Investigation of the conservation-treatment methods of the Dutch national monument: the role of microscopy

Joe A. Larbi and Rob P.J. van Hees
TNO Building and Construction Research, Division of Building Technology
P.O. Box 49, NL-2600 AA Delft, The Netherlands

The Dutch National Monument in Amsterdam, a World War-II memorial structure, was built with an outer face consisting of slabs of travertine. In 1995, the masonry structure forming the core of the monument showed severe deterioration. In order to determine the actual cause of deterioration and to advise on the most suitable method of restoration, an extensive investigation was carried out on the monument itself and on samples removed from it. The action of frost was found to be the primary cause of the deterioration to the brick structure. The travertine slabs and sculpture showed decay in the form of cracks and of smoothening of the sculptural details (superficial loss of material).

For purposes of restoration, the monument was completely dismantled and the inner masonry structure was replaced with prefabricated concrete. The sculptures (statues and reliefs) were treated with a special acrylic resin (polymethyl metha-acrylate, PMMA) in order to conserve them.

Polarising and Fluorescent Microscopy (PFM) was used in all phases of the investigation. The technique was found to be an excellent tool for such investigations. This article deals with various aspects of the investigation where PFM played a key role, such as the diagnosis of the cause of the surface deterioration of the travertine and the evaluation of the effectiveness of impregnation treatment methods of the sculptures.

Key words: **microscopy, travertine, conservation treatment**

1 Introduction

The National Monument, located at the heart of Amsterdam in The Netherlands was built in 1956 as a memorial structure of the Second World War (see Figure 1). It consists of vertical slabs and various sculptures of travertine. The inner (load-bearing) structure was built with concrete (up to 8 m) and brick masonry (higher part). The travertine, which was used for the sculptures and as a facing stone for the 20-meter high pylon was quarried from a large deposit in Tuscany in Italy [1].
The sculptures and slabs of the pylon were suffering from severe physical weathering which was manifested externally in the form of cracks of different sizes (see Figures 2-4). Some of the cracks were so wide that they could easily be seen with the naked eye from far off. Details of the sculptures had been smoothened as a result of material loss, resulting in of the expression of the sculptures.

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In December 1995, part of one of the travertine slabs fell down. Immediately following this event, a committee was set up by the Amsterdam Municipality Council and charged with the task of ascertaining the gravity of the deterioration and to give recommendations for restoration. A working group of experts within the committee was commissioned to investigate the extent and the causes of the deterioration of the monument and to propose suitable measures for its restoration. The initial accident turned out to be just an incident but more severe decay was found in the internal structure of the monument. Finally, the committee responsible for the restoration of the monument drew an extensive programme for the restoration of the monument.

Within the framework of the project, TNO Building and Construction was charged with the task of carrying out detailed site inspection of the entire structure. As part of the investigation various samples were removed from both the inner masonry structure and from the cover stone and the sculptures for further studies and testing in the laboratory.
This article deals only with part of the investigation that was performed in the laboratory by means of polarising and fluorescent microscopy.
The objectives of the PFM investigation were:

- to assist in determining the cause of the deterioration of the stone;
- to evaluate the effectiveness of two methods of impregnation (proposed to conserve the stone and to prevent further loss of sculptural details);
- to evaluate the effectiveness of the selected conservation treatment method for the sculptures (consisting of statues and reliefs).

Fig. 1. A view of the Dutch National monument in Amsterdam prior its restoration.

2 Method of microscopical examination

2.1 *Introduction*

The microscopical examination technique used in this investigation is based on a TNO standard procedure of thin section examination, which is similar to ASTM Standard C856-95 (1995): "*Standard Practice for Petrographic Examination of Hardened Concrete*" [2]. The examination was performed by means of polarising and fluorescent microscopy (PFM) on thin sections prepared from blocks and core samples removed from both the sculptures and the slabs of the monument.

2.2 *Principle of PFM*

PFM involves examination and characterisation of fluorescent thin sections by means of polarising and fluorescent microscopy. The thin sections are usually prepared by sawing a specimen (usually a piece of block) from the samples in question, gluing to an object glass, vacuum-drying, followed by vacuum-impregnation at about 40 °C with an epoxy resin containing a fluorescent dye. After hardening the required thin sections are prepared from each of the blocks for examination by means of PFM.

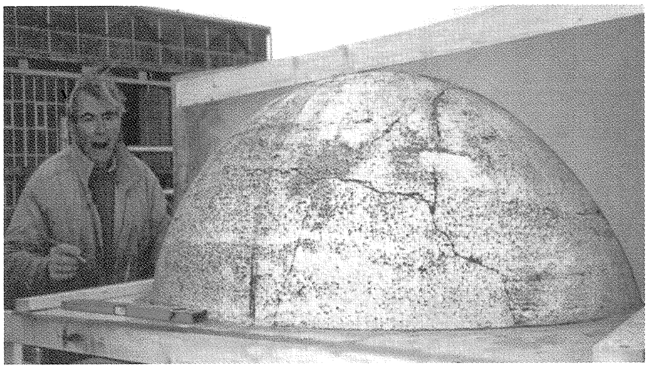
The purpose of impregnating the small blocks or specimens with a fluorescent resin is to make it possible to study the resulting thin sections by means of both transmitted (polarised) light microscopy and fluorescent microscopy. By means of transmitted light microscopy, the mineralogy, changes in the composition and micro-characteristics of the stones can be studied. These aspects are used to determine the type and the extent of deterioration of the stone. If the stones have been treated by impregnation, the penetration depth (for example, PMMA) can also be determined.
The purpose of fluorescent microscopy is to enable the microstructural aspects, such as the compactness and micro-porosity of the stones, as well as the amount and pattern of microcracks, voids and other defects in the specimens to be studied.
All the information gathered from various portions of the studies is used together to assess the extent of deterioration and to evaluate the effectiveness of the impregation-treatment methods applied.

2.3 *Samples*

Three series of samples, consisting of both treated and untreated specimens removed from the monument were examined. The samples for series I and II were removed from the cover stone of the central pylon. The reason for this was that the cover stone had been exposed from all sides to atmospheric conditions and so it served as a suitable representative material to study the effect of the environment on the sculptures (see Figure 2). The samples from series II were treated with 2 different products. The samples of series III were removed from the sculptures themselves after they had been treated. Information regarding these samples is presented in Table 1.

Table 1. Information regarding the samples examined.

Series	Treatment	Type of material
I	No	samples examined in order to establish the cause of damage; two samples from the cover stone were involved, one from the north-west (NW) and the other other from north-east (NE);
II	yes, one series with PMMA and the other series with **water glass**	small samples from the cover stone treated in the laboratory by either impregnating under vacuum with PMMA or a **water glass;** the intent of this part of the examination was to determine which of the two treatment methods is more effective in sealing-off the voids and cracks in the samples
III	yes with PMMA	samples removed from large sculptures after a real scale treatment by vacuum-injection with PMMA; the purpose of this examination was to assess the effectiveness of the treatment process



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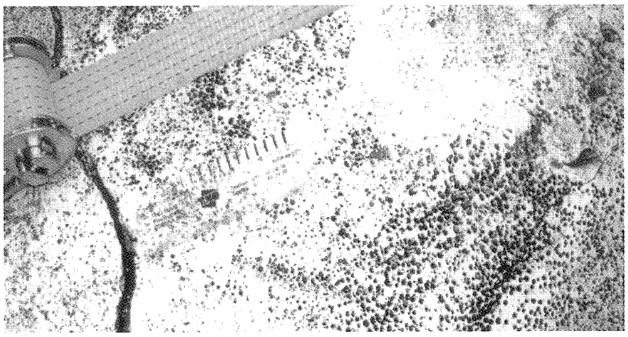
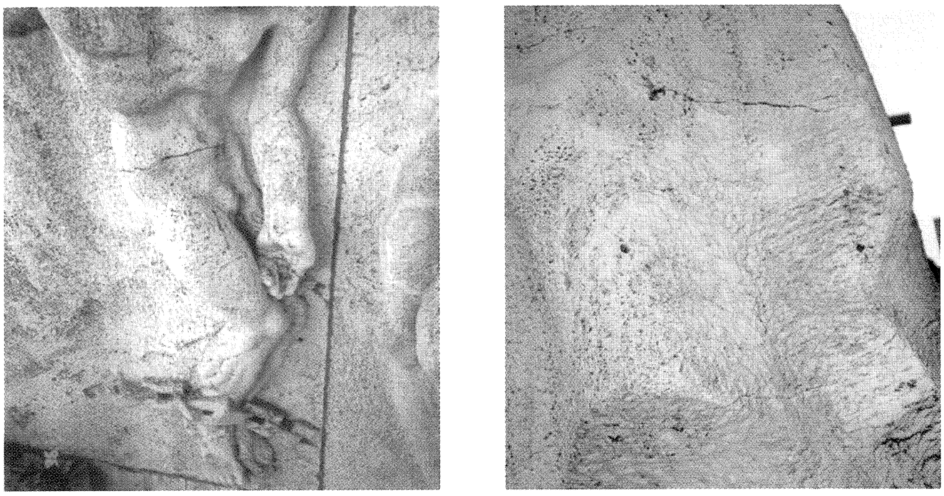


(b)

Fig. 2. Cover stone of the central pylon of the monument showing wide cracks (a) overview; (b) details.



(a) (b)

Fig. 3. Part of the statues at the front side of the pylon showing cracks. (a) overview; (b) details.

3 Investigation

3.1 *Determination of the cause of deterioration*

3.1.1 Objectives

The purpose of this part of the investigations was to obtain insight into the cause, nature and extent of deterioration (weathering) of the travertine. Two samples, removed from the sculptures were used for the studies: one from the north-west portion (NW) and the other from north-east portion (NE) of the cover stone.

3.1.2 Composition of stone

Results of the investigation showed that the travertine stone was composed of fine-grained calcite (micro- to cryptocrystalline calcite - *micrite aggregates*), alternating with fossil concretions, cracks and microfissures with different sizes (see Figure 4). Various "voids" were identified including "visible" pores and capillary pores. The visible pores are those voids that could be seen in the thin sections. In the case of the capillary pores, the sizes are so small (sizes 0.01–1μm), that the individual pores are not distinguishable with a normal optical microscope even with the highest magnification of about 400 times.

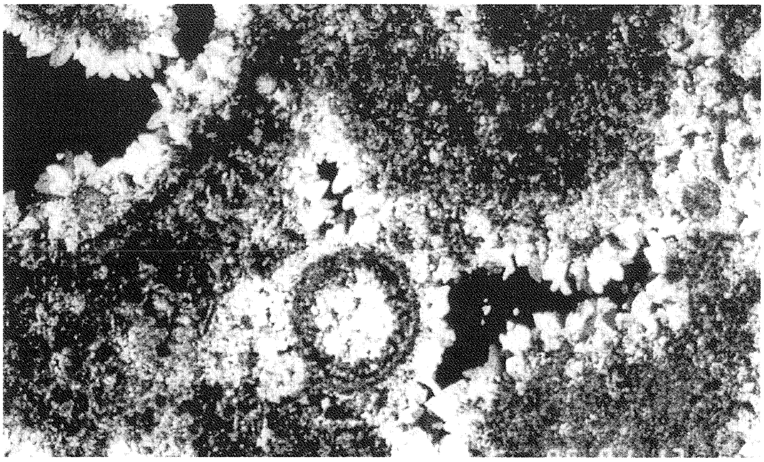


Fig. 4. PFM-micrograph (transmitted light) of an overview of the affected travertine stone showing re-crystallisation of calcium carbonate in large voids.
Size of micrograph is 2.7 mm × 1.8 mm.

The visible voids were found to be intrinsic in origin and "closed" (not connected with each other). The sizes of these voids ranged between 0.1 and 3 mm. At various places within the stone, re-crystallisation of calcite had resulted in the formation of smaller visible voids, ranging in size between 0.1 and 1 mm. The zones with micrite aggregates were found to be 'denser' (lower capillary porosity) with higher concentration of microfissures (≤ 10 μm) than in the other zones. In both samples examined, no difference in material characteristics was observed between the outer and deeper parts of the stone.

3.1.3 Deterioration

Deterioration was mainly physical, caused essentially by frost. This was limited to a depth of 25–30 mm from the exposed surface of the stone. This frost-initiated physical deterioration was manifested by some minor superficial flaking and by the formation of cracks parallel to the exposed surface of the travertine stone (see Figure 5).

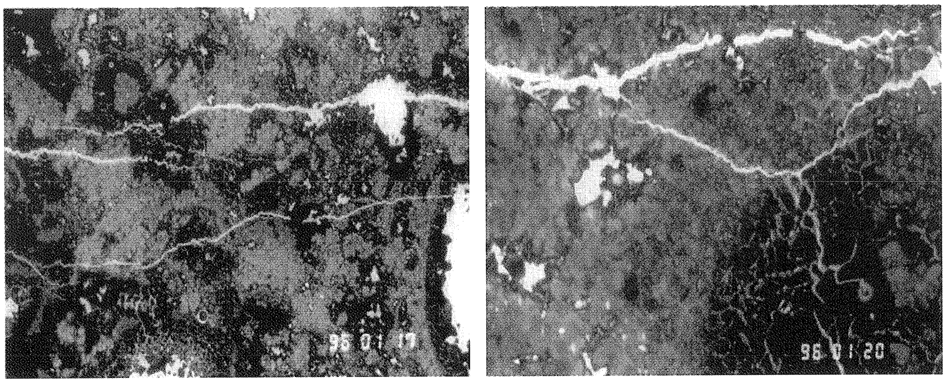


Fig. 5. PFM-micrograph (fluorescent light) of the cover stone showing very fine cracks parallel to the surface of the stone. Size of micrograph is 5.4 mm × 3.5 mm.

Most of the frost-initiated cracks measured in length up to about 2 or 3 centimetres and varied in width from about 10μm up to about 50μm. In addition, there was locally superficial loss of material (estimated to be about 1–2 mm) caused by scaling. The deterioration was considerably influenced by the intrinsic characteristics of the travertine. The presence of zones of dense and highly fissured material, alternating with relatively porous and permeable material enhanced the frost action. Chemical deterioration of the stone, usually caused by either conversion of the carbonate rock by sulphate to gypsum or dissolution by the relatively (acidic) rainwater was negligible. The results of the PFM investigation confirmed the results of the visual inspection and the physical tests (freeze-thaw and water absorption under vacuum), all of which pointed to frost as the primary cause of weathering but not chemical deterioration.

The results of the PFM studies provided a basis for selecting the most suitable method for conservation of the stone, namely by impregnation with a suitable agent in order to seal-off the cracks and voids.

3.2 *Evaluation of the two impregnation methods (pilot study)*

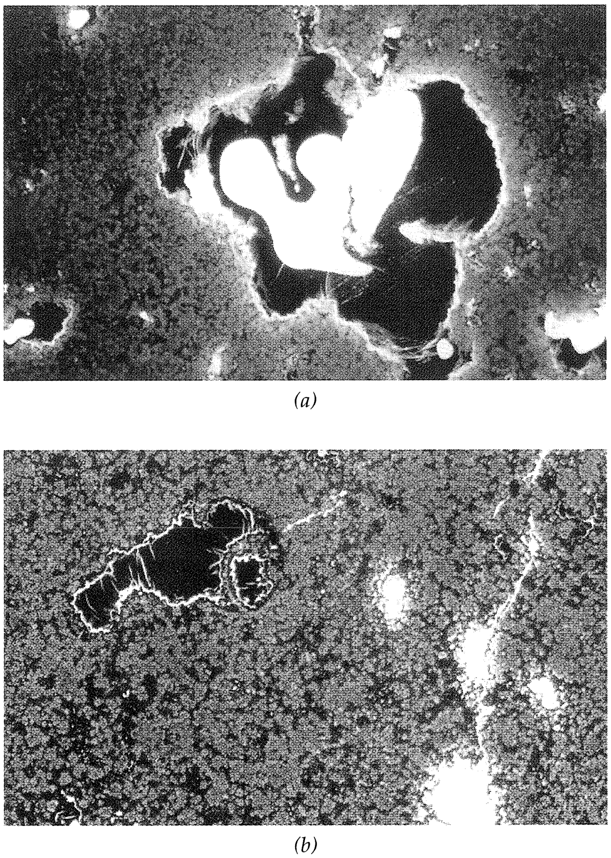
This part of the investigation was performed on small samples treated in the laboratory by separately impregnating them under vacuum with either PMMA or with **water glass**. The intent of the investigation was to determine which of the two treatment methods was more effective in filling or sealing-off the cracks and voids in the samples so that it could be selected for application on the complete sculptures on a real scale. For each method of treatment two samples were used for the PFM investigation. The results are presented in Table 2.

Table 2. Results of the investigation of the effectiveness of the two injection methods.

	Injection method	
Aspects	PMMA	Water glass
Extent of voids filled, % (V/V)	40-50	≤ 40
Nature of filling	Mostly along periphery of Voids, central portion Unfilled (see Figure 6)	Mostly along periphery of voids, central portion, mostly unfilled (see Figure 7)
Shrinkage of injected material	Considerable shrinkage showing de-bonding from crack and pore void walls	Extensive shrinkage especially along the walls of the voids
Filling of cracks and fissures	Partial filling of cracks and fissures	No
Filling distribution over the sample	Good, everywhere the same: 40–50 % filling of voids	Poor, varied from 5 % filling of voids locally and in one thin section, up to 30 or 40 %
Contact of the injected material with the walls of the voids	Moderate	Good

The shortcomings of the PMMA method were discussed with IBACH, the company that carried out the impregnation (the applicator). On the basis of the opinion of experts from IBACH, a too short drying period was thought to be the explanation for the shortcomings. Therefore it was proposed to use a longer drying period for the sculptures.

On the basis of the results of the investigation, in combination with the results of other tests, such as a frost resistance test, the method of impregnation using PMMA was considered to be the better option and as such selected as the conservation treatment method for the sculptures.


(a)

(b)

Fig. 6. *PFM-micrograph of one of the laboratory-treated samples showing voids (visible pores and cracks) filled by PMMA and the associated fine shrinkage cracsk along the walls of the voids. Size of micrograph is 2.7 mm × 1.8 mm.*

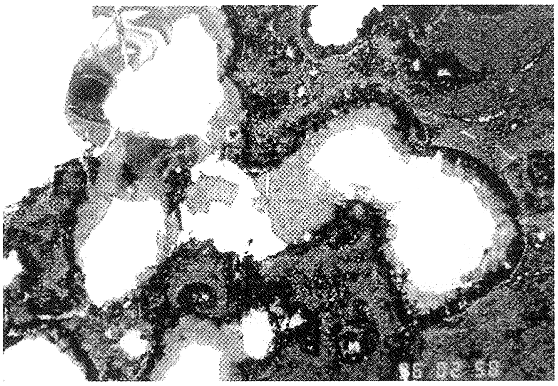


Fig. 7. *PFM-micrograph of one of the samples laboratory-treated samples showing visible voids filled by the water glass associated with shrinkage. Size of micrograph is 5.4 mm × 3.5 mm.*

3.3 *Evaluation of the effectiveness of the treatment of the actual sculptures*

3.3.1 Principle of the treatment method

This method of treatment involved vacuum-impregnation of the whole reliefs and statues.
The impregnation was done in the laboratories of IBACH in Germany. Prior to treatment, the stones were carefully dried and immediately subjected to vacuum-impregnation with methyl meta-acrylate (MMA), which converts to the polymer form, called PMMA. Further information regarding this method of treatment is given in [4].
After the treatment process, samples were removed from three of the treated sculptures and submitted to TNO Building and Construction Research for further studies. Three thin sections were prepared from 20 mm-diameter cores drilled from the sculptures and examined by means of PFM.

3.3.2 Visual examination of impregnated statues and samples taken from them

Visual examination of the samples before the PFM investigation revealed that locally some of the voids were not filled with PMMA. They concerned voids that were connected by large cracks or "open" voids to the exposed surface of the sculptures. Also it was remarkable that large cracks that were visible at the surface of the sculptures themselves were not filled with the PMMA. The inability of the PMMA to fill such cracks was considered a drawback of this method of conservation. As a result of these shortcomings, a special attention was paid to the width of the internal cracks in the samples that were filled with PMMA.

3.3.3 Results of PFM investigation on statues

The following results were obtained from the PFM investigation:
Most of the visible voids were filled with PMMA. In some cases, shrinkage of the acrylic resin was visible in the form of "hollow spaces" within the PMMA-filled voids or as very fine cracks (< 5 μm) locally along the walls of the voids (see Figure 8). The extent of shrinkage was considered not to pose any threat to the conservation of the stone since the extent of filling of larger cracks (widths 0.2 to 0.3 mm) was found to be satisfactory. In the case of the wider cracks, shrinkage cracks along the walls of the voids were not visible (see Figure 9). In general, the results of the treatment of the large reliefs and statues were found to be far better than the results of the pilot study.

3.3.4 Determination of the bulk density and porosity of samples of statues

In addition to the PFM investigation, the bulk density and porosity of samples of the statues were determined by the method of water absorption under vacuum according to the RILEM Recommendation CPC 11.3 [5]. This test was performed to supplement the PFM investigation. The results are presented in Table 4.
Although the number of samples used is small, it can be seen from the results in Table 4 that a considerable difference in porosity exists between the treated and untreated samples. This confirms the results of the PFM investigation that most of the accessible voids and cracks (> 85 %) in the stones had been filled with PMMA.

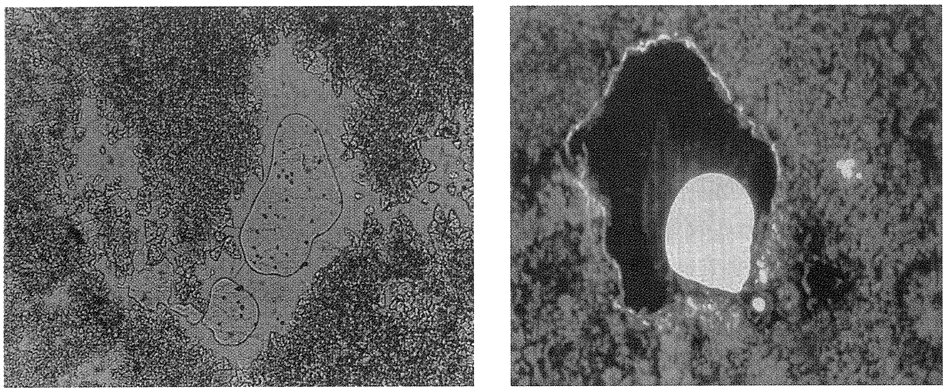
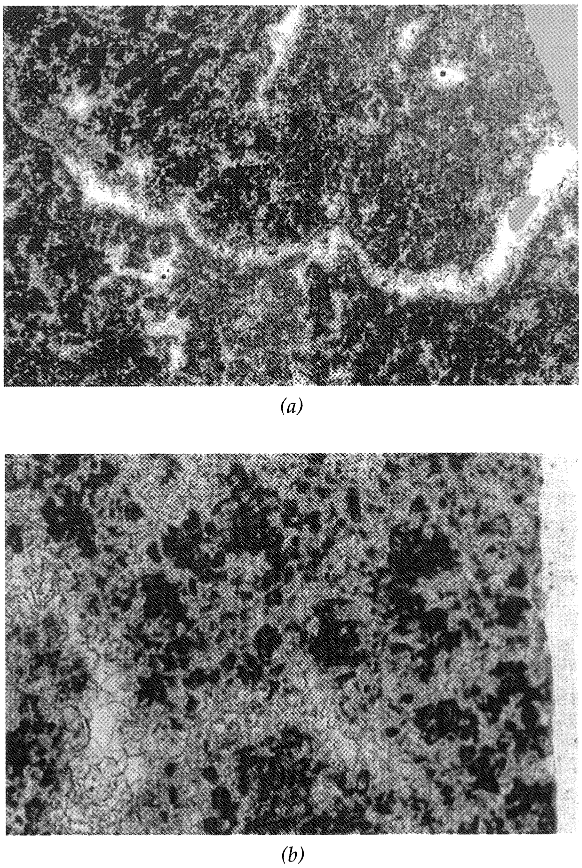


Fig. 8. *PFM-micrograph of one of the samples taken from statues after treatment with PMMA showing voids by PMMA. Size of micrograph is 2.7 mm × 1.8 mm.*



(a)

(b)

Fig. 9. *PFM-micrograph of one of the samples taken from the treated statues showing a long wide cracks filled by by pmma. (a) long, wide cracks; (b) indistinct voids by PMMA.*
Size of micrograph is 5.4 mm × 3.5 mm.

Table 3. Results of the bulk density and porosity test according to RILEM CPC 11.3.

Sample	Bulk density (kg/m^3)	Porosity (% V/V)
Not treated samples		
I Cover stone (pylon)	2470	7.7
II Cover stone (pylon)	2430	8.8
III Pylon	2415	11.7
Average	**2430**	**9.4**
Treated samples		
IV Sculpture	2480	0.9
V Sculpture	2420	1.7
Average	**2450**	**1.3**

4 Summary of conclusions

The PFM method is very suitable for investigating the cause and the extent of damage and for evaluating the effectiveness of the restoration treatment methods. The action of frost on the stones resulting in superficial physical weathering and the extent of the weathering was confirmed by the results of the PFM investigation. The method also made it possible to evaluate the effectiveness of the laboratory restoration treatment methods employed on the samples and the real-scale treatment of the reliefs and statues with PMMA.

As a summary, the PFM investigation revealed that:

- Voids, which were not connected by large cracks to the exposed surface of the sculptures, were very well filled with the acrylic resin. The shrinkage cracks associated with the hardened resin (PMMA), were minor and negligible.
- Cracks with widths from 0.2 to 0.3 mm were very well filled. It can be inferred from the porosity data in Table 4 that frost-initiated cracks, oriented parallel to the exposed surface of the sculptures, which were found in the untreated samples, were also equally filled by vacuum-impregnation.

The porosity of the samples was also considerably reduced after vacuum-impregnation with the acrylic resin. The chance of scaling as a result of frost attack is therefore very much reduced.

The purpose of the treatment, that is, to reduce further loss of details in the carvings of the sculptures, was thus achieved.

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