Previous evaluations conducted by the NHB focused on stone conservation treatments that characterize the stone (mainly Gotland sandstone) shortly after treatment has taken place. However, some studies aimed at monitoring the long-term effect of weathering and pollution on Swedish stone have also been conducted by the *Swedish Corrosion Institute* as well as within the framework of the *EU-marble project*. [12]

The first evaluation was executed during 1995 – 1996 by the conservator-restorer Misa Asp, the geologist Runo Löfvendahl and the engineer Erik Österlund. This evaluation was based on a survey directed at Swedish stone conservator-restorers and involved an *in-situ* examination of eleven stone objects conserved between 1988 and 1995 under the stewardship of NHB. The examination was both destructive and non-destructive and consisted of salt measurements both directly on the surface of the objects and from core samples (Ø18 mm) according to the Löfvendahl and Asp method, measurements of the moisture content (conductivity) on the stone surface with a Protimeter and of the moisture content in the core samples (by weight before and after drying), and finally using a Durabo Drill Hardness Meter (DHM) to try to measure the hardness of the stone. Visual and hands-on inspections were also carried out. The visual observation results were noted on an evaluation leaflet and mapped on drawings. In some places Karsten pipes were used to evaluate the water absorption, although this was not conducted systematically. The report mentions problems encountered in analyzing the DHM which meant that the results could not be presented. [13] The results of the tests confirmed that there were often problems with rising damp, colour changes on the restoration mortars, and that consolidations with Wacker OH were sometimes efficient and sometimes not. Moreover, the authors state that both the hydrophobic treatment and painting of stone for protection needed to be further explored, and that the mending mortars also required more investigation and test-

ing (both these areas have been and continue to be explored by the NHB). [14] In 1993, a more systematic testing of the Karsten pipes was conducted by Erik Österlund and Misa Asp. This study resulted in a report “*Karstens mätrör som oförstörande provmetod på sten*” (see below).

In 2003 the conservator-restorer Dr. Agnetha Freccero completed a survey of the evaluation that had been conducted by the NHB. She found that 245 conservation works had been carried out and that 60 of these had been evaluated. The evaluations were different in character, both in methodology and form. Freccero noted that this inconsistency made it difficult to gain any clear view of the situation. She stated that both the conservation documentation and the sampling methodology varied too much and that the evaluations in themselves also varied. Freccero therefore concluded that in the future it would be necessary to establish a system for evaluation that shared a common terminology. [15]

The NHB has noted that it is hard to find sufficient information to make any satisfactory conclusions about the previous conservation of stone in Sweden. Even though there has been a profound emphasis and significant improvement in conservation documentation, the available information is in summary form. For example, there is a lack of detailed information relating to previous treatments and data revealing the condition of the stone before and after treatment, such as water absorption measured with Karsten pipes, colour measurements and so forth. Moreover, the conservator-restorer has not always mapped the conservation and restoration treatments in detail and important information regarding climatic conditions during treatment is often missing.

2.3 Standardization, Evaluation and Conservation

In addition to the development of NDT methods, there is an increased need for the standardization of tests methods in conservation. It would, for example, be much easier to compare the evaluation results in different parts of the world if the test methods were the same. This need (together with other conservation issues) initiated a European collaboration. The Swedish standardization body, SIS, is currently involved in the *European Committee for Standardization in the Conservation of Cultural Property (CEN/TC 346)* which consists of five working groups. The group *CEN/TC346/WG3 Evaluation of methods and products for conservation* is led by the Italian conservation scientist Vasco Fassina. Unfortunately, not all the European countries are involved at present – for example Germany and Great Britain are noticeably absent (i.e. at the end of 2005). The first meeting was held in Venice in June 2004.

The scope of the standardization group is to normalize definitions, terminology, the methods of testing and analysis, support the characterization of materials and deteriora-

tion processes of movable and immovable heritage and the products and technologies used for the planning and execution of their conservation, restoration, repair and maintenance. The groups are as follows:

- CEN/TC 346/WG 1 – General guidelines and terminology.
- CEN/TC 346/WG 2 – Materials constituting the artefacts.
- CEN/TC 346/WG 3 – Evaluation of methods and products for conservation works.
- CEN/TC 346/WG 4 – Environment.
- CEN/TC 346/WG 5 – Transportation and packaging methods.

The work of the third group corresponds closely to the project represented in this paper. Aided by three Swedish experts, Dr. Daniel Kwiatowski, Skanska, Dr. Katarina Malaga, SP and conservator Misa Asp, NHB., the group aims to develop standard methods of evaluation. The work is still in its initial stages and no standards have yet been accepted. Nevertheless, some of the tests scrutinized within this report, such as the Karsten pipe, may be adopted as CEN standards. The existing *EN Natural Stone Standards Methods*, often equivalent to other national methods, will be used as a working base for the CEN/TC 346.

It is notable that the Italians are very active within the standardization group; perhaps because already at the end of the 1970s they had started to develop standardized methods for stone conservation within the **NORMAL** committee (**NOR**mative **MA**teriali **L**apidei e.g. the committee at first only worked with stone). It was established by the **CNR** (National Research Council) and the Ministry of Cultural Property. The first recommendations were for laboratory studies, although in the 1980s, the first standard tests for the *in-situ* cleaning of porous inorganic materials were conducted. [16] The **NORMAL** standards have now been transferred to the **UNI** (*Ente Nazionale Italiano di Unificazione*) within *La Commissione Beni Culturali UNI-NorMaL*. So far 44 standard methods have been published. Most of them specify analytical methods for porous inorganic materials, others normalize the terminology, others set up the criteria which the conservator has to adopt during different steps in the diagnostic process, such as how to take samples, plan, manage and evaluate conservation treatments and how to perform the graphic documentation and describe the weathering phenomena. Here are some examples: **Normal 11/82** and **Normal 11/85** substituted by **UNI 10859**; *Assorbimento d'Acqua per Capillarità* – *Coefficiente di Assorbimento Capillare* (Capillary Water Absorption – Coefficient of Capillary Absorption) and the **Normal 44/93** *Assorbimento d'acqua a bassa pressione* (low-pressure water adsorption), **Normal 12/83** *Aggregati Artificiali di Clasti e Matrice Legante non Argillosa: Schema di Descrizione*, (Artificial

and Clastic Aggregates and Cement (not in clay): Description Procedure), **Normal 13/83** substituted by **UNI 11087** *Dosaggio dei Sali Solubili* (dosage of soluble salts), **Normal 16/84** *Caratterizzazione di Materiali Lapidei in Opera e del loro Stato di Conservazione: Sequenza Analitica* (Characterization of stone materials in art and their state of conservation. Analytical sequences) and **Normal 22/86** *Misura della Velocità di Propagazione del Suono* (Measurements of the ultrasonic velocity). [17] There are moreover **Normal** standards concerning NDT methods such as **Normal 42/93** *Criteri generali per l'applicazione delle PnD* (General criteria for the application of non-destructive tests) and **Normal 43/93** *Misure colorimetriche di superfici opache* (Colorimetric measurements of opaque surfaces).

Naturally other national and international standards are also used in the field – although they are mainly used for engineering purposes. The Germans have developed DIN standards that are frequently used in conservation, however, such as **DIN 52 617** *Determination of the water coefficient of building materials* (w- and B-values), **DIN 18 550 Part 1**; *Kennzeichnung der Kapillareigenschaften von Putzen*, characteristics of the capillary properties in renders; **DIN 52 615** that is “the wet cup test”, *the value of water vapour diffusion resistance*, and **DIN 52 103** that measures *the water absorption capacity under both normal and vacuum pressures*. Moreover, tests concerning the visual appearance include **DIN 5033 part 3**, **DIN 55981** and **DIN 6174** – the latter using the CIE-LAB standards. There are also tests relating to surface roughness, e.g. **DIN 4760 ff**, **DIN 4768** and **DIN 4770 ff**. [18] In addition, RILEM elaborated standards for construction and engineering purposes in construction are also used in conservation. Some of the European RILEM research groups are working in related areas, for instance the **TC ACD: Acoustic emission and related NDE techniques for crack detection and damage evaluation in concrete**. Within the RILEM, the **RILEM 78 PEM** (Protection et Erosion de Monuments) working group is no longer active (its main purpose being to discuss cleaning methods during the 1970s). [19]

For a long time now the Americans have been involved with the discussions on standardization and have developed their own conservation standards. For example, within the ASTM sub-committee **ASTM E6.24** “Building Preservation and Rehabilitation Technology” some standard methods have been developed since the end of the 1970s. [19] Some of the ASTM standards concern the ultrasonic velocity on core samples of stone for the laboratory, such as **ASTM 597** and **ASTM D 2845–83**. [20]

Naturally other European countries also use their own standards. It is difficult to become conversant with all the standards, however, and it is hoped that future standardizations will benefit conservation practice and make it much easier to compare different studies.

2.4 Tactile and Visual Assessment

Before embarking upon the more high-tech NDT methods, it is appropriate to discuss the most unpretentious NDT methods, namely, visual (sometimes aided by photography or microscopy) and tactile methods. While these methods are valid in many situations, one serious drawback is that the results depend on the observer's experience. One obvious example is colour change. But how is it possible to objectively describe colour change? In a reliance on less subjective tools, conservator-restorers have always made use of scientific aids such as colour charts and examinations with ultraviolet and infrared light. It is important to remember, however, that many of these methods also depend on the experience of the viewer – even though they may help the human eye to discriminate the phenomena. Nowadays these techniques are combined with sophisticated detectors, use of computer-aided interpretation, modelling and analysis of the data and advanced software to display the data. This creates enormous possibilities as well as some problems. The data always needs human interpretation and the advanced data display can "lie". It is possible that the nicely packaged information will make it appear more scientific than it actually is.

Despite this, the naked eye and human touch are still valid methods in many cases. One example is the evaluation of 18 years of consolidation with alcoxysilane Brethane™ in Great Britain. For this purpose, English Heritage designed a methodology based on tactile and visual inspection. The methodology comprised an area of treated stonework which was then compared with an adjacent untreated area that was assessed visually and through touch against a set of pre-established criteria. [10] One drawback is that this methodology can make it difficult to compare the data with other studies, since the results are based on the interpretation of an individual person. For example, each person has to have the same understanding as to what is meant by a "slight amount of powder transferred to finger drawn across surface" in comparison to "substantial amount of powder transferred to finger drawn across surface". Moreover if the conservation fails, this methodology doesn't give any information or clues as to what might have gone wrong. In some cases, however, even scientists prefer the tactile assessment. One example

is Grissom and co-workers who scientifically examined the accuracy of stylus profilometry, reflected-light computer image and micro drop water-absorption time measurement, and came to the conclusion that "touch evaluation was the most successful method" (see below). [21, 22]

2.5 Mapping Methodology in Conservation

Whenever conservation or an analytical programme is planned, the gathered information needs to be organized in some way. Systematizing the documentation before, during and after conservation helps the conservator-restorer to analyze the information. Systems have been developed to map damage and forms of weathering on drawings or photographs, and there is a constant search for less subjective ways of observing and describing damage. The search has ended in standardization of the terminology. Several methods for mapping the condition of stones have also been developed – especially at the end of the 1980s. One example of this is the damage atlas, such as the damage atlas for brick construction (created by the European Commission in 1998). [23] In 2005, several projects dealing with documentation and terminology were up and running, such as the *Record DIM* at Getty's Conservation Institute, the CEN standardization group within EU countries (see above) and the *Internet accessible multilingual illustrated Glossary on stone deterioration* set up by ICOMOS. [24][25]

The Fitzner and Kowanatzki methodology, presented during the 1990s, is probably the best known in stone conservation. [26] The NHB followed suit and created a simplified documentation methodology. [27] One interesting example of how the Fitzner and Kowanatzki methodology can be used is the evaluation of stone cleaning by Ball and Young in Scotland. They created a survey system for evaluating the efficiency and effect of cleaned buildings in Scotland. The objective was to facilitate predictions on the effect of stone cleaning, i.e. to understand how fast the stone would be re-colonized by biological growth and re-soiled. The methodology comprised a comparison between cleaned and non-cleaned buildings in Scotland, using visual examination, colorimetric measurements and interpretations on rectified photographs according to the Fitzner and Kowanatzki weathering forms. [26]

3. NDT Methods in Conservation

3.1 Conservation and NDT Methods

In conservation, scientific analytical methods are used to evaluate both the materials (such as stone type) and effects of the conservation and weathering processes. K. Janssens and R. van Grieken have divided the analytical methods used in conservation into three groups. All the areas have been studied and are as follows:

- The chemical nature/composition of selected parts of cultural heritage artefacts and materials.
- The state of alteration (on the surface and/or internally) of objects as the result of short, medium and long-term exposure to particular environmental conditions.
- The effect/effectiveness of conservation/restoration strategies during and after application. [28]

There are many different methods to choose between depending on the aim of the analysis. It is obvious that it isn't possible to obtain all the necessary information from one single analytical method. The conservator-restorer therefore has to design a test series that gives complementary information. That isn't always sufficient, though, and further requirements are often necessary, such as tests should be *non destructive, fast, universal, economic, reproducible, easy to use, objective, available, sensitive and harmless to the environment*. As mentioned above, not all tests correspond to these requirements in that some are micro destructive (such as micro drilling resistance), very expensive or demand experienced personnel. The criteria must therefore be seen as an optimal aspiration and the conservator-restorer should keep these requirements in mind when designing an analytical programme.

This project deals with NDT methods. The search for NDT methods has been ongoing since the onset of conservation; the reason being that the conservator-restorer always strives towards preventing any damage to the objects occurring. The more sophisticated NDT methods are usually developed for engineering or medical purposes and after a while adopted and modified for conservation. Thanks to this it is nowadays possible to understand, characterize and evaluate conservation work without taking samples. The NDT methods are increasingly gaining in relevance and many articles have been published within the field. A survey conducted in the LABSTECH organization (published in 2004 – see below) demonstrated that NDT and micro destructive tests are not yet common among conservator-

restorers (100 conservator-restorers were asked about their work and 32 answered. Only a few of them used NDT methods). [29][30][31] The reason for this is often a lack of equipment, experience and routines.

The NDT methods are based on different physical phenomena and are usually divided into different groups depending on their scientific background:

- Geophysical methods; measure mechanical and electrical properties of the material.
- Spectral analytical methods; analyze properties in the surface by the use of electromagnetic radiation that is absorbed or emitted by the material.
- Tactile and visual assessment.

The first two are studied in the following chapter and the third is discussed in brief in this chapter. Katinka Klingberg Annertz divides the NDT methods into three groups, depending on what the method is able to do with the material:

- Geophysical methods that investigate the bulk of the material (seismic methods such as ultra sonic methods, hammer methods, acoustic emission methods and radar methods).
- Spectroscopical and chemical methods that investigate the surface of the material (absorption spectroscopy, diffusion spectroscopy, emission spectroscopy and radio chemical methods).
- Imaging techniques that investigate the bulk and/or surface of the material (laser scanning, analytical photography/reflectography, thermography, radiography, Computer Tomography and fotogrammetry). [32]

Some of these methods are discussed in this report. A few of these methods are expensive and difficult to use *in-situ* and are therefore discussed in brief.

Anders Bodare (1996) has divided the NDT methods used within the realm of geophysics into two types depending on the kind of wave being used.

- *seismic methods*, such as ultrasonic methods, the Schmid hammer method and acoustic emission methods.
- *electrical methods*, such as radar, resistive and electromagnetic methods. [33]

In this report, the methods are divided according to what kind of properties the methods help to analyze, such as moisture or surface roughness (see chapter 4).

3.2 NDT and Stone Conservation

In stone conservation, the use of NDT is restricted by the fact that the stone objects are often found in buildings. Movable objects are, on the other hand, possible to take into the laboratory (where several NDT techniques have been developed). In architectural stone conservation, the NDT methods must be portable and able to use in the field in a variety of conditions (such as on scaffolding in bad weather).

Conservation publications usually contain articles that describe both the use of NDT and destructive methods for the characterization of stone and the evaluation of conservation. The early techniques usually dealt with radiation of different wavelengths, i.e. they are kinds of absorption spectroscopy, such as ultraviolet or infrared light analysis. These still are the most common methods, although nowadays the analysis is made with the help of analytical tools and computer modelling.

The importance of the NDT in conservation is demonstrated by the number of conferences in the area. The first one, *International Conference Non-Destructive Testing of Works of Art*, was held in Rome in 1983. At the 4th Conference, which took place in Berlin in 1994, the methods were divided into groups: *radiography, Computerized Tomography, optical measurement, X-ray fluorescence analysis, measurement of environmental influence, X-Rays and computed tomography, optics, image processing, analysis, building, climate and environment and modelling* among others. Another ongoing Conference is the *non-destructive testing and microanalysis for the diagnostics and conservation of the cultural and environmental Heritage*; the last one being held between 15–19 May 2005, and moreover the *Non-destructive Testing to Evaluate Damage due to Environmental Effects on Historic Monuments*. Besides these, numerous periodicals and literature on NDT are available for scientists and engineers alike. Most of the methods deal with museum objects that are analyzed indoors in a laboratory.

The advancement of this trend is dependent on improvements made in detector technology, instrument-computer interfacing, focusing optics, and radiation sources suitable for use in various parts of the electromagnetic spectrum. For methods that need to be used *in-situ* there has also been an immense improvement in the miniaturization of components, making designs more compact, and the development of portable and handheld instruments. [28]

3.3 Problems to be Analyzed in Building Stone Conservation

We have already mentioned that some problems are characteristic to the conservation of building stones due to the fact that they are situated outdoors and are often part of a large structure. The investigation and conservation of building stone is therefore determined by these circumstances. If the conditions are too harsh, the object has to be moved indoors

(although this is against conservation ethics). Some of the questions that are necessary to understand include:

- The water absorption, the water content and the source of the water.
- If salts are present, what kind, their distribution, source and quantity.
- Climatic conditions that effect the weathering, such as air pollution and variations in humidity and temperature.
- The condition of the stone, for example the weathering degree and the rate of deterioration.

Several NDT methods are available for this purpose. They are not always applicable to the whole picture, however, and in some cases sampling is required. Moreover, knowledge of a number of facts that can be monitored and controlled with NDT methods is also necessary during and after the conservation:

- Changes in colour (with colorimetric measurements).
- Changes in strength and hardness (ultrasonic, micro drilling resistance and so forth).
- Whether water is present in the stone and where it comes from (moisture measurements).
- Loss of material (surface relief or roughness measurements).
- Changes in the stone's water absorption (pipe methods).
- Whether salts are present (measurements of salt extractions with paper pulp).
- Durability of conservation treatments (a mixture of methods mentioned above).

These questions are discussed in this report when dealing with the NDT methods.

3.4 NDT Methods and Stone Conservation in Sweden

All the NDT evaluation methods are not always used in stone conservation in Sweden. The conservators-restorers usually only use salt compresses to determine whether salts are present and to control their proper extraction. Colorimetric measurements and Karsten pipes methods have been performed by the NHB in different projects. The results of these measurements have not yet been fully evaluated. One exception is the Österlund report (from 1993) that evaluates several Karsten pipe measurements (he also tried Mirowski pipes) conducted by the NHB in the laboratory and in the field. Österlund moreover calculated the w- and B-values and found the calculation model was too sensitive. Small changes in the measured data distorted the values too much and he therefore developed his own simplified mathematical model. [14] In this study the w- and B-values are calculated since they are commonly accepted in the European context (see below).

In her licentiate thesis, Myrin (2004) used some evaluation methods to investigate the conservation of Gotland sandstone and described the situation prevalent in stone

conservation in Sweden. The main part of her thesis consists of a survey of ten conserved Gotland sandstone objects in the centre of Stockholm and in the countryside with the aim of evaluating previous conservation treatments. Myrin moreover placed Gotland sandstone samples (consolidated with Wacker OH) outdoors with the aim of studying the durability of consolidation. She has also tried to evaluate the efficacy of a mending material, “Billy’s Mortar”, commonly used in Sweden. [34] She has used visual assessment which involves making comparisons with old photographs and information found in conservation reports, and using Karsten pipes to measure the water absorption. She concludes that the old reports weren’t good enough and that no NDT methods were available to assess the condition. This remark demonstrates that her choice of methods was based on cost, since many NDT methods are available, albeit quite expensive. [35] In her recent research, published in 2006, she uses ultrasonic and colorimetric measurements.

Some sophisticated NDT methods have been tested in Sweden. One early example was the investigation of the base of the Carrara marble Gustav II Adolf sculpture in Gothenburg between 1992 and 1999. Germans Wolfram Köhler and Stefan Simon measured the ultrasonic pulse velocity (UPV) of the stone within the framework of the *Eucare-Euromarble* project. Köhler also measured other Carrara marble sculptures in Stockholm in 1992 within the same project [36], and Bylund and co-workers measured the Carrara marble outdoor sculptures housed in the National Museum of Fine Arts in Stockholm in 1995–1996. [37] Stefan Simon presented the results of testing Swedish sculptures using ultrasonic tomography of the interior of marble in his PhD thesis on the weathering of marble. [38] Moreover, Anders Bodare at the Royal Institute of Technology in Stockholm also tested the stone with Hammer wave propagation (see below) in combination with an impact-echo technique. [39] Simon has also used ultra-pulse velocity UPV on the marble sculpture, Flora, in Gothenburg’s Botanical Gardens. [38]

During the period of the Air Pollution Programme, the NHB financed several research projects using NDT methods. Anders Rehn at the Department of Electromagnetic Theory at the Royal Institute of Technology tested acoustic and electrical parameters in 1995 and 1996 on behalf of the NHB on different Swedish natural building stones. The electronic method consisted of the use of high resolution radar; transmission line radar that can detect contrasts in the electric parameters in the stone. Knowing the parameters of the fresh stone, the measurement can demonstrate if the stone has weathered. The test was performed on both homogenous and inhomogeneous stones: Ekeberg marble, Red Öved Sandstone, Gotland limestone from Norrvänge, Lingulid sandstone from Lemuda, chalk and Köpinge sandstone. The measurements were conducted on dry stone, on stones saturated with water, on weathered sandstone from the Royal Castle in Stockholm and on weathered Öved sandstone. Moreover, Gotland sandstone was impregnated

with alcoxysilane and these stones were measured dry and saturated with water. The technique can detect flaws in the stone, although the report demonstrates that this works well when the stone is dry (the signal can penetrate 0,2 – 2 metres). It doesn’t work particularly well on wet stone since the penetration isn’t deep enough. It is possible to detect cracks inside the stone, however. [40]

Rehn measured the acoustic parameters of the same stones using ultrasonic waves. He tried two methods: one where the samples were placed in a water tank, where the sound was reflected and received by a transducer and recorded afterwards, and one that transmitted the ultrasound through the stones, where the sound was also recorded afterwards. The results show that the velocity of the sound may differ in different directions of the stone (which is common in stones that aren’t homogenous). When the sound was transmitted through dry and fresh Gotland sandstone, the velocity ranged between 2.2 km/s and 2.5 km/s respectively. When the stones were saturated with water, the velocity was higher: 2.6 km/s. The sound that was transmitted through impregnated Gotland sandstone with alcoxysilane gave an even higher velocity: 3.2 km/s and when the impregnated stone was saturated with water: the velocity was 3.4 km/s. The measurements of the reflections in the water tank demonstrated also that it was not possible to measure stones with cracks. This method is thus not as useful as one might expect and also requires sampling, whereas nowadays portable ultrasonic apparatus is available for measuring the transmissions. [41] Rehn’s tests only give us the measured velocities on fresh stone, however. The tests have to be complemented by testing on weathered stone in order to understand the stone’s quality. Furthermore, the measurements have to be compared with other testing methods, such as the compressive strength and the tensile strength in order to find out where the actual critical breaking points or intervals are. This has been done with other stones, such as Carrara marble.

3.5 Recent and On-going Research in NDT and Conservation

At international level there has been a rapid advancement in the field of NDT methods, which makes it very difficult to form a complete picture of the present situation. Some projects of interest are presented in this report, however. One current trend in scientific conservation in Europe (in general) is to create strong and formal networks. These networks are supported by the European Commission. Within these networks, the institutions have to bring their experiences together in a more formal way than a normal exchange, which means that the results are considered rather more superior. It is sad that Swedish institutions very seldom participate in these networks and the consequence is that Swedish conservation institutions miss important opportunities to learn from and share with these experienced

institutions. It is therefore hardly surprising that we miss some of the exciting developments of the field. Today Swedish museums, universities and institutions (such as the National Museum of Art, Gothenburg University or the National Heritage Board) do not prioritise their resources for such a development.

There are some interesting projects in conservation going on in Sweden – using NDT methods – such as the Lidar Laser project at Lund University (see below), research at the NMK School at Chalmers University such as the PhD projects by Malin Myrin (see above) and Pär Meiling. This school is due to close in 2006, though. By and large the efforts are somewhat isolated from each other and have no natural place to grow, which means that there is a strong need for a conservation centre – perhaps a Nordic Centre – within which to conduct research and development in the field in Sweden. Otherwise, efforts are destined to remain short-term and researchers will be unable to seriously participate in current international research.

While this report only gives a first outline of the situation, it does provide an indication of which methods may be worth considering in the future. One valuable follow up would be to assess which institutions are able to provide help, which instruments are accessible, their limitations and cost and so forth.

Labstech

LABSTECH is an important international network consisting of several internationally distinguished European laboratories (and some American) working with scientific and technological applications in the study of cultural heritage. [42] The network was established in 1999 and is a European Infrastructure Cooperation Network within Improving Human Potential (IHP) in the Fifth Framework Programme (1999–2002). At present 47 laboratories, institutions and museums are included, although no Scandinavian institution has yet joined. The goal is to realize an effective interchange of know-how and joint resources.

LABSTECH supports many activities, such as workshops and conferences. Some of the leading organizations have accepted particular responsibilities. For example, the National Conservation Institute in France, *the C2RME*, has undertaken to gather and supply information about the research facilities available in each Institution, to review techniques and strategies for the characterization of the original materials of artefacts and their deterioration, to discuss and propose standardizations, to work towards using portable instrumentations for *in-situ* control and the maintenance of artefacts, to foster co-operation through the exchange of trainees, and to promote the creation of joint research projects.

The governmental body OPD (Opificio delle Pietre Dure) based in Florence is the official convener of the programmes concerning *Methods and Materials for Conservation*. With

in this programme there is a search for a critical analysis of methods and materials commonly used for the cleaning, consolidation and protection of artefacts (paintings, objects, stones, etc) and the review of substances suitable for use as consolidants or protectives, and the documentation and archiving of data. The governmental agency of ICR in Rome is responsible for the programme to provide information on archiving methodologies and hardware-software facilities available in each Institution, to indicate perspectives towards a compatibility or standardization of the archiving solutions and to work towards the development of joint research projects in an attempt to create a multilingual European thesaurus of analytical data devoted to cultural heritage materials. [43]

Within this network there are several activities that include NDT methods. For example, new portable apparatus has been developed and interchanged between the different projects.

Laser Technology

Several NDT conservation methods based on laser technology are currently under development and most of the research has been presented at various conferences and represented in international *LACONA network (International Conference Lasers in the Conservation of Artworks)* publications. The first conference was held in October 1995, in Heraklion, Crete, Greece, and the last Conference, *LACONA VI*, was held in Vienna in September 2004. [44] The next one is planned to take place in St Petersburg. Principally, laser is used in three areas:

- 1 Cleaning
- 2 Analysis of the absorption of the laser beam to determine surface materials
- 3 Creating an image of the surface of an object by using a laser scanner that measures the "time of flight" of a laser beam, i.e. the time it takes for a laser pulse to go from the instrument to a target and back again. The result is a cloud of points that is produced by the scanning. Every point has a specified coordinate in x, y and z. From this cloud it is possible to create drawings and 3D models and wire frames at both macro and micro level. [45]

The latter two fall within the frame of NDT methods and have numerous possibilities for conservation. For example, Åberg, Stijfhoord, Råheim and Löfvendahl used laser microprobes to analyze and determine the weathering depth and provenance by carbon oxygen isotopes on Gotland sandstone already in 1993. This is, however, a destructive method. [46]

Regarding the second type of laser use, a research project dealing with a mobile laser lidar remote-sensing system is currently underway (2001–2007) at Sweden's Lund Institute of Technology. The project is intended to help to "read" a building to determine its materials and state of conserva-

tion. The laser lidar technique analyzes the surfaces of objects or buildings using laser-induced fluorescence (LIF) and laser-induced breakdown spectroscopy (LIBS). The spectroscopic "reading" is designed to help the conservator-restorer to map different kinds of substances, differences in temperature and other physical phenomena. Through computer modelling it is possible to "see" information that the eye normally cannot detect. It is possible to use the technique for long distances and during difficult weather conditions. The most difficult task, however, is analyzing the results – which is in progress right now – within the project. The project is headed by Lund University, IAFC-CNR in Florence and the Swedish Institute in Rome. [47, 48]

The third possibility, which is becoming much more common, involves using laser scanning for calculating and evaluating the weathering of stone. The laser scanning method documents the surfaces of buildings and materials and can be used to study changes, such as those caused by weathering. One important and recently completed project is Swantesson's study of the weathering of rock art. He used a laser scanner and a fully computerized data collection system to analyze the changes in the surface. Fortythree sites were investigated, of which 26 were repeatedly measured between 1994 and 2003. The technique has developed considerably since the beginning of the project and some of Swantesson's problems included the equipment being too large and slow and superseded by rapid technological developments.[49] This is a possible future area of research, however, as the 3D laser scanning technique has recently also been used by Trinks and co-workers in Great Britain to visualize rock art. They found that while the result was satisfactory, the high costs involved need to reflect the greatly increased potential and possibilities for data analysis and visualization. [50]

Laser scanning is mainly used for documenting and surveying buildings and landscapes. The University of Ferrara in Italy used a 3D scanner to scan the Coliseum as early as 1998. [51] By this time the first commercial 3D laser scanner suitable for field surveys had reached the market (with regard to the Cyrax 2400, however, the cost was initially some US \$750,000 or more!). It was found that the system could interpret the data in the CAD-software and the accuracy could be up to ± 6 mm and the measuring distance from 0.1 to 50 metres. Later, the U.S. Capitol, the Wilson Automobile Plant in Kansas and the Kansas State Capitol were surveyed using the same technique. In Sweden several private consultant companies work with laser scanners, such as Metimur, Bergbygg Konsult and Aria Consulting. For example, Aria Consulting scanned Visingsborg's ruin in 2003 together with the NHB, and Metimur scanned a sculpture from the National Museum of Art in Stockholm. One good introduction to the 3D laser scanner system has been made by Hughes and Loudon (2005), who used the 3D Laser Scanner to scan and translate data into pictures and drawings from the Continental Grain Elevator in Brown-

wood Texas in 2002 to 2003, the Bluff Dale Bridge in Texas in 2003 and the Historic Roadside Parks across Texas in 2004. The result was more than satisfactory. [52] The technique is still time-consuming, expensive and requires experienced personnel, but will nevertheless be increasingly used in conservation.

MICRO drilling resistance and stone durability research

One project supported by the European Commission within Energy, Environment and Sustainable Development is the European project relating to integrated tools for *in-situ* characterization of effectiveness and durability of conservation techniques in historical structure, *DIAS (Drilling Indentation Acoustic of Stones)*, which ran from 2002 to October 2005. It was an essentially destructive test and the Project Coordinator was Dr. Piero Tiano from Italy. The main purpose has been to develop a valid and cost-effective tool for laboratory and *in-situ* assessments of the residual mechanical properties of building stones. A portable tool has been developed including Micro-drilling, Indentation and Acoustic devices and associated software (*DIAS*) in the *Drilling Resistance Measurement System (DRMS)*. The project aimed at assessing the effect of the weathering on stone materials and their mechanical durability, the inner microstructure of stone and other materials and the absorption and effectiveness of consolidants. [53] The technique cannot be used on sculptured stone, however, since it is destructive and leaves small holes in the stone. [54] The *DIAS* project is also associated with the McDur project relating to the effect of weathering on stone materials and the assessment of their mechanical durability, also organized by Piero Tiano from 2001 until 2005. This project is also supported by the European Commission and aims to establish and facilitate assessment of the durability of European stones. Some NDT methods have been used in this project, such as portable devices for determining the Drilling resistance (DFMS), Rebound value (Sclerometer) and Ultrasonic pulse velocity (Ultrasound devices). [55]

EU-ARTECH

EU-ARTECH (Access Research and Technology for the Conservation of the European Cultural Heritage) is another European collaboration within the field. The programme fosters the use of several NDT methods. Members of the European Community can apply to use the portable systems (see below at ALGEA and MOLAB). [56] The programme will run for five years and have many European partners – none from Scandinavia, however. [57] The EU-ARTECH consists of thirteen European infrastructures operating in the field of conservation. In selecting the research institutions it was important that they operated in an interdisciplinary way, with conservators, archaeologists and art-historians working together. The main objectives of EU-ARTECH include working towards a permanent exchange among the

participating infrastructures and establishing cooperation and exchanges of knowledge with the other infrastructures in the field, thus helping to structure a common European research area. The network aims towards:

- drawing together as many European cultural heritage facilities as possible in one network.
- enabling the European user community to have easy and transparent access to a range of advanced resources.
- offering an integrated, professional and consistent level of support.
- improving data products delivered by European facilities.
- extending opportunities of access to users of EC Associated Countries.

EU-ARTECH can do this through access to:

I – AGLAE, a single high-level infrastructure located in Paris at the Flore Pavilion of the Palais du Louvre (CNRS-C2RMF), where non-destructive elemental composition studies are carried out with high sensitivity and precision, in a unique environment consisting of art-historians, restorers and scientists with an extensive range of expertise on artwork studies and conservation;

II – MOLAB, a unified group of joint infrastructures, located in Florence and Perugia (UNI-PG, CNR-ICVBC, OPD, INOA), where a unique collection of portable instrumentations, together with competences relating to methods and materials, is available for in-situ non-destructive measurements. Access is devoted to artwork studies and/or the evaluation of conservation methods, in the setting of a museum, a restoration workshop or an archaeological site.

Getty's Conservation Institute

NDT methods have also been used in past and current projects at the Getty's Conservation Institute in Los Angeles, although none of the present projects are directly aimed at developing NDT methods. A project that was concerned with these methods was the *Evaluation In-situ of the State of Deterioration of Monumental Stones by Non-destructive Sonic Techniques*, which was undertaken between 1986 and 1987 in Spain and led by Modesto Montoto. Another important project currently in progress is *Salt Research: Mechanisms of Salt Decay and Methods of Mitigation*. Testing and monitoring are conducted in both the lab and in the field of salt reduction and desalination in order to assess potential damage of the salts, and there is scope here for the use of NDT methods. [58]

4. NDT Methods – Theory and Application

This part of the report describes the NDT methods discussed in the literature and gives examples of how they have been used. The division of the methods has been made according to the kind of questions they answer.

4.1 Methods for Measuring the Roughness/Relief and Weathering of Stone

Measurement of the surface roughness is usually connected to studies that assess the effect of weathering or cleaning. The same methods are thus used for both purposes – although the weathering studies usually involve longer periods of testing and more regular measurements, while the effect of cleaning is made once. Easily handled and less expensive equipment is desired for the former. Several NDT techniques can be used to measure the surface roughness, and some of these will be discussed in brief in this report. None of them have been tested in this project, however. The articles have been aired in the context of conference proceedings dealing with stone conservation and at special conferences directly concerning weathering, such as *Stone Deterioration in Polluted Environment* held in 2004 in Wolverhampton, UK, within the SWAPNET network and the workshop *Air Pollution and Cultural Heritage* within the European CARAMEL project held in Seville in Spain in 2003.

One study is of special interest, namely, the Grissom and co-workers article *Measuring Surface Roughness on Stone: back to basics* in *Studies in Conservation* (2000). The authors tried several surface roughness measurement methods and compared them, such as *the moulding technique, stylus profilometry, laser triangulation profilometry, reflected light image analysis (RLIA), micro drop absorption test* and *visual and tactile evaluation*. The conclusion was that the visual and tactile evaluation was the most efficient method. The visual and tactile evaluation consisted of a study of 64 blasted surfaces. They were compared with control areas and rated according to verbalized criteria like "slight", "moderate" and "significant" changes in roughness, as well as the presence or absence of a step at the interface between blasted and controlled areas. The result was so good that the authors continued and asked six blindfolded examiners to select sets of marble, limestone and sandstone samples that could serve as "standards". The examiners rated the samples from 1 for the smoothest surface and increased the digits for the rougher surfaces of each kind of stone. [59] None of these methods have been applied in this study.

The moulding/surface cast technique methods

The moulding/surface cast technique methods are simple in theory yet difficult in practice. The technique has usually been used to study deterioration processes and several moulds have therefore been taken regularly over a period of time. [38] A mould of a chosen area is cast, usually with silicone rubber. The mould is taken to the laboratory and studied, analyzed and photographed with a microscope. In the laboratory the researchers sometimes use other techniques, such as a profilometer or reflected-light image analysis (RLIA). [60]

Silicon rubber is the most commonly used material. It was used by Grissom and co-workers and poured onto the surface, with Plasticine to keep the liquid rubber on the surface. Test samples were thereafter de-aired in a vacuum for ten minutes and afterwards were allowed to cure for 24 hours. Their tests were conducted on stone samples in the laboratory, however, as in a field study it can be difficult to keep the vacuum sufficiently tight before the rubber starts to cure! Grissom and co-workers also observed the importance of using the same amount of silicone on each cast and that this was difficult to achieve in practice. There were further problems in that the Paraloid B-72 was used to protect the surface of the stone and left visible brushstrokes on the replica's surface, which complicated the laboratory study. They thus tried to use the silicone rubber without protection, which resulted in some silicone rubber sticking to the surface. The problem was finally solved using a protection consisting of a solution of 2 percent Paraloid B 72 in acetone and ethanol (proportion 1:1). [59]

The moulding technique has a long conservation history. It was, for example, used by the Bayerische Landesamt für Denkmalpflege (BLfD) to study stone deterioration in the vicinity of Munich from the 1940s until the beginning of the 1980s. The methodology was based on surface moulds being analyzed in the laboratory using a surface roughness meter – a Perthometer – constructed by the BLfD. The Perthometer distinguished four types of roughness: 25 µm, 50 µm, 100 µm and 250 µm. Moreover, the results from the Perthometer were used to calculate the measured profile in Leuchtdioden (LED) into an average roughness R_z , the maximum roughness R_{max} and the arithmetic average roughness R_a (the average height of the irregularities of the surface with reference to an ideal intermediate plane). [61] The Munich methodology was successful and meant that

the authors could ascertain that corrosion of the stone did not happen suddenly but was a continuous phenomenon. Furthermore, the results proved how much different stone weathers over a 20 year period (the granite had lost 45 μm and the marble 95 μm). Through calculations of the rate of deterioration it was thus possible to state that stone surfaces weather by several millimetres in a millennium. [62]

Grissom and co-workers used moulds made of silicone rubber and analyzed through the reflected-light image technique (RLIA) in the laboratory. According to them, the technique has the advantage that the replicas can be conveniently studied with a microscope and photographed in the laboratory. Several problems were found to outweigh this advantage, as illustrated here:

- 1 They used coloured rubber since the white rubber reflected the light too much during the RLIA analysis. This red rubber left colour stains on the stone.
- 2 It was hard to achieve the same thickness of the moulds and prevent the rubber sticking to the surface.
- 3 The method was time-consuming and thus not to be recommended for large-scale projects.

Despite these problems, they concluded that the RLIA analysis was a powerful tool to use for defining surface roughness. It is used by some laboratories in the US and equipment costs ca. US \$10,000. [59]

Surface Roughness Meters (Profilometer/Rugosimeter)

The stylus diamond profilometer and other surface roughness meters register the surface profile of a material. The technique consists of an instrument with a metal stylus (a needle) that transverses a line and the roughness is registered digitally. It was initially developed for measuring the roughness of metal surfaces and has thereafter been used on stone. Some of the problems with the technique stem from this. The technique has nevertheless been well used in stone conservation. Some studies have demonstrated that the technique isn't all that successful. [59] The scientific conservator Mathias Kocher in Bavaria has commented that the technique isn't useful on sandstone. [63] Some qualities of sandstone are too "rough" and the technique may not work on these. The profilometric techniques are therefore probably best for "denser" stone surfaces on limestone, granites and marbles.

There are different portable and reasonably cheap instruments on the market such as the Surtronic duo 3+ and the Perthometer M4P150. The Surtronic 3+ is a relatively cheap tool (less than US \$5,000). The technique is based on a diamond stylus with a 5 μm tip radius and a 6 mm-radius red ruby skid, against which the vertical position of the stylus is measured by means of a piezo-electric transducer. The maximum transverse length of the stylus is 25 mm, and the

gauge (vertical) range for measurements tiny; 100 μm . The instrument must be calibrated against a metal standard prior to each sample. Several measurements must be made on each stone before the average can be calculated. [59]

The technique measures a given distance and produces a profile of the surface and thereafter the software calculates the average roughness R_a and maximum roughness R_{max} . The average roughness R_a (μm) is a defined arithmetical average of the absolute values of the measured heights from the mean surface taken within an evaluation area. The average roughness is standardized (DIN 4762, DIN 4760 ff and DIN 4770 ff). Sometimes roughness is given as R_g the root mean square (rms) parameter corresponding to R_a , the maximum roughness R_{max} , the mean roughness R_z or to P_c the number of the recorded peaks.

The Italian scientific conservator Frederico Guidobaldi at CNR in Rome used the profilometric method for measuring the corrosion of marble quite early on. He used a Taylor-Hobson Surtronic 3 for this purpose. The marble was tested in the laboratory by dripping simulated acid rain that had been washed and measured. He came to the conclusion that after the test-programme the surface roughness was negligible – even though the acid rain actually caused damage. The technique was thus not suited for registering the weathering. [64]

In the *Euicare-Euromarble* project, some samples of European marble were put in place to study the deterioration. [38] One was the Swedish Ekeberg marble and two of the test sites were selected in Sweden (in Stockholm and Gothenburg). The stones were placed outdoors in 1992 and measured over a long period. Simon used a Perthometer and an UPV instrument to measure the deterioration. The results were presented in R_a , [12, 38]. In this work there is very little discussion about the technique's problems, however. It would thus seem that the technique gave reliable results.

Grissom and co-workers tested the stylus profilometer. In their report they demonstrate that the roughness meter cannot be used for very rough stones, i.e. when the roughness exceeds 10 μm . Despite this, they point out that a more expensive tool may be better for this purpose. According to them, data published from such measurements raise doubts in their minds and they conclude that when measuring sandstone, it is the weathering of the matrix that is recorded. Moreover, after decades of outdoor exposure the surfaces of weathered stones are found to maintain a constant roughness as the grains are dislodged, even though they actually recede significantly. These factors thus make them suspicious about the method. [59]

Optical Coherence Tomography (OTC) can also be used for the imaging of semi-transparent objects. Originally intended for use in medicine, it can also be used in artwork for the study of the structure of oil paint, varnishes and glazes, and can be used for profilometric applications for surface roughness measurements. [65]

The *Micro erosion meter (MEM)* is another mechanical instrument that can be used to measure rock weathering. It is a simple micrometer device that measures surface heights at a number of prefixed points relative to studs attached into the stone. It has been used to monitor stone decay at Saint Paul's Cathedral in London over a ten-year-period. [4]

The use of optical and laser techniques to study surface roughness

The surface of stone is often monitored using different optical techniques and cameras, such as micro monitoring that is used to study the deterioration. One such technique is the *Close-range photogrammetry* used for monitoring surface loss. One example is the Merchants Exchange building in Pennsylvania. This technique is the same as for close-up photographs; raking light at the same location and then compared in the computer. The latter method is used at the ICVBC-CNR in Florence to determine weathering – it was for instance used during the conservation of the Rape of the Sabine Women. [66] The techniques require experienced personnel and are difficult to use in the field, however.

Line profilometry is another contact-free NDT technique used to study surface roughness. The technique projects a grid of lines onto the surface at an angle of 45°. Any irregularities in the surface become immediately evident. This is because the lines are displaced depending on the protrusions and indentations on the surface. The measuring range is from 40 to 500 µm; structures from ca. 5 µm heights can be qualitatively determined and heights from 0, 5 to 1 mm can be well estimated. This method has been used by Aires-Barros, Mauricio and Figueirido in the laboratory as well as *in-situ* at the Basilica da Estrela in Spain (1994). The collected data was used to create a rock weathering index. [4, 67] Although the method was successful, it seems that it is not very frequently used in conservation and requires experienced personnel.

Laser micro-profilometry is an interferometric instrument that measures the distance between an optical head and the surface of objects with high precision. [68] Asmund and others were among the first to suggest *laser interferometry* to monitor surface loss in stone already in 1973. [4] *The laser micro-profilometry* has been applied to measure and monitor the surface of artworks, to understand micro colour detachments, and to monitor the roughness before and after the conservation intervention. It is possible to detect very small deformations (0,5 µm). This technique is well developed nowadays and there are portable systems based on electronic speckle pattern interferometry (ESPI) and video holography. Meinschmidt and co-workers used such a system to monitor deformations during the hardening of mortars and the growth of efflorescence. [69] The method can also be combined with a video holography. [4] The technique was used by Grissom and co-workers and while they

found it successful, it was too expensive and labour intensive. An instrument cost US \$ 25,000 or more. [59] The Florentine Conservation Research Institute (ICVBC-CNR) used a *Micro-Roughness Laser Meter* technique (that can take pictures on micro levels of 5 µm) to monitor the cleaning of the Rape of the Sabine in Florence. [70]

Laser 3D scanner for micro mapping of surfaces can be used to monitor weathering of all kinds of materials. It gives 3D pictures of the surface and makes it possible to calculate the surface roughness and loss of material (that is demonstrated in maps). We have already mentioned that Jan O. Swantesson has monitored the erosion of rock carvings in Sweden using a laser scanner. He found the technique quite time consuming (2 h for a 40x 40 cm area) and difficult to handle. The technique is sometimes hard to use in the field since good weather and strong currents are required. [49] The methodology has been further developed in England for studying rock carvings. [50] The technique will, without doubt, become easier to handle and less expensive with time. Moropoulou and Haralampopoulos, Tsiourva, Auger and Biginie have also tested the performance of consolidation materials on porous stones in Greece and Cyprus using a camera-laser scanning system. In this case the tests were performed on prisms in the laboratory. The researchers concluded that the technique "may provide unique information concerning the roughness increase in the surfaces." [71] The method is not yet common and quite expensive and requires experienced personnel. [46] Despite this technique will have great possibilities in the future.

"Tape method"

The "tape methods" are not very common in conservation and no articles have yet been found concerning the methods. An ASTM standard exists for testing the adherence of paint. Marisa Laurenzi Tabasso believes that it is useful for the evaluation of surface deposits (and sanding) but not for quantitative evaluations. The NHB has nevertheless employed a "tape" method that uses ready-made labels. The methodology is very simple:

- 1 In the laboratory, seven prefabricated self-adhesive labels 32x44 mm and manufactured by HERMA are weighed. The average ($A_{initial}$) weight is then calculated (from seven labels).
- 2 In the field, three HERMA label are attached to the stone's surface. After a few seconds the labels are taken off, folded and put into a sealed plastic bag.
- 3 In the laboratory, each label is weighed individually and the average of each sample ($A_1, A_2 \dots$) is calculated (A_{field})

$$A_{field} = \frac{A_1 + A_2 + A_3}{3}$$

These are compared with the previous average to find the difference from the average (D) :

$$D = A_{initial} - A_{field}$$



Figure 1. Labels attached for the granular disintegration test.

The results of the test will be presented in the next report. The tape method is micro destructive (it takes some stone material on the surface). The result depends on the granular disintegration (sanding) of the stone and the presence of surface deposits. The loss of material depends on the weathered state of the stone.

4.2 Methods for Analyzing and Measuring of the Salt Content in a Material

It is well known that water-soluble salts are one of the primary factors that provoke severe damage in porous materials and one of the most common causes of deterioration in historic masonry. It is thus of particular interest to assess the salt situation before conservation. Moreover, some of the NDT methods, such as the UPV and moisture measurements, are potentially disturbed by salts and is it therefore important to check whether they are present when using these methods. This report focuses on NDT methods and does not intend to describe the salt problem in depth. The mechanism of salt weathering in historic masonry has not yet been fully understood and is continuously being studied by several researchers such as Bläuer Böhm, Price, Steiger, Doehne, Charola and Arnold. [73–79] In order to learn more it is necessary to look at their research and several scientific disciplines publications. A recent summary of the situation is provided by the article *Salt Weathering. A Selective Review* by Eric Doehne. [79] A bibliography on salts is con-

Table 1. Table of Surface Roughness NDT methods

NDT Method / Instrument type	Measuring area	Instrument's weight, accuracy, range, speed, and resolution	Possible to use outdoors	Needs personnel trained in science	Use in conservation	Expensive/inexpensive
Imprint moulding techniques / surface cast techniques	Measures surfaces as big as the imprint – usually quite small.	Time-consuming and laborious; both outdoors and in the laboratory. Depends on the type of measurement that is performed such as RLIA or profilometry.	YES	YES	YES	Follow-up-work is expensive since it is time-consuming. The material is cheap.
Surface Roughness Meters profilometer/ rugosimeter	Depends on the instrument. The settings of a Surtronic 3+ are: 25 mm traverse length, 500 mm maximum gauge (vertical). The upper limit for Surtronic 3+ is 2.5 mm. Will give standards measurement of R_z , R_p , and R_v	Depends on the instrument (there are many). Surtronic 3+ : Weight: ca 200 mg, Accuracy: ca 5 % of reading, Resolution: 1 mm for horizontal measurements and 10 nm for vertical. Speed: ca 2 mm/sec. The data is analyzed in computer programs.[72]	YES Can normally be used in normal temperature and from 1 until 80 % humidity.	NO Easy to use.	YES The instrument is made for metals, but also used in stone conservation	Moderately expensive.
The use of optical and laser techniques to study surface roughness	Depends on the instrument, can be very small (μm) or large (measurements of whole buildings).	Depends on the instrument (the older techniques were quite heavy and time-consuming); however the latest techniques are very accurate, fast and less heavy with high resolution.	YES	YES	YES	Expensive; both equipment and personnel.
Tape method	32x44 mm	Very easy to use, the accuracy has not yet been explored but is very fast.	YES	NO, Very easy to use.	NOT YET as far as I know.	Very cheap.

nected to this article, which is available on the Internet. [80] One particular conference is worth mentioning as problems with removing the salts were specifically discussed: *Le des-salement des matériaux poreux* held in Poitiers in 1996. [81] The Getty's Conservation Institute is also involved in an on-going research programme entitled *Salt Research: Mechanisms of Salt Decay and Methods of Mitigation* and deals with salt problems in conservation. [82] Proceedings for dealing with such problems have also been discussed, such as at the *SALTeXPERT Meeting* in Prague in 2002 (to be published). [83] Even though many articles discuss salts and their problems and laboratory studies demonstrate how the salts destroy porous material, very few articles discuss using NDT methods to identify salts.

Before we present the NTD methods, I will briefly discuss the salt problem in general. The salts either reach the building material from the ground by capillary absorption or derive from the actual building materials themselves, from rain water, dust, pollution and/or earlier conservation interventions. The most common salts found in building materials are carbonates, sulphates, nitrates and chlorides of alkali metals and magnesium (with cations such as Na^+ , K^+ , Ca^{2+} , NH_4^+ and Mg^{2+}). Some of these salts are soluble in water and others are barely soluble. The water-soluble salts are readily transported inside a porous material together with water. The crystallization and solubility of the salt is effected by environmental conditions such as the RH, pH and temperature. When the soluble salts dehydrate they form crystals that cause deterioration; for example, granular disintegration, exfoliation, spalling, and eventually total collapse of the material. Some salts, such as sodium sulphates, are particularly destructive. Consequently, the salts found inside historic stonework are complex dynamic systems that are subjected to continuous phase transformations like hydration/dehydration and crystallization/dissolution. Hence, when severe salt problems exist in a building it is necessary to map and assess both the salt and the climatic situation for longer periods both before and after conservation. [73]

The analytical methods therefore need to assess whether salts are present, what kind of salts, where they come from and their concentration. Several methods are available for these purposes; some of them are non destructive, some are *direct* (that is, for example, chemically analyzing the salts) and others are *indirect* (that is, for example, measuring the conductivity). Since the behaviour of the salt is closely connected to the climatic conditions surrounding the masonry, it is necessary to combine the investigation with a monitoring of the moisture, RH and temperature. The investigation of salt in masonry is thus quite complex, and several methods are normally needed to provide the whole picture. There are several examples of such complex investigations; one being the investigation of the church San Pietro Martire in Gnosca in Italy from 2002 until 2003. [84] It is, however, not always necessary to conduct such a complex analysis.

Considering that the most important factor is to know whether salts exist and in what concentration, a salt investigation in a historic masonry can be divided in two stages or steps:

Step 1) Water soluble salts are the most destructive and only those are normally analyzed. The conservators-restorers thus begin to measure the conductivity of a salt solution extracted from the material (see below). The salts are extracted from the masonry using paper pulp or by soaking drilled core samples of the material. The advantage of the core samples is that the method will make it possible to gain a picture of the salt distribution in the masonry (by dividing the samples in a depth profile). The samples or paper pulp tissues are immersed in water and both the pH and the conductivity of the solution is measured. This result gives a good indication as to whether the situation is serious. If the conductivity is too high, the analysis of the salts continues into Step 2.

Step 2) When the conductivity is high (depending on the salt between 100 and 300 $\mu\text{S}/\text{cm}^2$) it is necessary to investigate the kind of salts there are, where they come from, the climatic conditions, and to map when the salts go in and out of solution over a period of time. Sampling is required to analyse which ions are present. It may also be necessary to take several core samples and map the distribution of salts in the wall and to monitor and investigate what happens to the salts depending on RH and temperature. A building may demonstrate different salt problems depending on the season and the weather. Ion Chromatography (IC) is usually used to determine the salt ions. Further analysis is necessary, however, to determine the precise nature of the salts, such as whether the salts are hydrated. For example, if Na , SO_4 and CO_3 ions are found in the sample it could indicate the presence of Na_2SO_4 or Na_2CO_3 . In this case, X-Ray Diffraction (XRD) analysis and/or polarization microscopy (PLM) analysis is required. When the anions and cations are analyzed as well as the climatic conditions, it is possible to plot the results to special data-programs that simulate and calculate the salt situation. Steiger in Germany and Price in England have developed computer models (ECOS among others) that help to understand the salt complexity. [85] This has recently been carried out (after some trouble) successfully on Cleeve Abbey in England. [86, 87]

The next problem is how to remove the salts completely. Only a few methods are available for this purpose, such as extraction by the repeated application of paper pulp compresses or poultices and electro-migration with the help of electrochemical processes that force the water soluble anions and cations of the salt towards electrically charged electrodes, thus removing them. [88] The second kind of extraction is very complicated which is why the paper pulp/poultice method is more frequently used. One problem with this method is that it is hard to reach the depth of the structure. The measurements of the conductivity are also con-

ducted after conservation to control that the salts have been sufficiently removed. The salt methods have not been tested in this study.

Methods for analyzing the salt composition

Portable NDT methods are available to determine what kind of salts are present in the masonry. One such portable NDT method is the *Neutron Probe* which uses gamma/neutron activity to determine the elemental composition of a material and is extensively used to measure the moisture content in soil. The elements are identified by characteristic gamma rays being emitted from the material when bombarded with neutrons. It can consequently be used to determine materials and locate water and salts. [89] Nevertheless, the most common method is to scrape off some of the salt efflorescence, take core samples or extract water soluble salts with compresses to be analyzed in the laboratory. The salts are often analyzed with Polarization microscopy (PLM), Micro chemical tests, Ion Chromatography (IC), IR spectroscopy, X-ray diffraction (XRD) and other analytical tools.

Methods for determining the quantity of the soluble salts

In order to determine the quantity of the soluble salts it is common to either use wet paper pulp or tissues to extract some of the water soluble salt or take core samples and afterwards measure the conductivity of the extracted salts in the laboratory (as well as the pH and alkalinity). The conductivity gives an indication of how much water soluble salt is present in the masonry.

The Borelli method – several similar methods are in use in stone conservation. One is described by the scientific conservator Ernesto Borelli at ICCROM in Rome. He has suggested a standard NDT methodology to analyze water soluble salts both *in-situ* and in the laboratory. The methodology is based on the removal of salts using paper pulp compresses (Arbocel BC 1000) and distilled water (with a conductivity less than $5 \mu\text{S}/\text{cm}^2$). The methodology comprises:

- 1 Distilled water is mixed with paper pulp (20 l of water to each paper pulp kg) and applied 5 mm thick over a 150 cm^2 area on the surface that is being examined. The pulp is left to dry for between 24 to 48 hours (depending on the climatic conditions).
- 2 When the pulp has dried, it is divided in three parts; each ca $5 \times 10 \text{ cm}$. One is measured *in-situ*; one is taken to the laboratory to be tested and the last is archived.
- 3 The conductivity is measured *in-situ*, using a portable conductivity meter. One of the paper pulp pieces ($5 \times 10 \text{ cm}$) is washed in 200 ml distilled water and filtered in a cellulose filter and the conductivity is measured.

- 4 In the laboratory, the same procedure is performed with the second piece of pulp ($5 \times 10 \text{ cm}$) and using the same amount of water (200 ml). The nature of the salts is also analyzed. This characterization of the salt ions is usually conducted with a normal *Ion Chromatograph* (IC) or *Inductively Coupled Plasma Atomic Emission Spectroscopy* (ICPAES). The results are presented in mg/cm^2 . [90]

Borelli discusses the problems concerning the measurement of the conductivity *in-situ*. The conductivity only directly corresponds to the salt concentration if only one kind of salt is present in the sample and if the nature of the salt is known. The reason for this is that different salts ions have different conductivity. Hence there might be errors in the field tests (since one does not know which kind of salt it is). The comprehension of the situation is complicated when several salts are present in the material. Borelli thus recommends presenting the salts in tables that exhibit the percentage of each salt ion, and after that compare the ions with the conductivity. Nevertheless, he concludes that conductivity measurements around $50 \mu\text{S}/\text{cm}^2$ usually don't usually create any salt problems, while measurements of around 100 and more than $500 \mu\text{S}/\text{cm}^2$ often do. [90] These values can thus be regarded as boundaries that determine whether the salts do, or do not, lead to damage.

The Skanska method – while this report concerns NDT methods, I also describe a destructive methodology that calculates the soluble salt content in a porous material. It is used by the scientific conservator Daniel Kwiatowski at Stenkonservatorn at Skanska in Sweden. The result gives the amount of soluble salts found in the material in both weight and percentage (of weight). The methodology is as follows:

- 1 1–2 g sample of the material is taken to the laboratory and ground as finely as possible. It is then dried at 60°C and weighed. The weight is registered (g).
- 2 The water soluble salts are extracted from the samples with 100 ml distilled water. The solution is shaken for an hour in such a way that the stone powder becomes separated from the solution. The solution is then filtered.
- 3 The pH and the conductivity are measured in μS .
- 4 The salt content (Z) is calculated in g. Depending on the temperature of the solution two formulas are applicable.
At 25°C $Z = H \times k$
At 20°C $Z = 0,9 \times H \times k$ (according to the calculation of $Z = H \times 0,0527$)
Where H = conductivity at 25°C
 k = coefficient to calculate the weight (g) from $\mu\text{S} = 0,0586$.
- 5 The percentage of the salt is calculated
 $Z \% = Z/m \times 100$ where m = the weight of the stone sample (g).

This method estimates the salt content by making several approximations and is therefore not completely reliable. The percentage reached must be regarded as a rough calculation. The salt ions are, for instance, not analyzed, which may cause distortions in the calculations as different salt ions provoke different conductivity.

Scientists at the Centrallabor at the Bayerische Landesamt für Denkmalflege in Munich also use destructive methods to analyze salts in buildings. They drill small holes into the substances (4–5 mm) and collect drill dust from different depths (0–3 mm, 3–6 mm and 6–9 mm, and so on). The material is leaked and quantitatively analyzed using IC-analysis and AAS-analysis. Bläuer Böhm in Switzerland discusses a water extraction method used to calculate the quantity of salts. The method is also destructive since it is performed on drilled core samples. Her article describes the methodology of water extraction in detail and demonstrates how to interpret the results. Her conclusion is that the measurement of the electrical conductivity of the solution in relation to the loss of weight of the sample after the salt has been extracted gives a very good indication of the amount of salt in the sample. [91]

The Löfvendahl method – Geochemist Runo Löfvendahl at the NHB uses a non destructive methodology to evaluate the concentration of soluble salts in porous material similar to the Borelli method. The aim of the method is to give a preliminary indication as to whether there are enough salts in the stone to cause damage. The methodology is as follows:

- 1 Clean cellulose wadding is folded three times (the wadding being 21x15 cm) and applied to the surface with a brush using distilled water.
- 2 After an hour the wadding is removed, put into a plastic bag, and kept in the refrigerator until further tests can be conducted.
- 3 In the laboratory, the wadding is put into a bowl and immersed in 300 ml distilled water. The plastic bag is rinsed twice using 50 ml distilled water each time. At this point there is 400 ml water in the bowl. The solution (with the wadding) is left at room temperature for at least three hours. The solution is thereafter filtered using a water suction device. The bowl is cleaned with 50 ml water that is added to the rest of the solution, together with another 45 ml that is poured on the wadding. After this, 250 ml of the water is poured into a glass container and the temperature measured. If the temperature doesn't reach 25 °C the conductivity meter has to be corrected to the accurate temperature (the result is later recalculated to 25 °C). The conductivity measured with the conductivity meter is written down in $\mu\text{S}/\text{cm}^2$ (the NHB uses an YSI Model 33 S-C-T). The pH and alkalinity are also measured at 25 °C. It is especially important to measure the alkalinity in core samples since the carbonate concentration may vary depending on the depth. Löfendahl decides that further chemical analysis is needed to determine the salts when the conductivity exceeds 300 $\mu\text{S}/\text{cm}^2$ (using a Liquid Ion Chromatography, LIC). [92, 93] This is the limit at which the salts are considered to pose a danger for the stone material and consequently have to be removed.

Table 2. Table of NDT Methods for Analyzing and Measuring the Salt Content in a Material

NDT Method / Instrument type	Measuring area	Instrument's weight, accuracy, range, speed, and resolution	Possible to use outdoors	Needs personnel trained in science	Use in conservation	Expensive/inexpensive
Neutron Probe	Depends on the instrument.		YES	YES	Rare	Expensive
The Borelli Method	The method uses 150 cm ² pulp and 200 ml of water. The pulp and water relation is: 1/20	It is important to follow the instructions.	YES	NO	YES	NOT very expensive
The SKANSKA Method	Calculates the salt percent of 100 ml of water and 1–2 g of the material.	The method has problems with approximations.	YES, but destructive	NO	YES	Inexpensive
The Löfvendahl method	21x15 cm (315 cm ²) wadding, a total of 700 ml of water.	It is important to follow the instructions and the scale has to have three decimals. It can be hard to find the Polish wadding. The limit for further analysis is 300 $\mu\text{m}/\text{cm}$.	YES	NO	YES	Inexpensive
The Bläuer Böhm Method	See article.[91]	It is important to follow the instructions and the scale has three decimals.	YES, but destructive	NO	YES often	Inexpensive

4.3 Methods for Measuring the Water Absorption of the Material

Pipe Measurements; the German Karsten pipe, the RILEM pipe, the Italian pipetto and the Polish Mirowski pipe. All the above mentioned are non destructive NDT methods that can be used *in-situ* to evaluate the water absorption of a porous material under low pressure. The water absorption of the material corresponds to the pore structure of the material and gives information about the condition of the material. The pipe methods are used to evaluate the result of conservation; often a hydrophobic treatment. Standard laboratory tests are frequently used to determine water absorption, for example, the ASTM and DIN standards for water absorption. These tests are not possible to use *in-situ*, however, and are consequently not discussed here.

The *Karsten pipe* was developed in 1958–1960 in Germany. The method is non destructive and easy to use *in-situ*. It is thus often used by conservators-restorers. The pipe is made of an open cylindrical body that is attached to a surface (horizontally or vertically – there are two kinds of pipes for these purposes) of the material to be measured. A graded pipe, filled with distilled water, emerges from the cylindrical body. The water absorption is determined with the help of a time-bound schedule. The water absorption is registered according to the gradations on the pipe and the time. The pipe always points upwards and the body is attached to the surface by means of a sticky gum. Plastic FERMIT is often recommended to attach the pipe. In Sweden a Bostic sticker has been used for a many years, although as it is hard to find nowadays other sticky gums have also been tried. There are two sizes of pipes: one holding 4 ml and one holding 10 ml of water. The larger pipe body's inner diameter is 4.4 cm, the thickness/height of the body is 3 cm, the total height of the pipe is 19 cm and the pipe's inner diameter is 0.9 cm. The smaller pipe's body has an inner diameter of 2.5 cm and the thickness/height of the body is 2.4 cm, while the total height of the pipe is 15 cm and the inner diameter of the pipe is 0.9 cm. The differences in size facilitate measurements on different kinds of stones. The larger pipe was used for absorbent stones and the smaller one for less absorbent stones.

Methodology: The pipe is properly attached to the stone so that it will stay fixed for at least one hour. It is important that the sticky gum doesn't spread into the body's volume in such a way that it diminishes the contact area of the water. The pipe is filled with distilled water until it reaches 4 or 10 ml. The measurements are made according to a prefixed time-schedule: after 1 minute it is noted how much water has been absorbed and the same procedure is then repeated every minute for 5 minutes and then noted every 5th minute for 30 minutes. The measurements continue for 60 minutes and the absorption is noted every 10th minute in the last 30 minutes.

The results can be plotted on a graph (where the absorbed volume is a function of the time) or by calculating the capil-



Figure 2. Measurement with Karsten pipe at Riddarhuset.

lary water absorption coefficient – the w-value ($\text{kg} / \text{m}^2 \times \text{h}^{0.5}$) that is comparable to the DIN-standard to the water coefficient that is measured on drill cores DIN 52 617) and the water penetration coefficient – the B-value ($\text{m} / \text{h}^{0.5}$). The mathematical model for this was developed by Wendler in 1989 and is commonly used in Germany. [94] Both these values depend on the porosity and capillary force of the material. The BASIC-computer program is used to calculate the w-Value when using the Kartsen pipes. [95]

The *RILEM pipe* is similar to the Karsten pipe. It was tested by RILEM but never became a real standard and is thus no longer available. It nevertheless sometimes occurs in the literature. The *Mirowski pipe* was invented by Ryszard Mirowski at the Laboratorium Naukowo-Badawcze (PP PKZ) in Torun, Poland. It differs somewhat from the Karsten pipe since the tube is closed and the water is carefully measured before poring into the tube. It is not used very often and there is a risk of default values caused by low pressure since the tube is closed. The *Italian pipetto* is another similar method. Made in Plexiglass the contact area is 3,7 cm^2 and has a graduated pipe that is filled with water. The pipe is attached to the surface by clamps or sometimes by sticky gum. [96]

It has been suggested that there are problems with the pipe methods because they are not always repeatable, which means that the results can differ depending on the dexterity of the person conducting the measurements. [97] The pipes can also leak or even fall off during the measurement. However, as Wendler and Snethlage have proved that the measurements are reproducible and that the method is thus reliable, the pipes are often used in stone conservation. [94] The methodology gives good results, is cheap and relatively easy to handle. Thus it has also been tested within this project.

The *contact sponge test* is a new method that measures the water uptake of a porous material. It is designed to assess the effect of conservation treatments in the field, and

has been developed by the Italian conservation scientist Piero Tiano at ICVBC-CNR. In his opinion, the method is easier to use than the Karsten pipe. The method is based on the absorption of water of a wet circular sponge (made of natural fibres, diameter 55 mm, Spontex type Calypso) pressed towards a porous material. The sponge is weighed both before and after application. Either the circular plastic container provided with the test (1034 Rodac Plate produced by Falcon) or a prefabricated dynamometric spring (made by SINT) can be used for pressing the sponge towards the surface. It is important that equal pressure is applied and that can be obtained by pressing the container towards the surface (with the wet sponge inside) until the borders of the container hit the surface (maximum pressure). The sponge is saturated with different amounts of water depending on the absorption of the material being tested. The precise amount of water, the pressure and the time that the sponge is pressed all have to be tested before the actual testing starts. This initial test is made on the object by adding as much water as possible to the sponge without it starting to drip during the measurement. Moreover, the test can be appropriated to the stone by pressing the sponge for shorter or longer periods. The precision of the test depends on the scale, which has to have three decimals. Humidity and temperature influences the method and the test can therefore only be compared with other measurements made on the same day and in the same climatic conditions. Hence it is important to have one reference area and one treated area, both of which are measured on the same day so that comparisons can be made.

Tiano has tested the method with and without a spring, both in the laboratory and in the field. He also tested the method on several materials, including brick and stone. With brick he used 5 ml of water that was held for 60 s using the pressure of 770 g at 23 °C. When he repeated the test the results were very similar. He then tried using different quantities of water and with different pressures for shorter

or longer periods of time, and found that during the shorter periods the low porosity materials hardly absorbed any water at all. On the other hand, the very porous materials initially absorbed a lot of water. When pressing the sponge for longer times, the differences between the two tests were less.

Methodology for the Contact Sponge test *in-situ*:

- 1 The container (with the tap) and the dry sponge are weighed together (P_{dry}).
- 2 The sponge is filled with distilled water until it is wet. It is wiped with absorbent paper until it is almost wet.
- 3 The sponge is put back into the container and weighed (P_{wet}). The amount of water (Q_{wet}) is calculated ($Q_{wet} = P_{wet} - P_{dry}$).
- 4 While the container is still on the scale, water is poured in until it reaches the previously determined and desired quantity ($Q_{desired}$) (i.e. in tests that decided which pressure, how much water and how long the test was to be conducted). The container is then closed and weighed ($P_{initial}$).
- 5 The sponge is pressed to the material (a flat surface) with the desired pressure during the pre-determined time.
- 6 The sponge is sealed in the container and the final weight (P_{final}) measured *in-situ*. The result is calculated mathematically. The quantity of water (W_a) absorbed by the material is calculated: [97]

$$W_a (g/cm^3 \times min) = \frac{(P_{initial} - P_{final})}{23.76 \times t}$$

The contact sponge method has not yet been tested by the NHB.

The *permeability box method* is another *in-situ* water absorption test that is sometimes used in conservation. It consists of an apparatus with a flat chamber which, when applied to a wall, will isolate a rectangular surface of penetration that measures 16 x 34 cm. After placement, the

Table 3. Table of Methods for Measuring the Water Absorption of the Material

NDT Method / Instrument type	Measuring area	Instrument's weight, accuracy, range, speed, and resolution	Possible to use outdoors	Needs personnel trained in science	Use in conservation	Expensive/inexpensive
The Karsten pipe	Measures a flat surface area; two kinds are available; one is 2.4 cm in diameter and the other is 4.4 cm. The amount of water is 4 ml or 10 ml.	The result is repeatable according to Wendler and Snethlage. The large pipes measures one hour the small 30 minutes.	YES	NO	YES all over the world.	Inexpensive
The contact sponge test	Measures a flat surface area. The diameter is 5,5 cm.	The results are repeatable. A scale with three decimals is required and a spring to calculate the pressure. It is fast (ca 1 minute), however time-consuming pre-work is needed.	YES	NO	YES in Italy	Inexpensive – however can be hard to find the material outside Italy.
The permeability box method	Measures a flat surface; 16 x 34 cm	—————	YES	NO	Rarely	—————
Micro drop measurement	The test is made on horizontal flat surfaces. The area is small and the distance between the drops is 2 x 5 cm.	The method is quite fast and accurate.	Conducted in the laboratory	NO	YES all over the world	Inexpensive

chamber is filled with distilled water through an opening at the top. Once filled with water the level is maintained by using a Mariotte tube. This tube constantly compensates the flow of water percolating through the wall. The quantity of water that has penetrated the wall is read directly on the tube which is graduated in cubic cm. [98]

Micro drop measurement is a laboratory water absorption test that, despite being infrequently used *in-situ*, is often used in conservation. The test measures the absorption time of a fixed quantity of water on a horizontal surface. The samples to be measured are left in the same climatic conditions in which they have been stored for several days. Distilled water is dropped from a height of 1 cm above the sample. The quantity of the drops is usually $10 \pm 2 \mu\text{l}$ and the dropping distance is $2 \times 5 \text{ cm}$. The test is usually conducted five times. The time it takes for the drops to be completely absorbed is then measured. [59]

4.4 Methods for Investigating the Inner Structure of the Material

Several NDT methods can be used for investigating the inner structure of a material. The methods described here are related to those methods that measure the strength of the material (both use energy and waves). However, as the aims of methods are different they have therefore been divided in this report. Most of the methods described below generate pictures of the inner structure of the object that can help to locate structural flaws in the material. The methods can give quantitative results even though this is not so common. Many articles in conservation publications discuss these methods and they are briefly described here, even though they are expensive. The methods are based on different wave and energy phenomena:

- 1 short wave energy, such as X-ray, gamma or neutron waves.
- 2 electrical pulse energy, such as ground penetrating radar.

The techniques are based on penetrating radiation measurements of physical structures and measure the attenuation of a beam of particles as it is transmitted through the material. The attenuation follows Beer's Law, which states that the intensity of the beam decreases exponentially with the distance travelled through a material. [99] The methods will only be briefly described in this report since they have not been tested in this study and are usually quite expensive. Several of the methods are described in more detail by Anders Bodare. [33] Most of the techniques are relatively expensive, difficult to find and require experienced personnel.

X-ray, neutron or gamma techniques, such as *radiography (photographs)*, *radioscopy (with a detector such as the image intensifier)*, and *Compton X-ray backscatter techniques* (see also tomographic and holographic techniques). The X-ray methods were discovered by William Röntgen in 1895. The roentgen and gamma rays are short-wave radia-

tion. The wavelength depends on the voltage used to drive the electrons. Higher voltages give shorter wavelengths that penetrate the object more. The X-ray technique was already being used in the field of conservation at the end of the 19th century. The method detects flaws in the material and creates images of the inner structure. The classic radiography results in two dimensional photographs. This technique has problems with overlapping when used on three dimensional stone objects. A 3D picture can be obtained using computed tomography (see below). Portable penetration radiation instruments are available for use *in-situ*. The traditional X-ray techniques are transmissive and contact with both sides of the object must be established. The Compton X-ray can be used with access from only one side. The results depend on the attenuation properties of the material, the imaging technique and the radiation energy. Results are best on organic material. Inorganic materials, such as steel or stone, have comparatively high attenuation coefficients and X-rays do not penetrate deeply. As a result the X-ray technique has often been used to study wooden structures.

Electrical methods such as *ground penetrating radar (GPR)*. The GPR technique makes use of electrical and short electromagnetic pulses within the radio frequency area. It is a NDT method that can be used *in-situ* and is consequently sometimes used in conservation to survey buildings or sculptures. The GPR can be valuable in detecting flaws and also for assessing deterioration of the stone.

When the electric pulse hits a material that conducts electricity differently (depending on the dielectric constant, as well as temperature, pore structure, moisture content, and salt content) some of the energy is reflected and some is transmitted, and the radar system measures the time it takes for the reflected energy to come back. It is important to know whether salts and water are present since this affects the result. The GPR instrument repeats the measurement while the antenna is moved and the signals are registered and plotted in a graph that depicts a profile. The choice of frequency will determine the precision in detail.

The technique is well established in geophysics and engineering and was introduced in Sweden by Ulriksen in 1982. Possible uses of the method in conservation are described by the German Gabriele Patitz. [100] One problem with the method is that the distances that need to be detected in conservation are smaller than the wavelength of the waves in normal instruments. More energy is needed to detect smaller objects. Some examples of the use of GPR in conservation include the US Capitol, the Colosseum in Rome, Hamar Cathedral in Norway [33] and the Burg Stolzenfels south of Koblenz in Rhein Pfalz in Germany. [100] It has recently been used by Moropoulou in Greece to investigate buildings, together with other NDT techniques such as ultrasound velocity, infrared thermography and fibre optic microscopy. [101] It has also been tested on the "Heiligen Halle am Markt von Priene" to search for metals inside the structure in conjunction with stereoradiographic and videoscopic measure-

ments. In this case, the radar was able to establish where the marble and the mortar met and to localize metallic nails. It was not possible to establish the exact form and dimensions of the metals with the GPR, however. For this purpose the radiographic method was better. [102] The Royal Institute of Technology in Sweden has tested electrical methods to define the weathering of stone (see above). [40] Such electrical techniques can also be used to determine the moisture content of a building or material (see below). [32]

Tomographic methods, such as Computer Tomography (CT) using neutrons, acoustic parameters, electrical signal and/or x-rays. Tomographic methods are used to create images that demonstrate both the interiors and flaws in materials. The technique provides an image of a selected plane of the object in a 3D object. Different waves can be used for the purpose, such as neutrons, ultrasonic, electrical, gamma or x-rays. [103] It is frequently used in stone conservation and can be used *in-situ* – the pictures can be macro or micro depending on the waves used.

With maximum resolution, tomography demonstrates the numeric value of the specific physical property in each zone of a rock mass inaccessible to direct measurement. The zone to be studied is divided into a matrix of regular cells of predetermined size and geometry. In a two dimensional tomography these are squares (in pixels), and cubic (in voxels) in three dimensional ones. The final results in a tomography are thus numerical values that cover the whole rock mass. It is thus a two or three dimensional representation of variations inside the rock mass, depending of the structure of the stone. In practice, it involves the transmission and reception of signals that travel through the material from positions where the coordinates are known. These are interpreted by the computer into numerical values that are then plotted in images. [104]

The first example of tomographic pictures was conducted in 1935 in Germany by Kallman. The technique was developed in 1970s by Hounsfield into computerized transversal axial tomography (CAT scanings) and used in medicine for the examination of organic materials. Tomography has also been used in conservation. There are examples from laboratory tests as well as from *in-situ* using portable instruments – both using ultrasonic and X-ray waves. One example is the X-ray computed microtomography (μ CT). This is based on the attenuation of X-rays from different positions of an object and provides a resolution of 12 μ m. It thus can help to interpret images in terms of a stone's microstructure and thus monitor the deterioration of stone. However, it is still necessary to take samples that are "tomographed" in the lab. [105] The CT methodology was used by Illerhaus and co-workers to reconstruct hidden surfaces in objects. [106] A further example is the use of ultrasonic tomography to characterize the state of conservation of Megaliths in Spain. In this case the method was also used *in-situ*. The aim was to assess the state of deterioration and it was very useful. [104] Stefan Simon has also used ultrasonic tomography to assess the state of deterioration of marble. This was conducted on the marble base of the Gustav Adolf sculpture in Gothenburg. [38] Another example is Atkinson's and Schuller's acoustic tomographic study on masonry – both on test brick piers and on site on the Red Armory and Gymnasium at the University of Wisconsin in the USA. They found that the technique was easy to use and interpret and that the necessary computer power was moderate. The drawback was that the work was labour intensive. [107] The technique is thus expensive and requires experienced personnel, even though it is developing rapidly and may become more popular with more advanced computer modelling and visualization software.

Table 4. Methods for Investigating the Inner Structure of the Material

NDT Method / Instrument type	Measuring area	Instrument's weight, accuracy, range, speed and resolution	Possible to use outdoors	Needs personnel trained in science	Use in conservation	Expensive/inexpensive
X-ray, neutron or gamma techniques	Usually measures through the material (access from both sides or one side – back scatter).	There are several portable instruments. The resolution depends on the instrument. The method is fast and accurate.	YES	YES	YES	Expensive
Electrical methods Ground penetrating radar (GPR)	The entire size of the object.	There are small fast instruments (ca 2 kg). The properties and accuracy depend on the instrument.	YES	YES	YES, also used in Sweden	Expensive
Tomographic methods; Computer Tomography (CT) - Tomography using neutrons, acoustic parameters, electrical signal and/or x-rays	The entire size of the object.	Depends on the instruments and the type of radiation used; time-consuming.	YES	YES	YES	Expensive
Holographic methods such as ultrasonic, X-ray and digital holography and Microscopic ESPI.	Depends on instruments that can be used at the micro-level.	Depends on the instruments; time-consuming.	YES	YES	YES	Expensive

Holographic methods, such as *ultrasonic*, *X-ray* and *digital holography* and *Microscopic ESPI*. *Holographic methods* are rarely used in conservation. They have their origin in the field of light and optics and can make use of all waves for real-time visualizations of three-dimensional images of internal discontinuities. The methodology is thus a kind of advanced imaging and can be used for the assessment of flaws and deterioration of materials. *Microscopic ESPI* can monitor micro deformations of objects of a few cm and up to several metres. In conservation, a holographic contouring technique has been used by Paoletti and Schirripa Spagnolo to study marble erosion. [108] The technique is not used very often since it is difficult to use *in-situ*, is time-consuming, expensive and requires experienced personnel.

4.5 Methods for Identifying and Analyzing Substances at the Surface

The following NDT methods can identify and analyze substances at the surface or a few millimetres in depth. The methods are often referred to as *micro-analytical methods*, since they can determine substances at the microscopic level. Such methods are frequently used in the conservation of art objects in the museum environment and are rarely used on buildings. Nevertheless, as the instruments are developing quickly, some of the methods are now available for use *in-situ*. Some of the methods will therefore be described in this report, even though they have not been used in this particular study. The MOLAB project (part of the EU-ARTECH, see above) makes it possible to utilize some of these methods *in-situ*. Through MOLAB, conservation projects in European countries can make use of portable instruments hosted by some of the distinguished conservation laboratories in Europe.

Several conferences deal only with *NDT micro-analysis* in conservation, such as the ART; *International conference on non-destructive testing and microanalysis for the diagnostics and conservation of the cultural and environmental heritage* (the 7th Conference was held in Antwerp in June 2002). Furthermore, specialist conferences and periodicals are available for detailed studies of some of the methods. One specific example is the conference dealing with the use of Raman microscopy in conservation.

The techniques are based on *spectroscopic methodology*, i.e. the study of radiation (electromagnetic radiation of different wavelengths or particle beams) that is absorbed or emitted by the material. The material radiated emits some type of radiation which causes it either to absorb the incident radiation at certain frequencies or to emit some type of radiation of its own. The intensity of the observed signal is plotted as a function of wavelength to provide a spectrum, and each element causes its own characteristic set of peaks in the spectrum. Hence the spectroscopic methods are used for chemical characterization.

LIDAR technology has already been discussed and is therefore only described in brief here. LIDAR (Light Detection and Ranging) is a laser radar that can measure reflections from surfaces at a distance. The laser beam is directed towards a goal, the technique measures the beam that is reflected and displays the results on coloured maps. Laser Induced Fluorescence (LIF) detects the fluorescence signal that is emitted from the surface (being hit by the laser beam) of the material. The result gives a spectral image that thereafter can be analyzed and transformed into a false-coloured image. The technique can help to discriminate different materials and algae and biological growths. Although the technique is under development, it is still difficult to evaluate the results. It is also quite expensive and requires experienced personnel. It has been used by Swedish scientists at Lund University on Lund's cathedral, on the cathedral in Parma and on the Coliseum in Rome. [47]

Portable Ultraviolet light methods such as the *fluorescence lamps* (registered with a camera), *fibre optics UV-VIS fluorescence* and *FLIM Fluorescent lifetime imaging and spectroscopy* with an *analytical spectroscopy unit OMA* (Optical Multi Channel Analyzer).[109] Ultraviolet techniques use wavelengths close to those of visible blue light, which are slightly shorter. The light is therefore invisible and can provoke temporary modifications (by absorption, excitation and emission of energy) on the surface of the object illuminated. The phenomena can both be detected by the human eye – ultraviolet fluorescence – and by receivers outside the visible area. The UV-VIS fluorescence and UV-reflection analytical method creates real time images and photographs. The method is used *in-situ* for the identification of organic (and some inorganic) substances and reveals something of their nature. It can also be used to discriminate retouching. The technique is useful as a first approach to the study of organic substances and to guide micro-samplings for chromatographic or stratigraphic studies. It is also used on stone, for example, to analyze the surface deposits on Michelangelo's David. [110]

FLIM (Fluorescent lifetime imaging and spectroscopy) is a new innovative NDT technique in bioscience based ultraviolet radiation. It is used by the ICVB-CNR in Florence *in-situ*. The technique has been developed because of the difficulty in quantifying the fluorescence intensities in traditional ultraviolet techniques. It provides two kinds of data: the *image frequency-domain* and the *image life-time domain* of the fluorescence in the area examined. [111] It differs from traditional fluorescence analysis where the intensity is measured. In this case, the lifetime of the fluorescent signal is measured and, at the same time, it is possible to record the UV fluorescence spectrum at any given point. The frequency-domain measurement can also help to separate mixtures of substances. By changing the phase of the reference signal it is possible to spatially separate the lifetime of each. The measurements must be correlated with micro samples

taken from the surface that are analyzed to establish the chemical compounds (references in the laboratory). FLIM helps to identify pigments and organic materials. A portable instrument has been used on Michelangelo's David to verify that the cleaning work was properly conducted.

Portable infra red light scanning / photography, IR-colour reflectography and IR thermography. [112] The infra-red radiation uses wavelengths that are close to the visible red area. IR-colour technology helps to characterize painting techniques through the identification of under-drawings and the identification of undocumented interventions and the characterization of pigments. The method is often used to see under-paints such as drawings and pigments and is thus seldom used in stone conservation. Another technique, this time using longer wavelengths, is *IR thermography*. This is often used to find moisture in buildings (see below). *Passive thermography* is used to detect organic materials at the surface, such as oils, waxes and patinas, while *active thermography* is used for detecting structural defects. For this purpose it has been used on Michelangelo's David. [110]

Portable IR-Spectroscopy (PIMA Portable Infrared Mineral Analyzer SP Infrared Spectrophotometer) is another recently developed NDT technique that can also be used *in-situ*. It can be used for the identification of the composition of mineral building materials, to investigate their deterioration and to analyze salt and gypsum problems. Some of the problems with the technique are that not all minerals have IR-spectra, such as quartz and feldspars, and that the spectral information can overlap in mixtures. Moreover, interpretation of the spectra has to be carried out with the help of databanks with spectral information that does not always exist. The method has been used by conservation scientist Angela Ehling to investigate marbles and to study the deterioration of stone (2004). [113] Ehling believes the method is good for NDT examination of complex salt situations together with a Georadar. Köhler (2002) has also used it for the examination of salts. Blanco and co-workers examined salts in deteriorated mortars, grouts and plasters with a non portable technique in Spain in the 1990s. [114]

Portable XRF (X-Ray Fluorescence) instruments and portable ED XRF Energy Dispersive X-Ray Fluorescence. [115] This technique conducts an elementary qualitative analysis that excludes light components such as hydrogen, oxygen and nitrogen. The instrument can also be used for elemental analyses (elements >12) on stones, metals, wall and easel paintings and other objects. It encounters some problems with the quantitative measurements when a lot of matrixes are present, which means that only a qualitative analysis can be performed. The technique is thus complementary to FT-IR and micro-Raman spectroscopy. A portable XRF has been used in the Chapel of Scrovegni in Padua. A portable ED XRF instrument was used on Michelangelo's David to investigate and map the gypsum sulphation.

Another example is the examination of the *Portale della Sagrestia Aquilone Nord* in Milan. Here the data from the XRF was integrated with a computerized 3D model. [116] The technique requires experienced personnel and is thus expensive.

Portable FT-IR Fibre optics [117] is a technique that gives spectrums with characteristic peaks for each molecule and identifies molecules and organic compounds. It is the only NDT technique to give information about compounds. It can identify alterations on stones, mortars and metals and identifies pigments and binders in paintings. A portable instrument was used to investigate Michelangelo's David. The portable FT-IR instrument is not yet as exact as the laboratory technique and the quality of the spectra is inferior to those of the laboratory. One needs to take samples as a complement. The technique is expensive, requires experienced personnel and, in addition, the instrument is relatively heavy (35 kg).

Portable Micro-Raman spectroscopy is a technique that consists of portable equipment for *in-situ* studies of inorganic and some organic substances on stones, metals or polychrome surfaces. It can identify alterations on stones, mortars and metals as well as pigments in wall paintings. The technique is complementary to FT-IR and X-ray fluorescence. Another NDT method that has been developed for portable use is the *PIXE technique (Particle Induced X-ray Emission)*. This is used to analyze elementary substances of objects of art. PIXE builds on a technique that detects the characteristic roentgen radiation emitted by a substance when it is radiated with ions. Both of the techniques are expensive and require experienced personnel.

4.6 Methods for Analyzing the Strength and Mechanical Properties of the Material

Methods for analyzing the strength of a material or structure are usually connected to mechanical wave methods and/or the drill hardness methods. Some of these are among the most frequently used NDT methods in stone conservation. As geophysical methods they are well described by Anders Bodare and also included in many engineering journals, such as the *Journal of Geophysics*. [33]

Ultrasound pulse velocity (UPV) methods are now well established in stone conservation, which is why many of the articles mentioned in this study relate to this technique. It analyses the structure of the material and thus only indirectly the hardness of the material. Many studies have tried to correlate the compressive and tensile strength to the UPV measurements. Some researchers are especially connected to the UPV methods in stone conservation, such as the Frenchman Mamillan, the Spaniard Montoto and Germans Köhler, Weber, Simon, Weiss and Siegesmund. The main reason for the method's popularity is that it is well established in civil engineering and is rapid and easy to use

Table 5. Methods for Investigating the Surface of the Material

NDT Method / Instrument type	Measuring area	Instrument's weight, accuracy, range, speed and resolution	Possible to use out-doors	Needs personnel trained in science	Use in conservation	Expensive/inexpensive
LIDAR technology / LIF (Laser-induced fluorescence)	Measures small spots from a distance or large areas with a whisk-broom scanning technique.	Can measure from distance, it is labour intensive and the resolution and speed depends on the laser technique and the different wavelengths used.	YES	YES	Very little.	Very expensive, although the moveable equipment is available for research.
Portable fluorescence systems; UV-VIS fluorescence (sometimes with fibre optics) and FLIM Fluorescent lifetime imaging and spectroscopy with an analytical spectroscopy unit OMA	Depends on the instrument.	Depends on the instrument and the camera that records the phenomena. It is fast.	YES	YES	YES	Moderate to very expensive OMA can also be used with LIDAR technology.
Infra red light scanning and photography IR-colour reflectography	Depends on the instrument.	Depends on the instrument and the camera recording the phenomena. It is fast.	YES	YES	YES	Moderate to very expensive.
Portable XRF (X-Ray Fluorescence) instruments	Depends on the instrument	Depends on the instrument, not heavy, very fast (less than a second) and accurate.	YES; there are small ED XRFs from 1,4 kg	YES	YES	Moderate to very expensive.
Portable IR-Spectroscopy PIMA (Portable Infra-red Mineral Analyzer) IR Themography	Depends on the instrument.	Depends on the instrument. It is fast.	YES	YES	YES	Expensive
Portable FT-IR	Depends on the instrument.	Depends on the instrument. Fast.	YES	YES	YES	Expensive
Portable Micro-Raman spectroscopy Raman spectroscopy has a conference for the use in conservation![118]	Depends on the instrument.	Depends on the instrument.	YES	YES	YES	Expensive
Portable PIXE technique (Particle Induced X-ray Emission).	Depends on the instrument.	Depends on the instrument.	YES	YES	YES.	Expensive

in the field. Portable equipment is also available. In this project the method has been tested in the laboratory as well as in the field.

Many of the methods use mechanical waves (*the sound methods*), such as *seismic methods*, *ultrasonic methods* (ultrasonic pulse-eco, the ultrasonic transmission – these can also be sorted under the seismic methods – see also ultrasonic holography and tomography), *hammer methods* (Schmid, Brinell, Vickers and Bercovich and so forth) that measure the surface and *acoustic emission methods*. Some methods used for investigating the status of the material are slightly destructive. One example is the flatjack *in-situ* measurement. A flatjack is a thin steel bladder that is pressurized with fluid to apply a uniform stress over a small area. Large flatjacks used to determine the *in-situ* state of stress and deformability of rock were modified for masonry structures in the US in the 1980s. Several ASTM standards are used to evaluate the *in-situ* compressive strength (ASTM C 1196) and for the evaluation of a masonry's deformation proper-

ties (ASTM C 1197). [89] Similar methods include the locally destructive shear tests and pull-out tests – these methods are not discussed here.

Sound methods (mechanical methods) use mechanical waves that are usually transmitted through a material. They therefore employ physical stress waves. Acoustic technology has a long history in material science. The simplest methods involve the use of a hammer to tap the material and then listening to the sound. Sound techniques were improved during World War I. In 1942, the American, Floyd A Firestone, received a patent for a Reflectoscope instrument and since then there has been a steady development. The ultrasonic method for stone characterization was established in the 1950s. The acoustic methods help to evaluate the condition of the stone (the state of conservation), provide information about the result of consolidation treatments and can help to detect flaws in the material, such as cracks and voids. They moreover help to characterize the mechanical properties and thus the materials' degradation.

The classic sound method involves the transmission of a pulse of sound of a single frequency into a structure. The time required for the pulse to be reflected back from a feature such as a void is measured; the higher the frequency of the sound, the smaller the distance that can be measured. [99] The waves are actually a kind of transmitted energy that moves in different but typical patterns. The speed of the energy is the *speed of wave* or *wave velocity* (mm/s or km/s). Actually it is the individual particles that move around their points of equilibrium and retain their position after the wave has passed that are measured. The mechanical waves move in different ways in relation to the direction of the propagation – *longitudinal* or *transversal*. The longitudinal wave travels at a higher speed and is named by the *P-wave*. The transverse wave is slower and is called the *S-wave*. There are also so called surface waves that travel along surfaces of discontinuities between materials, such as the *Rayleigh wave* (*R-wave*) where the waves move the particles in ellipsoidal orbit (the speed is 93 % of the S-wave) and the *Love-wave* (*L-wave*). The speed of the waves correlates to the strength of the material. The waves travel at the same speed in all directions if a material is homogenous and isotropic, but the speed may be different in different directions if the material is inhomogeneous or anisotropic, such as sandstone. Another effect of the property of the material is of importance, namely, the *attenuation*. In the short-term, the consequence of the attenuation is that the amplitude of the wave diminishes as the wave propagates. That is determined by Baer's law. It is also important to know that the velocity is faster in denser material and slower in air. [33]

The *seismic transmission method (mechanical pulse)* harnesses the transmission of body waves (S or P) from one point to another. The mechanical wave is generated by a surface impact and the pulse is recorded using one or more accelerometers. [89] The method introduces a short pulse from an emitter, for example, a hammer blow (or steady-state vibrations) which are received and the travel time calculated (or phase differences between two signals in case of the steady-state vibration). Measuring the material in several locations and having different travel times may be caused by inner structural differences. The method has been tested on the Gustav II Adolf sculpture in Gothenburg. The problem with the method is that it only gives the average value of the wave speed and it is not possible to detect precisely where the damage lies (if not combined with a tomographic methodology). [33] This method is not very common and requires experienced personnel.

The *seismic refraction method* uses refraction of the P-waves. It is used on stone or masonry that consists of two different materials. According to Bodare, the method has not been used in stone conservation and no article could be found within this study. The *seismic refraction method* is similar to the GEO radar method, although uses mechanical instead of electrical waves. [33] The *impact echo meth-*

od also uses mechanical wave pulses that are introduced by a hammer similar to the seismic reflection method, this time measuring the frequency of the motion rather than the travel time. It is also described by Bodare and was used on Gothenburg's Gustaf II Adolf sculpture in 1994. [33]

More frequent is the *acoustic emission (AE)* method, which can be used to analyze composite materials. It consists of transient mechanical vibrations generated by the rapid release of energy from localized sources in the materials. The AE can be recorded and analyzed on the surface. An acoustic emission is caused by micro cracking or dislocation movement. The method is used to study earthquakes and can be very sensitive; the instruments being available commercially. The method can detect different problems inside the material by use of compressive stresses, tensile stresses, bending stresses, thermal stresses, swelling stresses and other stresses caused by salts and so forth. AE has been used by Montoto and co-workers on the church of Saint Zeno in Verona, the statue of Marcus Aurelius in Rome and medieval Polish castles, etc. [33, 119]

Ultrasonic methods use ultrasonic waves and consist of several methods such as *ultra pulse velocity UPV*, *ultra pulse echo*, *seismic echo* and *acousto ultrasonic* (also see *tomography*). The vibration uses waves of a higher frequency than can be heard by the human ear – usually around 20,000 Hz. The velocity in air is 330 m/s, and in stone up to 6000 m/s. In the field it is usually the P-wave that is measured. The methodology of UPV is very similar to the seismic reflection method (and thus GPR, see above). The velocity depends on the properties and the structure of the material. The denser the material, the faster the sound travels. By measuring the differences it is possible to detect where the damage is located. When there is a crack in the material the signal is reflected and the stronger the differences of the material, the stronger the reflection. [120] By measuring this it is possible to assess the degree of deterioration of the stone. It is used to measure the velocity transmitted through the material, on the surface. [121, 122]

The UPV method is well established in civil engineering for detecting cracks and flaws and to control concrete. The testing of stone with ultrasonic wave measurements started early in the history of stone conservation. The testing was developed in the 1950s by the conservation scientist Mamilan in Paris. It was used in stone conservation in the 1970s and some of the results can be found in the '*1er Colloque international sur la détérioration des pierres en oeuvres*' held in la Rochelle in France in 1972. Another conservation scientist to use the technique was Rossi-Manaresi at the *Centre for the Conservation of Stone* in Bologna. [121] The method was, for example, used on the Trajan Column in Rome at the beginning of the 1980s, and in 1981 in Budapest. [123] The use of UPV increased rapidly so that at the 5th *Congress on the Deterioration and Conservation of Stone* held in 1985 in Lausanne, several articles dealt with the ultrasonic

measurement of stone. Today it is one of the most used NDT methods in stone conservation. Many articles using UPV have been consequently presented in the conference publications. One article of particular interest is that written by Chiesura and co-workers: *La technique d'auscultation micro-sismique por le diagnostic et l'evulation des traitements sur materieaux pierreux*. This article reviews the use of ultrasonic techniques in historic preservation until 1995. The authors found over 200 texts on the subject and 50 were selected for more detailed study. Two types of articles were found, namely, those:

- 1 Dealing with the ultrasonic technique to assess and characterize the stone and its deterioration (85 percent).
- 2 Dealing with the ultrasonic technique to assess the success of conservation treatments (23 percent).

Many of the articles dealt with samples studied in the laboratory. One conclusion was that there were many articles that dealt with the correlation between the UPV and other qualities of the stone, such as the compressive strength, porosity and so forth. This is natural since it is important to establish a correlation between these parameters and the UPV. Another conclusion was that until 1995 the ultrasonic methods had essentially been conducted for *in-situ* evaluations of stone and that the parameters of the limits of the techniques had not yet been studied. These parameters have recently been studied by Weiss, Siegesmund and others. In one article, *Die Schadensanalyse von Naturwerksteinen mittels Ultraschalldiagnostic: Möglichkeiten und Grenzen* (1999), the authors state that the decrease of velocity of the weathered stone cannot only be explained by the porosity increase. [124] Following his study of UPV and marble weathering, Weiss concluded that it is necessary to know the physical properties and fabric of the stone being measured in order to analyze the data. In marble, the V_p (the velocity of the P-waves) differs up until 30 percent depending on the anisotropy of the stone. The differences are less when the stone is saturated in water. This naturally complicates the *in-situ* measurements and is tested within this study. [125]

Chiesura and co-workers also noticed that several standards are available for ultrasonic measurement, most of them being found within the ASTM standards (see above). The *NORMAL 22/86* is the only one directed towards historic masonry. The standardization of the methods is a necessity, particularly as very few of the authors describe the techniques and methodology they use in detail. Chiesura consequently discusses some parameters that should be considered when one uses a UPV instrument in conservation:

- 1 To use punctual transducers (2 to 5 mm)
- 2 to use an instrument that visualizes the reception of the signal (an oscilloscope)
- 3 to use an instrument that both registers the emission and reception signal. [126]

Within the *Euicare-Euromarble* programme, several famous marbles in Europe have been tested with UPV techniques in an attempt to to understand the weathering. The Carrara marble has been especially tested. Köhler has thus established a classification of the state of deterioration of Carrara marble according to the UPV. This is based on an empirically derived correlated function between the V_p and the porosity. The same tests have also been performed on the Italian Lasa marble and on a Polish marble by Weiss and co-workers. [125] In Sweden the UPV has been used a few times; in the 1990s by Wolfram Köhler who examined the marble base of the sculpture of Gustaf II Adolf, by Simon on the Flora sculpture in the Botanical Garden in Gothenburg and by Bylund and co-workers on sculptures at the National Museum of Fine Arts in Stockholm. Moreover, laboratory tests with UPV have been conducted on some Swedish stones by Anders Rehn at the Royal Institute of Technology. [41] Katarina Malaga and Malin Myrin tested the UPV on conserved Gotland sandstone in 2004–2005 in connection with Malin Myrin's PhD thesis work on Gotland sandstone and conservation.

The equipment is expensive and requires experienced personnel. It has nevertheless been proven to be very useful in conservation and the method has therefore been tested within this project. One article states that the accuracy of the method is 0.5 percent. [126] Marini and others have tried the method in the laboratory on Carrara marble and found it to be satisfactory for weathering tests. [127] There are some drawbacks with the method, however, as one needs to know whether water is present (which affects the result), and in laminated sandstone the velocity usually differs depending on the direction being measured. Moreover it can be difficult to couple to rough surfaces as well as on complex shapes. One recent article also claims that there are problems when correlating the UPV data with the Uniaxial Compressive Strength. [128]



Figure 3. Ultrasonic measurements on the German church.

Hammer impact methods, such as the *Schmid*, *Brinell* and *Vickers hammer* (ASTM C 805–75), are slightly destructive but well established in civil engineering and relatively inexpensive. The Brinell and Vicker methods are constructed for the laboratory, while the Schmid hammer is used in the field. The drawbacks are that the methods only measure exposed surfaces and the hardness index is poorly correlated with the Uniaxial Compressive strength of the material. [128] However, the methods cannot be used for weak stones even though they were, for example, used in Germany to examine free stones in the Bamberg area in the 1980s and in Budapest to assess limestone deterioration. [129, 130] The hammer impact method has been tested by Anders Bodare at the Royal Institute of Technology. [33]

Drilling Resistance Measurement System (DRMS). Some European researchers, like Exadaktylos and Tiano, are currently developing the technique. [131, 132] The method has also been tested in a European project (see above). The method consists of a portable system that is essentially destructive, since the equipment makes small holes (5 mm) in the tested stones. The system makes it possible to determine the cohesion profile of a stone material based on data of its mechanical and abrasive properties. The tool has been developed to make it possible to determine either the degree of decay of the stone or the consolidation (the efficiency and

depth of penetration) of conservation treatments. Similar methods are also used in industry, such as the *Durabo Drill Hardness meter (DHM)*. This has been tested by the NHB but the results were not satisfactory. [14]

4.7 Methods for Measuring Moisture in Building Materials

Several methods are available for measuring the moisture content in building materials. Some methods are good for high water content, others for low water content, while some are only applicable in the laboratory and some in the field. The methods are usually divided into *direct* and *indirect* methods. The direct methods measure the moisture content directly by weighing the water in a sample (gravimetric methods). The indirect measurement measures properties in the material that can be related to its moisture content, for example, the electrical conductivity. Another way to differentiate the method is between *destructive* and *non destructive methods*. Within this project an electrical moisture meter has been tested as it is indirect and non destructive. The method has drawbacks, however, in that it is not reliable when salts are present and only registers the surface.

The *DGZfP (The German Society for Non-Destructive Testing)* discussed the measurement of water in building

Table 6. Methods for Measuring the Strength and Mechanical Properties of the Material

NDT Method / Instrument type	Measuring area	Instrument's weight, accuracy, range, speed and resolution	Possible to use outdoors	Needs personnel trained in science	Use in conservation	Expensive/inexpensive
Seismic transmission method (mechanical pulse)	-----	Depends on the instrument.	YES	YES	YES, but not very often	Expensive
Seismic refraction / reflection method	-----	Depends on the instrument.	YES	YES	It does not seem to have been used.	Expensive
Acoustic emission (AE)	Depends on the instrument.	Depends on the instrument.	YES	YES	YES	Expensive
Ultrasonic methods such as ultra pulse velocity (UPV), ultra pulse echo and seismic echo	Depends on the instrument.	Depends on the instrument. The method is fast and accurate.	YES	YES	YES, widely used.	Expensive
Hammer impact/rebound methods: Schmid hammer among others	The area is usually quite small and the impact force depends on the instrument.	Depends on the instrument. There are many products on the market. It is fast and lightweight (ca 1 kg), but micro destructive.	Yes, but is micro destructive.	YES	YES, not very often.	Inexpensive
Drilling Resistance Measurement System (DRMS)	Measures the resistance in 5 mm holes.	It is fast and not so heavy, but is destructive.	Yes, but is destructive.	YES	YES	Moderately expensive
Drill Hardness Meter (DHM)	—	—	—	—	—	—

materials at a symposium held in 1999 in Berlin. The publications resulting from this occasion are very informative and useful and can be accessed via the Internet. [133]

In building physics one differentiates between moisture content and moisture condition. The moisture content is the absolute content of moisture in the material. It is given as moisture ratio (mass of water per mass of material; u), moisture content (mass of water per volume of material; w) or the degree of capillary moisture saturation (S_{cap} , when completely saturated $S=1$). The moisture condition, on the other hand, is a measurement that takes into account how strongly the water is bound to the material. Water is sometimes chemically bound into the building material (for example in gypsum). This water is strongly bound and needs a lot of energy to be released, whereas water added by capillary absorption needs less energy. The chemically bound water is not normally a problem for the material. Measurement of the moisture condition makes it possible to study moisture transport in the material so that it is possible to state where the critical amount of water is for a specific material.

Moisture measurement methods are divided according to the methodology:

- 1 Absolute measurement methods (gravimetric measurements and chemical methods)
- 2 Hygrometrical methods
- 3 Electrical measurements
- 4 Other methods, such as the thermographic method, radar methods, microwave methods and methods using gypsum blocks with electrodes that are inserted into the wall.

All the methods will not be described in this report. Several of the methods are destructive, which means that tests are conducted in the laboratory.

Absolute measurement methods

The gravimetric methods provide the best way of obtaining an accurate quantification of the water content. The measurements are always conducted in a laboratory and are destructive. Such methods include the measurement and calculation of:

- Moisture ratio/mass of water per mass of material (u) kg/kg
- Moisture content/mass of water per volume of material (w) kg/m³
- Degree of capillary moisture saturation (S_{cap})

The choice of measurement depends on the properties of the material/materials being investigated. The first two methods are performed gravimetrically through weighing the total water content in the material. It is important that the sample is enclosed in a water sealed container so that water cannot evaporate. The samples are weighed before and after drying. The drying of the sample is normally done in an

oven at +105 °C (hydrated materials at +40 °C). The mass moisture ratio (u) and the moisture content (w) are calculated according to:

$$u = m_w / m_{dry} \times 100 \text{ [weight \%]} \quad \begin{array}{l} m_w = \text{the weight of the water [kg]} \\ m_{dry} = \text{the weight of the dry material [kg]} \end{array}$$

$$w = m_w / V \quad \text{[kg/m}^3\text{]} \quad \begin{array}{l} m_w = \text{the weight of the water [kg]} \\ V = \text{the volume of the material [m}^3\text{]} \end{array}$$

$$w = \rho \cdot u \quad \text{[kg/m}^3\text{]} \quad \rho = \text{the dry density [kg/m}^3\text{]}$$

The measurement of the degree of capillary moisture saturation is also conducted in the laboratory. This calculation makes it possible to demonstrate how much water is contained in the material in comparison to the capillary saturation point of that specific material. This calculation can only be made on homogenous materials (the saturation point in an inhomogeneous material cannot be calculated). The degree of capillary moisture saturation is calculated according to [134]:

$$S_{cap} = \frac{u}{u_{cap}} = \frac{m - m_{dry}}{m_{cap} - m_{dry}}$$

u = moisture ratio

u_{cap} = capillary moisture saturation proportion

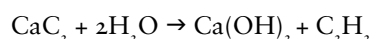
m = weight of the wet material

m_{dry} = dry weight of the material

m_{cap} = weight of the material at the capillary saturation point [kg]

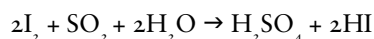
The gravimetric methods have several drawbacks (apart from being destructive). For example, it is common to use drilling to extract the core samples. The drilling adds heat to the material and can provoke evaporation of the water. The method is also time-consuming and has to be conducted in the laboratory. The accuracy of the gravimetric method is usually good ± 0.5 %, and it is thus the most accurate method available.

Chemical Methods – it is also possible to use chemical methods to calculate the water content in a material. These methods depend on chemical reactions with water, for example, the *Calcium Carbide Method* (CM Method). The measurement is destructive, indirect and possible to use in the field. Portable equipment is available. Small samples (usually 10 – 50 g) are enclosed in a container with a certain amount of calcium carbide. The calcium carbide reacts with the water which results in acetylene gas and calcium hydroxide. The amount of acetylene gas is analyzed and the water content can be calculated. The chemical reaction is:



In some of the testing sets the pressure of the acetylene gas is measured. The accuracy of the method is less than that of the gravimetric method: ± 3 %. The method is nevertheless often used since it can be performed on site and portable equipment is available. [135]

Furthermore, another chemical method, the *Karl-Fisher method*, utilizes iodine and sulphur dioxide to chemically bind the water:



This method is very seldom used, however. [135]

The *hygrometrical methods* are indirect, destructive and can either be used *in-situ* or in the laboratory. The methods are based on the fact that when a damp material and a specific amount of air are enclosed in a container, this system will, after some time, reach equilibrium in RH. By measuring the RH in the air and/or the dew point and temperature of the system, it is possible to measure the moisture content of the material. The measurements are carried out in a drilled hole that is sealed, in a cup directly placed on the material (also sealed) or on a sample inserted into a sealed tube. As the RH depends on the temperature, measuring the temperature is therefore essential to the test.

Electrical methods measure the conductivity of the material and are indirect, non destructive and can be used *in-situ*. The electrical methods depend on the difference of the electrical properties of the material being measured and the properties of water. Several cheap portable instruments are available. Electrical methods can be based on two principles:

- 1 the resistivity
- 2 the capacity

The resistivity methods are carried out by measuring the resistance between two electrodes tapped into the material. The measurement is based on the principle that the electrical resistance decreases when moisture is present in a material. The measurement is compared with a calibrated curve for the specific material. Together with the measurement of the temperature it is possible to measure the moisture content by mass (u).

The capacity method, on the other hand, uses a condenser with two electrodes that are isolated from each other by a *dielectric material*. Several moisture measurement methods based on the determination of the *dielectric constant* are available. The constant is much higher for water (ca 80) than for most of the building materials (usually between 3 and 6). This makes it suitable for moisture measurements. A dielectric material, on the other hand, is defined as a material that has little or no electrical conductivity. When the measurement is carried out, the measured material becomes the dielectric material in the condenser that creates the electrical circuit. The instrument measures the change of frequency when the dielectric properties in the material change. The result is plotted onto a calibrated curve and the moisture content calculated. The portable instruments that are available usually measure frequencies around 100 MHz.

The available electrical instruments have several defaults in that they do not penetrate more than a few millimetres

and the conductivity and resistivity are increased when salt is present in the material. Hence a wall or stone containing salt will give the impression of a too high water content. [134]

Infrared (IR) thermography is an indirect and non destructive method often used to detect and analyze moisture in buildings. The IR technique utilizes and measures long-wave infrared radiation. The IR thermography measures the temperature of the surface. From this measurement the presence of moisture can be estimated, since the reflectance of a specific wavelength decreases with dampness. [136] A thermographic system usually consists of an infrared camera (a scanner), electrical direction devices and a monitor. The infrared technique is both passive and active – the passive method measures the temperature on the surface and the active method radiates the surface with long-wave infrared radiation. [139] The Deutscher Dom in Berlin was examined with an active method, *the infrared reflectance*. In this case the technique radiated thermal radiation wavelengths towards the object. The energy was emitted, recorded and interpreted into visible images that demonstrated the distribution of the differences in temperature. The *IR thermographic camera* measures selected spots on the surface using an infrared detector. The specific temperature is measured for every spot and the result is plotted into a thermogramme in black and white or in false colours. The method has been used for a long time in conservation, for example by Mamillan in France in the end of the 1960s, [137] in the Kapelle Saint Silvester in Goldbach in Germany and in the chapel housing Piero della Francesca's paintings in Arezzo in Italy. [138] It is sometimes difficult to interpret the thermogrammes since temperature differences are not only caused by water. The method must thus be combined with other methods. Although the technique requires experienced personnel and is quite expensive, it is used quite often.

Microwave (radar) moisture measurements have been used for at least 25 years. The techniques depend on the measurement of the dielectric constant (see above). In an interesting project in Finland and Germany, the method was tested in the laboratory as well as in the field (the result was published in 1998). [140] Microwave measurements are indirect and usually locally destructive. There are also non destructive techniques available. In most of the techniques antennas are inserted into drilled holes in the material (at a depth of 20 to 30 cm). The method is similar to a microwave oven. It determines the attenuation of a freely propagating electromagnetic wave over a certain distance in a moist material. The water molecules absorb the energy from the surrounding electromagnetic field and transform it into thermal energy. The resonance frequency of the process is 22 GHz (this high frequency is used by some researchers even though enough energy is absorbed between 6 to 12 GHz, although within this range salts may disturb the measurements – see below). One drawback is that surface roughness may produce uncertainties. [140]

A portable microwave instrument (MOIST-Verfahren) has been developed in Germany by Göller. It measures the moisture on the surface of the material and is thus non-destructive. The instrument uses frequencies close to 2.45 MHz and can penetrate to around 30 cm in depth. In addition, the temperature and the absorption and reflection coefficient are measured. The collected data can be used to create moisture maps. Salts can disturb the measurement, although according to Göller, such disturbance is not substantial. [141, 142] Furthermore, Leschnik has proved that the microwave frequencies around 200 MHz are not influenced by salts. [143, 144] The method has been successfully tested over time at the Deutsche Dom, in the Rauthaus and in the Ratsbierkeller in Lübeck and at the Dresden Zwingers. [136, 145] Göller finds the method to be quick, easy to use *in-situ* and enormously time-saving. [141] According to the tests made by BAM (Bundesamt für Materialforschung und – Prüfung) and VTT (Technical Research Centre of Finland) the accuracy of the method is good: between 0 and 2 percent in uncertainties. [140]

Portable Nuclear Magnetic Resonance (NMR) [146] is another instrument that can be used to measure moisture content. It is non destructive and indirect. However, it must be used in combination with an investigation of the salt situation since salts disturb the measurement. A portable device has recently been used in Italy by Poli and Toniolo. [147] They tested the limitations of the method and found that the instrument showed a good sensitivity to the presence of water between 1 percent to 20 percent humidity. They also proved that the NMR signal is proportional to the number of water molecules contained in the material. The technique requires experienced personnel, though, and is thus expensive.

In a report on the measurement of water content in historic masonry, the Danish conservation scientist Poul Klenz Larsen has tested the *electrical measurement*, the *neutron moisture meter* and wooden studs and gypsum blocks with electrodes. According to Larsen the electrical measurement is not good enough, since it only measures the surface and the measurement is distorted by salts. He thus tested the *neutron moisture meter* method instead, sometimes referred to as the *neutron probe method*, which is not disturbed by salts.

The *neutron moisture meter* is an indirect non destructive method. It has been used for a long time in soil science. It is based on radioactive isotopes that send a weak neutron radiation into the damp material. The neutrons hit water molecules that are "moving faster" since they contain H atoms in comparison to the neutrons that hit the porous material that are "slowed down." The difference between the slow and fast neutrons are measured and calculated in order to establish the moisture content of the material. The instrument can measure up to 25 cm in depth and is therefore a

good instrument to use as the first examination. The instrument cannot measure the moisture profile of the whole wall and might create problems with other materials that contain water. It is moreover quite heavy. The method has also been used in the Rathausbierkeller in the Lübeck Rathaus. [145] An ASTM standard exists for this purpose, namely, the *ASTM Standard D-3017-78 Standard test Method for Moisture Content of Soil and Soil-Aggregates in Place by Nuclear Methods (Shallow Depth)*. [148]

An interesting article that describes several moisture measurement methods has been utilized in the Lübecker Ratsbierkeller in Germany. Visser and Gervais tested a *microwave method*, a *gravimetric method*, a *radar method* and a *neutron probe method* (in this case, meaning the neutron moisture meter that measures the amount of H atoms bound in the water molecule). They found one serious drawback of the neutron method: that water is often bound into other building materials and substances found in the masonry, such as gypsum mortars, salts (water crystallization), cement, organic materials and water in argillaceous materials. The authors therefore recommend that this method should be combined with gravimetric methods (that are destructive) to eliminate any possible problems. [145]

There exists another *Neutron Probe method* that has been used in the Smokehouse in Colonial Williamsburg. This method however differs from the neutron moisture meters but is not commonly used. It is detecting the characteristic gamma rays of specific energies that are typical of the target elements. It can thus measure the moisture content directly. [148]

The wood stud/ gypsum block method. Larsen also tested two other methods that measure the moisture content in a material. The wood stud method originally derives from the wood industry and utilizes small beech tree studs that are attached to electrodes. The electrodes are inserted into holes in the masonry. The holes are sealed so that the system can reach equilibrium (e.g. the moisture content in the stud will adjust to the moisture content in the material). As soon as equilibrium is reached, the electrical resistance between the electrodes is measured. The result will give a measurement of the moisture content in the system. There is a key problem with the method however, in that it is developed for wood rather than masonry. Hence the studs are only able to measure the RH between 50 and 95 percent. The microwave measurement can be disturbed by the water content in lime mortar, although on the other hand the weight percentage of the wooden stud is only ca. 1 percent in the same conditions. To avoid this problem Larsen utilized gypsum blocks instead (a method that is used in soil science). The gypsum blocks are made of a similar material to masonry, which makes it possible to measure the actual moisture content more accurately. [149]

Table 7. Methods for Measuring the Moisture in Building Materials

NDT Method / Instrument type	Measuring area	Instrument's weight, accuracy, range, speed, resolution and so forth	Possible to use outdoors	Needs personnel trained in science	Use in conservation	Expensive/inexpensive
Gravimetric methods	Destructive. Small samples are required; the drilled cores are usually very small.	Accurate but time consuming. The drilling process can make the materials become too hot and provoke evaporation of the water.	NO, the samples are measured in the laboratory.	-	YES	Inexpensive
Chemical methods	Destructive, small samples are required.	Fast and easy to use in the field. The accuracy is +/- 3 %	YES	YES	Do not know	——
Electrical methods	Depends on the instrument, however only on the surface.	Light-weight, the accuracy depends on the presence of salts.	YES	NO	YES	Cheap
Infrared (IR) thermography	Depends on the instrument, whole buildings can be weighed and analyzed.	There are portable instruments. The method is fast and quite accurate; the resolution depends on the instrument.	YES	YES and NO Interpretation may be difficult	YES	Expensive
Portable Nuclear Magnetic Resonance (NMR)	——	——	YES	YES	YES, but rare	Expensive
Portable Radar and Microwave moisture measurements	Depends on the instrument. Often the senders are drilled into the material. The portable method measures 30 cm into the material.	There are small portable instruments. Fast. The accuracy depends on the signal and whether salts are present in the material.	YES	YES	YES	Expensive
Portable Neutron moisture meter (Neutron Probe meters)	Depends on the instrument, usually 30 cm into the material.	There are portable instruments.	YES	YES	YES	Expensive
Neutron Probe method	——	The method was used in Colonial Williamsburg. It is often mixed up with the neutron moisture meter. (see above)	YES	YES	Not common	Expensive
The wood stud/ gypsum block method	——	Micro destructive. The measurements are not exact and only estimate the water content.	YES	YES	No	Inexpensive

4.8 Methods for Measuring the Colour of a Material

Colorimetry is the science that studies and measures colour. It is often used in conservation. Ruth Johnston-Feller has written the most comprehensive book on the subject for the conservation field. [150] *Colour measurement* is used in stone conservation to control the effect of cleaning and to monitor when re-soiling of the stone occurs after conservation. Moreover, it is used to identify pigments. The methods are not only important for monitoring colour changes for aesthetic reasons, but also for understanding weathering, since alterations in colour might indicate chemical changes. Such changes might include chemical processes of dissolution, an alteration of minerals and the formation of ageing patinas in stone materials. Colour measurement methods are non destructive and can be used *in-situ*.

Colorimetric studies in conservation have been used for many years. For example colorimetry was used to study Duccio's Maestà at the ICR (Istituto Centrale del Restauro) in Rome in 1953. [151] The Italians have devoted a lot of

research to this area and as a result an interesting Italian publication (including articles from two congresses) is available, *Colormetria e Beni Culturali* (2000). During the last decennium there has been an increase in *in-situ* colour investigations and nowadays colorimetry has become standard in many studies on stone deterioration. One illustrative example is the *Colorimetric cataloguing of stone materials (biocarcernite) and evaluation of the chromatic effects of different restoring agents*. [152]

It is well known that the experience of colour is subjective. We perceive colour as a response to a nervous pulse reaching the brain, due to the joint action of the spectral composition of light emitted by the luminous source and the level of spectral sensitivity in the eye of the observer. The human eye can detect wavelengths between 380 and 780 nm. [153] Special *colorimeters* and *spectrophotometers* have been developed that measure the reflected or transmitted visible light of an object. Nowadays these are available as portable instruments. The *Colorimeter* measures light emission using receptors of red, green and blue. The result is isolated numerical values in the different colour notations. The

Spectrophotometer, on the other hand, measures the light in the 380–780 nm wavelength area. The colour is plotted onto a curve obtained by joining the values measured at each wavelength. [153] A *Spectrophotometer* has been tested in this project.

The only way to be precise when describing colour is to detect the reflected wavelengths (in the visible area) of an object illuminated with a predestined light. For this purpose sets of standard illuminants have been chosen by the Commission de l'Eclairage (CIE, established in 1931). In colorimetry, the wavelengths intensities of a number of standard illuminants are selected by the CIE and the wavelength sensitivities of two observers measured. One of these standard observers represents distance viewing, while the other represents arm's length viewing. By measuring the reflectance of an object that is followed by calculations incorporating the data for one of CIE standard illuminants and a standard observer, three values can be obtained that can be used to describe colour. These were introduced by the CIE in 1931 and are accepted worldwide. [150] They include the three dimensional colour descriptions, XYZ tristimulus value or the Yxy colour space. The XYZ tristimulus values are based on the three-component theory of colour vision, which states that each eye has colour receptors for all three primary colours (red, green, blue) and that all colours are a mixture of these. [150] Problems were found with the Yxy colour space, however, in that it was useful to describe colour but difficult to visualize. [154] In 1976, the CIE developed a new 3D colour space system, based on CIELAB notations ($L^*a^*b^*$) values. The CIELAB colour space thus reduces some of the earlier problems. It utilizes the principle of opposing colours in a sphere where the centre of the space is achromatic. The lightness is the L^* , and the hue and chroma are expressed by a^* and b^* values. Positive a^* values refers to red hues and negative values to green hues. Similarly, yellow hues have positive b^* values and blue have negative b^* values and chroma C^* is the CIELAB colour space. The data is either expressed graphically or in tabular form. [152]

The spectrophotometers available on the market are usually constructed to calculate colour differences for industrial uses and are often based on the CIELAB values. With the instrument it is possible to store data from one measurement and compare the result with later measurements. Changes in chroma are expressed in ΔC^* and in lightness as ΔL^* . The colour difference are, on the other hand, $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{(1/2)}$. [155] Some of the method's drawbacks are that it can be difficult to return to measure the exact same spot, and that moisture and temperature changes might influence the result. These factors have been tested in this study (see above).

The *Munsell* or *NCS systems* are also often used to determine colour. The systems consist of colour chips or charts that are compared with the colour as apprehended by the eye. These methods are not completely trustworthy since they depend on the human eye. These are based on three important concepts:

- Hue – the colour that is registered by the eye through the wavelength that reaches the eye.
- Value – indicates the degree of lightness of the colours.
- Chroma – the degree of dilution of any colour by a neutral grey (also called saturation).

Many of the colour systems are based on these concepts and are, for example, combined in the charts in the American Munsell system (introduced in 1905) based on reference chips categorized by the *Hue Value/Chroma* H V/C. [152] Each colour chip's values are described by alphanumeric labels, with five principal hues of red (R) yellow (Y), green (G), blue (B) and purple (P), and five intermediate hues of yellow-red (YR), green-yellow (GY), blue-green (BG) and purple-blue (PB). These are divided into four segments, with the same visual step determining the depth of the colour. The numbers are 0, 2.5, 5 and 10 (a deep red colour is 5R). [153] The Munsell chart requires the same illumination, and that it is done by the same person with a good colour perception. The method is therefore very subjective.

Table 8. *Methods for Measuring the Colour of a Material*

NDT Method / Instrument type	Measuring area	Instrument's weight, accuracy, range, speed and resolution	Possible to use outdoors	Needs personnel trained in science	Use in conservation	Expensive/inexpensive
Portable Spectrophotometer	Small, depends on the instrument.	Easy to handle, lightweight, fast and accurate.	YES	NO	YES	Moderately expensive
The Munsell or NCS systems	————	Lightweight and fast, accuracy is not so good – it depends on the observer.	YES	NO	YES	Inexpensive

Notes

1. According to ECCO and ICOM-CC conservator-restorer is the chosen term for the conservation professional. Even though it is a "heavy" term, it is internationally accepted and therefore used in this report. The activity of the Conservator-Restorer (conservation) consists of the technical examination, preservation and conservation-restoration of cultural properties: Examination is the preliminary procedure taken to determine the documentary significance of an artefact's original structure and materials; the extent of its deterioration, alteration, and loss; and the documentation of these findings. Preservation is action taken to retard or prevent deterioration of or damage to cultural properties by control of their environment and/or treatment of their structure in order to maintain them as nearly as possible in an unchanging state. Restoration is action taken to make a deteriorated or damaged artefact understandable, with minimal sacrifice of aesthetic and historic integrity. See ICOM-CC: <http://icom-cc.icom.museum/About/DefinitionOfProfession/>
2. The chosen sites in Stockholm are (in age order): The German Church (15th century, although the stone is from the 19th century), The Gustavian Memorial Chapel at Riddarholm's Church (16th century), a portal at Bollhusgränd 3A (middle of the 17th century), a portal at Lilla Nygatan 2 (end of the 17th century), a portal at Svartmansgatan 6 (middle of the 17th century), The Karolin Memorial Chapel at Riddarholm's Church (18th century – the stones are from different periods), a portal at Slottsbacken (18th century), a portal at Stortorget 3 (18th century), a portal at Strandvägen 46 (1893), the Bernadotte Chapel at Riddarholm's Church (the beginning of the 19th century), stones in the socle at Skeppsbron/Brunnsgränd (1901), a portal at Narvavägen 30 (1903), a portal at Strandvägen 7 C (1907), a portal at Skeppargatan 61 (1914), a portal at Engelbrektsgatan (1912–1914) and stones in the lower part of Östermalmsgatan/Skeppargatan (1924–1925).
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54. http://www.eu-artech.org/index.php?option=com_content&task=view&id=24&Itemid=71 and <http://minelab.mred.tuc.gr/dias/> The DRMS is a new portable system developed and validated for directly determining stone mechanical features such as the "hardness" by measuring its drilling resistance validated within the European project HARDROCK: Development of a new measurement method to determine the superficial hardness of exposed monumental rocks

- (SM&T, CT96–2065)]. The force is measured with a monaxial load cell (estensimetric transducer) which transforms the gauge deformation into an electrical signal (tension). The load cell deformation is correlated with the "resistance to penetration" hence to the "stone cohesion". The measuring unit of the Drilling Resistance (DR) is Kgf (Newton) with sensitivity of $2.0115E-0.3$ (V/V) corresponding to 1 N and has 100 N as maximum load. The DRMS has no competitors for its application in comparative tests "in situ" and it has been suggested as a standard method to UNI Normal (Italy) for assessing the quality assurance of consolidating treatments.
55. <http://www.icvbc.cnr.it/mcdur/Europe> is quickly losing its leading position in the natural stone market due to increasingly aggressive competition from new, non-European manufacturers. Based on current market data, it is therefore reasonable to assume that European natural stone districts will experience an ever worsening decline, with foreseeable social and economic consequences. It is therefore imperative that Europe takes steps to improve its competitive edge by focusing on the aspect of stone material qualification to improve European performance in this market, through an improved capability to qualify the characteristics of its stone products in the short-, medium- and long-term. To achieve this objective we need affordable measures and methodologies to draft standard methods for evaluating the "Stone Durability" of the natural stone market as stressed by the technical Committee CEN TC 246, because of the differences between test procedures in use in the different European countries and the lack of correlation of the results of accelerated decay tests with the natural decay in the different climatic areas. The main project deliverables will be the building of an "Abacus for Stone Durability" based on the behaviour on ageing (natural and accelerated) of stone mechanical properties of selected "rock categories" for pre-normative use, and the validation of the portable NDT integration system for evaluating the "quality" of a stone "in-situ". Side deliverables will be a draft of the sampling strategy and philosophy on the sensitive topic of studying the effects of weathering agents on stone quality, and Sequential Image Analysis software for the dynamic analysis of stone microstructures.
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 63. This is Mathias Kocher's answer: The success of using a roughness measurement systems in the field of stone conservation/restoration depends on the question what will you measure with the system. For example, if you look onto a highly polished marble surface during time you could see a slowly increase of the surface roughness. This means the surface is increasing and the higher the surface the faster is the degradation. On the other side, the roughness of a sandstone is primarily caused by the grain size of its minerals. If this sandstone degrades during time it will lose grain by grain and grain layer by grain layer. The surface roughness did not increase noticeable. Please do in advance a comparative roughness measurement on fresh sandstone and on the same stone, exposed on a monument since a few hundred years. Only if you could see any difference this method could help you.
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112. The equipment allows the recording of colour and IR reflected images free of optical distortion and with the best resolution presently available in the field. The system simultaneously collects images corresponding to four different regions of the electromagnetic spectrum, infrared, red, green and blue, which can be perfectly superimposed. From the collected data it is possible compare directly, on the monitor of a PC, the visible image of the object under study and its IR reflectogram. This allows users to study the execution technique of a painter and, as in a recent study of Perugino's technique, clearly identify hand-drawings, pentimenti, and cartoon re-use. In addition, it is also possible to identify regions of undocumented restorations or the presence of some pigments. For this purpose, the imaging survey can be usefully complemented with punctual spectral analyses carried out by fiber optic vis-NIR spectroscopy. http://www.eu-artech.org/index.php?option=com_content&task=view&id=24&Itemid=71
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Appendices

Appendix 1

Scientists Consulted for the Literature Study

People consulted include:

- Dr. Rolf Snethlage and Dr. Mathias Kocher, Conservation Scientists at Centrallabor at Bayerisches Landesamt für Denkmalpflege, München, Germany
- Dr. Stefan Brüggerhoff, Conservation Scientist at Preservation of Cultural Heritage / Material Science, Deutsches Bergbau-Museum, Bochum, Germany
- Dr. Stefan Simon, Conservation Scientist at Rathgen-Forschungslabor at Staatliche museum zu Berlin, Germany (he has recently worked at Getty's Conservation Institute, Los Angeles, USA)
- Dr. George Wheeler, Professor, Stone Conservator and Conservation Scientist at Columbia University, New York, USA
- Dr. Marisa Laurenzi Tabasso, Private consultant and Conservation scientist specialized in Stone Conservation, Rome, Italy
- Dr. Piero Tiano and Dr. Susanna Bracci, both Conservation Scientists at ICVBC-CNR, Florence, Italy
- Dr. Susan Bradley, Conservation Scientist at the British Museum, London, Great Britain
- Dr. Jadwiga Lukaszewicz, Professor and Conservation Scientist at Nicolaus Copernicus University, Torun, Poland
- Dr. Daniel Kwiatowski, Conservation Scientist and Stone conservator and Dr. Malin Myrin at Stenkonservern Skanska, Sweden
- Stone conservator Jarema Bielawski, JWB ARK & ART, Sweden
- Stone conservator Svante Nilsson, Prolithos stone conservation, Sweden
- PhD candidate Jenny Hällström, Lund Institute of Technology, Lund University, Department of Architectural Conservation & Restoration.

Appendix 2

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