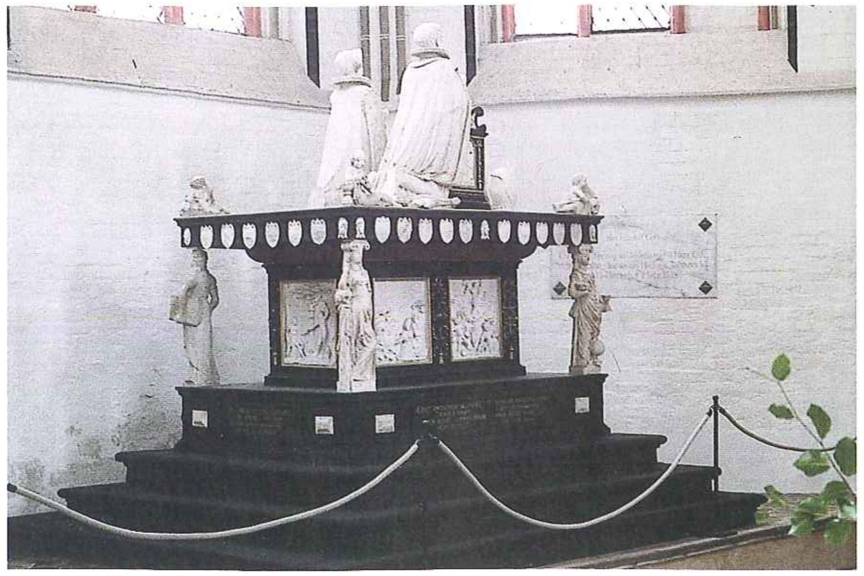
9 

9
Endzustand des Grabmals im September 2007.

Die Kittmassen sind nach aufwendigen Testreihen ausgewählt worden. Abbrüche und Löcher bei den plastischen Altergänzungen aus Gips sind mit Gips wieder ausmodelliert wurden.

Für Rekonstruktion größerer bildplastischer Ergänzungen wurden Silikonformen angefertigt, welche mit der Alabastermasse ausgegossen wurden. Die Herstellung der Gussformen für eine Frauenmaske, die Maskenkordeln und Füße erfolgte durch Abformung an ähnlichen Bereichen des Grabmales. Für die Füße einer Karyatide und einer Genienfigur mussten zunächst in Plasteline Modelle angefertigt werden, auf denen anschließend Gipsteilformen hergestellt wurden, um eine Negativform zu erhalten. Die Gussteile sind abschließend mit Dübeln aus Edelstahldraht und dem Epoxidharzkleber punktuell am Alabasteruntergrund befestigt worden. Den fehlenden Kopfbereich im östlichen Alabasterrelief modellierte der Bildhauer frei als Gipsergänzung (Abb. 7 a und b). Aus dem leicht zu bearbeitenden Zinkblech wurde der fehlende Metallsporn am Fuße der Herzogsfigur angefertigt und angesetzt

Schließen der Fugen

Für die Schließung der Fugen wurde eine Mörtelmischung aus Quarzsand, Flusssand und Sumpfkalk verwendet. Nach der Anmischung der Masse, ist diese so weit wie möglich in die offenen Fugen gepresst worden. Anschließend erfolgte ein Auftrag mit weichem Restauriermörtel, um ein glatte Oberfläche zu erhalten.

Retusche und Rekonstruktion der Farbfassung

Zur farblichen Anpassungen der Ergänzungen des Alabasterschmucks an die originale Oberfläche wurden Retuschen vorgenommen. Die Ergänzungen an den Karyatiden, den Reliefs und den Genien wurden mit Aquarellfarbe retuschiert. Der Auftrag erfolgte in dünnen Lasuren von hell nach dunkel. Die Rekonstruktionen an den Wappen wurden mit Nussbaum- und Teebeizen eingefärbt. Nach der Bemusterung verschiedener Proben auf Ersatzgestein zeigten die beiden Beizen die besten Ergebnisse, um das optische Erscheinungsbild einander anzupassen. Der Auftrag erfolgte mit mehreren dünnen Lasuren.

Die Fehlstellen auf den vergoldeten Profilleisten der Reliefs, der Inschrift und im Bereich der Rahmung des Betpultes erfolgte eine Teil- bzw. Strichretusche mit jeweils abgetöntem Muschelgold sowie Malergold (Abb. 6b). Die Rekonstruktion der ehemals vergoldeten Fruchtgehänge auf der Tumba und dem Betpult, sowie die Jahreszahl 1595, erfolgte ebenfalls mit abgetöntem Malergold. Die Auswahl der Produkte für die Retusche und die Abtönung des Malergoldes orientierte sich am Bestand und erfolgte mit Pulver- bzw. Perlglanzpigmenten. Eine Rekonstruktion der Blattvergoldung wäre im Vergleich zum Befund zu kontrastreich.

Im Sandsteinbereich wurde die Farbfassung vollständig nach Befund mit einer Ei-Öl-Harz-Emmulsion als Bindemittel rekonstruiert. Die verwendeten Pigmente Rebschwarz, heller Ocker, Umbra und Eisenoxidrot wurden vor dem Auftrag einige Tage in Terpentinersatz bzw. in einer Mischung aus Wasser mit Ethanolzusatz eingesumpft. Der Farbauftrag erfolgte zweifach lasierend.

Erlebnis Grabmal

Nach der Restaurierung (Abb.9) ist das ursprüngliche Erscheinungsbild des Grabmals weitgehend wiederhergestellt und damit ein bedeutendes Zeugnis protestantischer Bildhauerkunst des 16. Jahrhunderts aus der sogenannten flämischen Florisschule. Das Bildwerk wirkt wieder in seinem zeittypischen Kontrast von hellen Alabasterreliefs mit partiellen Vergoldungen und schwarz gefassten Steinbereichen. Im Anschluss ist auch der wertvolle Fußboden mit polychrom glasierten Keramikplatten, die durchaus älter sind und vermutlich schon bei der Errichtung des Grabmals in der Kapelle zweitverwendet wurden, aufwendig restauriert worden. Zudem wurde vom Auftraggeber zuvor im Fundamentbereich eine zweite hinterlüftete Wand vor die Kapellenaußenwand gesetzt, um den weiteren Feuchtigkeitseintrag ins Innere zu minimieren.

Anmerkung

1 Neben Restaurator Boris Frohberg waren an diesem Projekt auch Dipl.-Ing. Thomas Bolze, Dipl.-Geologe Christian Krempler, Dipl.-Chemiker Dr. Dietrich Rehbaum, Dipl.-Rest. André Streich sowie MA Claudia Arnold, Dipl.-Restauratorin Wolfram Vormelker, Dipl.-Restauratorin Nora Pietrowski, die Studentin Karina Wilke (im Rahmen einer Facharbeit an der FH-Erfurt) und Dipl.-Rest. Bernhard Schellbach beteiligt.

Abstract

New Splendor for Duke Christoph

Conservation and Restoration of a Renaissance Tomb

The Duke's megalomania did not end with his death as his magnificent tombs demonstrate. They also bear witness to the art of sculpture in his time. Such a tomb was restored in the Cathedral to Schwerin. First the foundation was stabilized, uneveness was corrected, and the stone ensemble was desalinated. Then, the sandstone and alabaster surfaces were cleaned, consolidated and finally restored.

Keywords: sandstone, Cotta-Elb sandstone, alabaster, surface cleaning, consolidation, stone restoration, stone putty, putties

Tine Frøysaker, Costanza Miliani and Mirjam Liu

Non-invasive Evaluation of Cleaning Tests Performed on "Chemistry" (1909–1916)

A Large Unvarnished Oil Painting on Canvas by Edvard Munch

No original varnish has been reported or observed on "Chemistry", one of eleven large Aula paintings by Edvard Munch. The problems specific to the cleaning of unvarnished oil paintings on canvas, especially in the case of Munch and other painters working in the early twentieth century, have received relatively little study in comparison to varnished oil paintings. "Chemistry" provides a compelling example of the problems faced by such works. It has been severely soiled by airborne pollutants and its surface is more difficult to clean and more fragile than comparable varnished oil painting. During two periods in 2008, a total of 88 cleaning tests were performed on "Chemistry", 80 prior to and eight during one week of EU-ARTECH MOLAB Access Service in July. The aim was to propose a method to assess the cleaning system. The non-invasive mid-FTIR spectroscopy measurements allowed the identification and mapping of various types of surface contaminants like sulphates, silicates, metal soaps and zinc oxalates. Visual examinations and infrared measurements were applied to evaluate the effectiveness and harmfulness of the cleaning tests.

Introduction

Soiling and cleaning of Edvard Munch's unvarnished oil paintings on canvas (1909–1916) in the Aula (assembly hall) of Oslo University (UiO) was cyclical and the consequences were severe.[1] The eleven large scale motifs (all told c. 220 m^2) had been cleaned up to six times in the seven decades between their installation in 1916 and the last treatment in 1986.[2] In the course of the same period, the average interval between cleanings was eleven years and never exceeded sixteen.[3] Comparable cycles for similar paintings and treatments at the neighbouring National Museum of Fine Arts, Nasjonalgalleriet are forty-six years.[4]

In 2008, clean colours were found under random splashes of flour paste from the mounting of the Aula canvases on masonite boards in 1946 [Fig. 1] (the boards were held together by a wooden framework on the reverse side).[5] It seems the aim of the first three surface cleanings in 1926, 1937 and 1946 was to remove the airborne dirt. The same may also have been the case of the two later cleaning projects in 1957 and 1973,[6] but the sixth and last cleaning only consisted of limited dirt removal on three paintings ("The Sun", "History" and "Awakening Men in the Flood of Light").[7] This means that the dirt on the surfaces of the other eight had accumulated since 1973 and without intrusion of any known conservation treatment.

The previous cleanings involved the use of various mechanical methods and solvents.[8] Low-suction vacuum cleaners, common school erasers and hand-size lumps of bread dough (rolled, pressed and rubbed over the surfaces) were employed to remove the dirt.[9] The first reported cleaning also involved the use of cotton swabs dipped in distilled water and in a fast evaporating petrolether (boiling point 45–60°C). During the last cleaning of the three paintings in 1986, the conservators used 'Wish ab'; ammonia and water in different concentrations; 'Winton Picture Cleaner' and other solvent mixtures (acetone, alcohol, white spirit and petrol ether in different proportions).[10] A limited number of cleaning tests was performed the last two times dirt was removed and visually inspected in situ.

In January 2008, and prior to the recent cleaning in 2009, "Chemistry" (≈ 444 x 220 cm^2, weight c. 90 kg) was demounted and moved to the studio at Conservation Studies at UiO [Fig. 2].[11] Cleaning tests were carried out to evaluate more than thirty years of exposure to airborne pollution.[12] After a successful application to the EU-ARTECH MOLAB Access Service,[13] it also became possible to explore the effects of cleaning with MOLAB's portable and non-invasive analytical equipment.[14, 15]

1
Detail of "Chemistry" from its lower left side corner after removal of flour paste from 1946. Courtesy of Mirjam Liu 2008.

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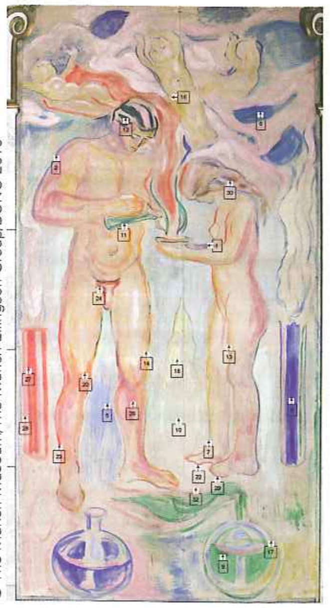

2
Metigo Map of "Chemistry" (≈ 444 * 220 cm²), (1909–1916) including the XRF tests prior to MOLAB Access Service in July 2008. Courtesy of Mirjam Liu 2008.

Research questions

Cleaning tests and MOLAB analyses addressed the following questions: Which of the tested cleaning agents are suitable for removing dirt from Munch's unvarnished oil paintings in the Aula of UiO? How do they affect a selection of Munch's colours when examined using visual methods commonly applied by conservators? How can conservation scientists expand on these results by employing mobile non-invasive examination techniques? What are the surface contaminants and what may be their causes? Finally, can the tests and the results of the examinations be used to propose a future cleaning regimen for Munch's Aula paintings?

Materials and methods

A preliminary, non-invasive study of Munch's techniques and materials in "Chemistry" was published in 2009.[16] The investigation established that the painting consists of two twill-weave canvases. Results from a hand-held XRF suggest that the white, lean and porous ground contains zinc, lead and chalk.[17] The greyish black underdrawings are an organic material, presumably a carbon-based black. The various oil colours often contain a mixture of pigments. Another major finding was that Munch used the same support, ground and painting technique without varnish for these four paintings. A recent examination of the other seven Aula paintings suggests (with minor exceptions) the use of similar materials and techniques throughout. This implies that the results from the cleaning tests on "Chemistry" may also apply to the other Aula paintings as well as to other contemporary paintings by Munch. However Munch's Aula paintings are quite young and some of the paints are still quite soluble. Thus organic solvents were considered unsuitable. This research therefore concentrated on a selection of aqueous systems such as natural saliva, distilled water, non-ionic surfactants in aqueous solution and gels, a chelating agent as well as dry mechanical cleaning.

Cleaning agents, application and removal

Selection of cleaning agents for "Chemistry" was guided by a rather defined review of publications on dirt removal from unvarnished oil painting,[18] however restrictions were posed by the young paint films and the large size of the painting. Another important factor was the availability of the cleaning materials. Cleaning agents had to be prepared, applied and removed by painting conservators working without the support of a specialist laboratory for their individual design (as most conservators do). The following cleaning tests (see Table 2), their application and removal[19] were carried out as follows:

- Test 1: Natural saliva[20] was applied with a cotton swab for c. 16 seconds.
- Test 2: Marlipal[21] 1618/25 (2 w/w% in distilled water with pH c. 5) was applied with a polyurethane sponge[22] for c. 16 seconds. The sponge was slightly moistened with the cleaning agent and softly pressed onto the test area. The sponge was never rubbed, wiped nor rolled back and forth. Application of the cleaning agent was followed by two subsequent removals with a cotton swab dipped in distilled water.
- Test 3: Brij 700-gel[23] (pH c. 6) was applied by brush. The gel was immediately removed with a dry cotton swab. This was followed by two additional removals with a cotton swab dipped in distilled water.
- Test 4: Triton X-100-gel[24] (pH c. 8) was applied by brush. The gel was immediately removed with a dry cotton swab. This was followed by two additional removals with a cotton swab dipped in distilled water.
- Test 5: Tri-ammonium citrate (TAC)[25]: 1w/w% TAC in distilled water was applied with a cotton swab for c. 16 seconds. Application was immediately followed by two removals with a cotton swab dipped in distilled water.
- Test 6: TAC: 1w/w% TAC in distilled water was applied with a polyurethane sponge. The sponge was slightly moistened with the cleaning agent and softly pressed onto the test area. The sponge was never rubbed, wiped nor rolled. Application of the cleaning agent was followed by two individual removals with a cotton swab dipped in distilled water.
- Test 7: A sponge of vulcanized natural rubber gum was rolled using slight pressure for c. 30–60 seconds.
- Test 8: A small lump of fresh white bread with washing soda without the crust[26] was rolled back and forth until the lump picked up dirt and turned greyish (not timed).

All the tests except the sixteen preliminary tests (see below) were performed on 2 x 2 cm squares. Their dimensions were controlled by using same size Melinex frames. Each application was timed with a stopwatch. When cotton swabs were employed, they were rolled in two opposite directions, except on the dark blue and dark green colours, where the cotton swabs were rolled only in one direction to reduce the risk of removing original pigments.

Table 1
shows how the cleaning tests were evaluated.

Table 1

Pigment loss						No pigment loss					
Minimal cleaning but pigment loss		Average cleaning but pigment loss		Good cleaning but pigment loss		Minimal cleaning		Average cleaning		Good cleaning	
Uneven	Even	Uneven	Even	Uneven	Even	Uneven	Even	Uneven	Even	Uneven	Even
-6	-5	-4	-3	-2	-1	1	2	3	4	5	6

Location of the cleaning tests

According to empirical experiences gained at the Munch-Museum, some of the paints used by Munch have proven to be more vulnerable to dirt removal than others: lean blues and greens, yellows, inorganic reds and white, weakly bound grounds all bound in oil.[27] "Chemistry" contains all these colours, but its yellows were omitted from testing[28] with the selected set of cleaning agents. The recent XRF examinations suggested that the majority of colours in "Chemistry" is composed of more than one individual pigment.[29] Including rather large areas of bare white ground (both lean and water sensitive),[30] the painting contains more than thirty different applications of oil colours. Half of these are painted as rather slim lines. When the number of cleaning agents to be tested was decided, it became clear that many lines and colour applications in "Chemistry" were too small to accommodate all the eight cleaning tests, including adequate uncleaned areas of the same colours.

The cleaning tests were performed in two periods in 2008, prior to and in the course of MOLAB's one week visit in July. First, two sets of eight tests were applied as preliminary tests and not timed to make

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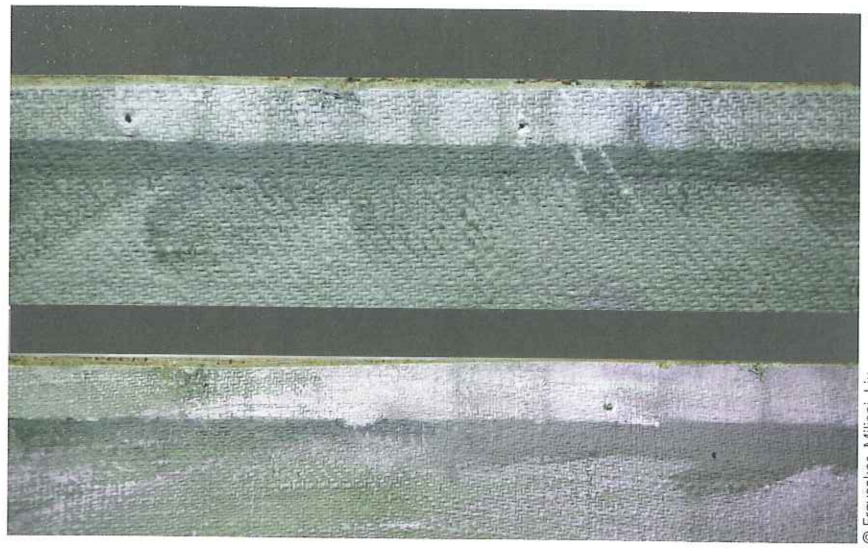

3
Detail of "Chemistry" with the preliminary two sets of cleaning tests along the extreme upper edge of the motif. This area has always been protected by a picture frame. Courtesy of Karen Mengshoel 2009.

Table 2
shows the first sets of 64 individual cleaning tests on *Chemistry* and their visual appearance judged with the naked eye, a microscope (magnification 63 x) and a UV lamp.

Table 2

Colours / Application area / Pigments	Dark blue, diluted / Laboratory flask below boy / Ultramarine, Prussian blue, Cobalt blue	Blue mixed with white / Background behind girl / Cobalt blue, Emerald green + white	Dark green, diluted / Laboratory flask below girl / Emerald green, Chrome green	Green mixed with white / Background behind boy / Emerald green, Chrome green + white	Dark red, diluted / Test tube / Vermilion	Red mixed with white / Cloud behind boy / Vermilion + white	Semi-solid paint on underdrawings on girl's ankle / Vermilion + white on Carbon black	Bare ground / Omnipresent / Zinc white, Lead white, Chalk
Test 1 Natural saliva	2	6	2	5	-4	3	1	1
Test 2 Marlipal 1618/25	-5	6	6	5	-3	6	4	3
Test 3 Brij 700-gel	-1	6	6	3	-1	3	-1	3
Test 4 Triton-X-100-gel	-1	6	6	6	-1	6	-1	6
Test 5 Tri-ammonium citrate (cotton swab)	-5	6	6	6	-3	3	-1	6
Test 6 Tri-ammonium citrate (sponge)	-5	3	4	3	-3	6	1	1
Test 7 Vulcanized natural rubber gum	2	2	2	2	2	2	2	2
Test 8 Bread dough	2	2	6	3	4	2	2	3

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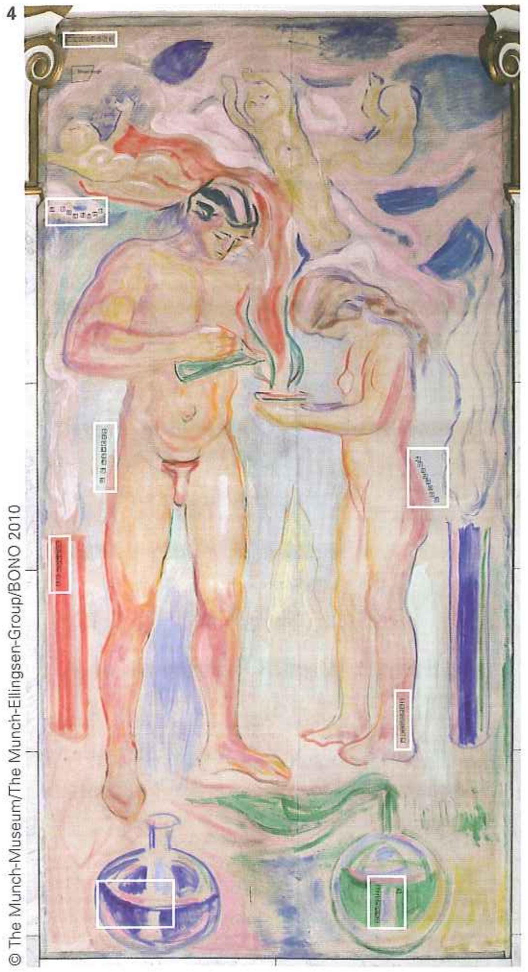

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4
Metigo Map of "Chemistry" with the eight sets of eight cleaning tests prior to MOLAB's analysis in July 2008. Courtesy of Mirjam Liu 2008.

5
Detail of "Chemistry" with the additional set of eight cleaning tests which were applied during MOLAB's visit. Courtesy of Karen Mengshoel 2009.

sure that at least some of the chosen cleaning agents would remove the dirt. It was also important to ensure that none of the cleaning methods would dissolve the water sensitive ground, and to get an impression of how the most effective cleaning agents would change the visual appearance on one of the light, opaque colours. Sixteen tests were located along the extreme upper edge of the motif, one set on the bare ground and the other set on a light red colour [Fig. 3]. This test area has always been protected by different types of picture frames. New frames will be attached when the painting is reinstalled in the Aula in 2011.

The initial procedure was followed by eight sets of eight tests, which were applied on eight different colours when the painting lay horizontally on a studio table [Fig. 4]. In all, this represented sixty-four individual test areas (in addition to their corresponding uncleaned areas) which were believed to be appropriate for one week of MOLAB analysis. At first, the horizontal position of the painting confined the tests (16 + 64) to the edges of the painting within reach of the laboratory microscope and proper working positions around the table.[31]

Finally, a single set of the same eight tests was performed during MOLAB access and when the painting was placed vertically and sideways towards one of the studio walls [Fig. 5].[32] These last tests were carried out in the light-red area close to the blue laboratory flask. During this step, the fibre-optic FTIR measurement was taken at the same spot prior to and after every application of the individual cleaning agent.

Visual examination

The visual appearance of the sixteen preliminary cleaning tests and the first sixty-four tests, their corresponding colours and the used cotton swabs were evaluated with the naked eye, under a microscope (magnification 63 x) and with a UV lamp. The eight additional tests were judged only with the naked eye.

Non-invasive reflectance FTIR

The MOLAB instrument consists of a portable JASCO® VIR 9500 spectrophotometer (50 x 50 x 50 cm^3; 35 kg) equipped with a REMSPEC mid-infrared fibre-optic sampling probe. The bench components include a MIDAC illuminator IR radiation source, a Michelson interferometer and a liquid nitrogen cooled MCT (Mercury Cadmium Telluride) detector. The fibreoptic probe, a bifurcated cable containing 19 chalcogenide glass fibres, allows for the collection of spectra from 6 000 to 900 cm^{-1}. An excellent signal to noise ratio (S/N) is obtained throughout the range with the exception of the 2 200–2 050 cm^{-1} interval, where the S/N decreases by approximately one order of magnitude as a result of the fibre Se-H stretching absorption. The investigated sample area, as determined by the probe diameter, is approximately 12 mm^2. Correction for background absorption is achieved by obtaining the reflection spectrum of an Al-plate mirror. The spectrum intensity was defined as the pseudo absorbance A' where A'= log (1/R). The fibre-optic probe was positioned perpendicularly (0°/0° geometry) and at a distance of approximately 2 mm from the sample surface. In "Chemistry", spectra were collected with 400 scans at a spectral resolution of 4 cm^{-1}. Two measurements were carried out for each individual cleaning area resulting in about 130 spectra of the surface. In addition, for comparison, spectra were also collected from painted areas that had been covered by the picture frame and from the picture frame itself.

5

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Mould fungi analysis

In March 2009, after dismounting the other Aula paintings, some small amounts of visible mould were observed on some of their reverse sides only, which might pose a risk to the painted surfaces [Fig. 6].[33] By then, the surface of "Chemistry" had already been cleaned mechanically (with polyurethane sponges).[34] "Harvesting Women", which was also mounted on an external wall and has similar materials, techniques and cleaning history, was examined for possible biological contamination. Samples of surface dirt were taken from its surface, edges and reverse side.[35] This sampling was performed with tape; brush onto tape and with polyurethane sponges. In addition, on the reverse side of "Women Reaching Towards the Light", two pieces of paper with visible dark spots as well as scrapings from the same surfaces were examined. All samples were studied under a microscope (Olympus biological microscope, AX 70, magnification 400 x). Cotton blue dye was added to highlight fungi.

Results

Cleaning tests evaluated by visual observation

The two preliminary sets of eight cleaning tests on bare ground and on light red in the area that had been protected by the picture frame showed no evidence of pigment loss. The final tests undertaken during MOLAB's visit on an exposed light red showed quite similar visual features as the light red in the following group of tests. The sixty-four individual cleaning tests resulted in six main categories of visual appearance. Table 1 shows how the tests were evaluated. Within each category of appearance there are two sub-categories: uneven and even. The three first main groups are assigned negative values (-6 to -1) because pigments were removed, which is unacceptable. The categories for minimal, average and good cleaning without the loss of pigments are given positive values (1 to 6).

Unacceptable

At least fourteen of the sixty-four tests were given negative values since some pigments came off, once (from dark red) with the cotton swab used for cleaning with saliva and thirteen times with either the first or second cotton swabs with distilled water during the additional removals (Table 2).

Inadequate

Other undesirable effects after cleaning were uneven appearance of gloss (which also included potential removal of original surface materials or characteristics), or dirt stains or a combination of the two. As many as sixteen tests among those that received positive values could be classified as uneven in one way or the other (see values 1, 3 and 5 in Table 2).

Acceptable

Minimal but even improvements after cleaning imply that colour and gloss were still obscured by dirt, although there might be circumstances where this is the only option for dirt removal. There were fourteen tests that were classified as acceptable, that is, no pigment loss, and the results were even but were minimally effective (see value 2 in Tables 1 and 2). The best cleaning results were found among the remaining twenty cleaning tests characterised as average and good. Three tests were given the value of 4 and seventeen tests 6 (see Table 2).

Colours in relation to the cleaning agents

To sum up the sixty-four tests, only one colour (dark green) can be cleaned with all of the cleaning agents, one (blue mixed with white) with seven agents, one (red mixed with white) with five, four (dark blue, green mixed with white, red mixed with white and bare ground) with three and finally, one (dark red) with only two. Simultaneously, the cleaning agents can be arranged according to the evaluated results: natural saliva (test 1) seemed useful for three colours, Marlipal 1618/25 (test 2) for four, Brij 700-gel (test 3) for two, Triton-X-gel (test 4) for five, 1w/w% TAC (applied with a cotton swab, test 5) for four, 1 w/w% TAC (applied with a sponge, test 6) for two, natural rubber gum (test 7) for eight and finally, bread dough (test 8) for six (see Table 2).

Analysis of the surface contaminants

Fibre-optic FTIR spectroscopy allowed analysis of the composition of the paint layers and of the superficial deposits. The equipment works in 0°/0° geometry collecting both specular and diffuse reflection; for these reasons fibre-optic FTIR measurements are very sensitive to surface materials active in infrared.[36, 37, 38, 39]

The FTIR measurements on the uncleaned areas provided a basis for identification of variable amounts of the following compounds: metal (Pb/Zn) carboxylates, sulphates, silicates and zinc oxalates [Fig. 7]. Metal carboxylates, also known as metal soaps, are identified by the absorption at 1560, 1540, 1510 cm^{-1} assigned to asymmetric stretching ν_a COO$^-$, appearing as derivative bands due to the effect of specular reflection. Sulphates and silicates are identified by the inverted signals at about 1 140 and 1 000 cm^{-1} respectively, ascribable to the asymmetric stretching of S-O and Si-O.[40] Oxalates are identified by observation of signals in the region of 1 300–1 370 cm^{-1}, and in particular by the presence of three inverted bands at 1320 and 1360 cm^{-1} (ν_s (-C-O) + σ (O-C=O) and 1634 cm^{-1} (ν_a (O-C-O), σ (H-O-H) that are specific for Zn-oxalates. Moreover, the small signal at 3 385 cm^{-1} (OH stretching) characterises them as hydrated zinc oxalates.

The results were compared with spectra collected on the areas that had been covered by the pic-

6

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6
Detail of visible mould fungi found on the reverse side of "Women Reaching Towards the Light". The dark spots were found on the paper and on the glue between the paper and the wooden framework. Courtesy of Mirjam Liu 2010.

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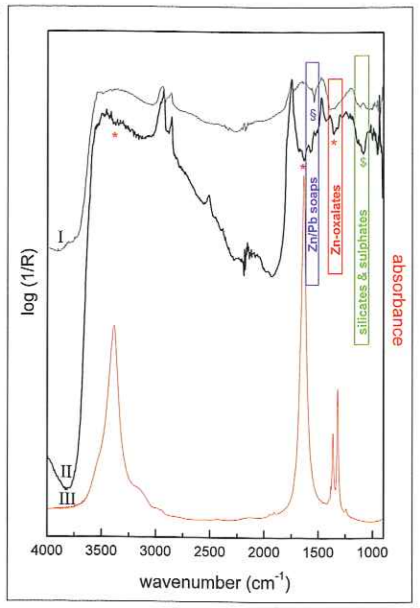

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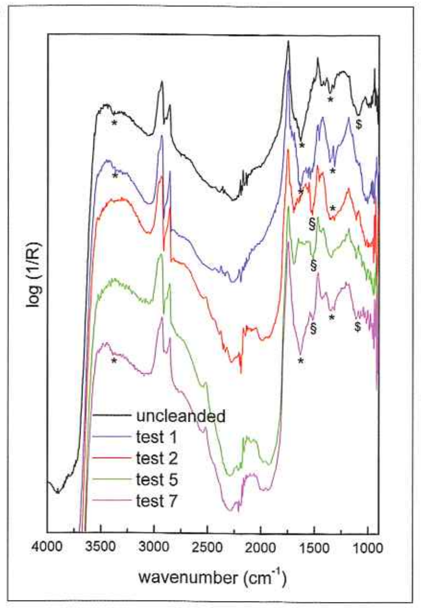

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Non-invasive reflectance mid-FTIR measurements: comparison of a spectrum collected on a uncleaned area with a spectrum of an area below the frame, the spectral region used for the identification of the different compounds are evidenced and a transmittance of hydrated zinc oxalate is reported.
* indicates diagnostic bands for Zn-oxalates, § for carboxylates, $ for silicates and sulphates.
Courtesy of Costanza Miliani 2010.

8
Comparison of spectra collected on the light red area close to the blue laboratory flask before and after the cleaning with:
test 1 = natural saliva,
test 2 = Marlipal 1618/25,
test 5 = TAC, test 7 = vulcanized natural rubber gum.
* indicates diagnostic bands for Zn-oxalates, § for carboxylates, $ for silicates and sulphates. Courtesy of Costanza Miliani 2010.

ture frame. Among the above-mentioned compounds, only metal carboxylates were identified in these areas. Metal carboxylates are formed by saponification, a reaction between the free carboxylic acids of oil binders and metallic pigments, lead white and zinc oxide being the most reactive. Conversely, sulphates and silicates were identified on the exposed areas in different concentrations, but not in the areas under the frame. They both are derived from an incoherent deposition of dust (as observed on indoor objects[41] and most likely are responsible for the grey appearance of the surface.

Zinc oxalates were detected in all the glossy solid or semi-solid paints, but not on the framed areas (nor on the frame that had been painted with a carbonate white pigment). This type of distribution suggests that zinc oxalates are the result of a specific interaction of the painting layer with the atmosphere rather than a degradation process of the painting itself or a simple deposition. It must be noted, however, that the oxalate signals are barely visible on the matt paint and probably because the spectra collected from these areas showed a worst signal to noise ratio due to surface roughness.

Evaluation of the cleaning efficacy was performed spectroscopically by comparing the spectral signatures of the various areas prior to and after the cleaning test. An example of the comparison of cleaned and uncleaned areas is shown in figure 10. It must be noted that the spectroscopic data are not strictly quantitative (since in reflectance mode the proportion between absorbance and concentration, Lambert-Beer's law, is not necessarily obeyed). However these data establish qualitatively whether or not contaminants and/or alteration products have been removed. It is worth mentioning that reflectance FTIR can also detect residues of cleaning agents as previously reported[42, 43] for sepiolite and dodecylsulphate in studies devoted to the optimisation of cleaning processes for a carbonate sculpture and a mural. In the present case, no residuals of chelating and surfactant agents were observed in "Chemistry" because they had been removed.

Biological contamination?

None of the twenty samples of surface dirt taken from the surface, edges and reverse side of "Harvesting Women" showed evidence of mould or any other biological activities.[44] On the reverse side of "Women Reaching Towards the Light", however, seven species of fungi were found in a few areas on the paper and in the glue attaching it to the wooden framework (*Acremonium sp., Aspergillus glaucus, Aspergillus niger, Aspergillus versicolor, Cladosporium cladosporioides, Eurotium herbariorum and Ulocladium chartarum*). Fungi on the reverse side of the rigid support may contribute to some biological activity on the surface of this single painting and must remain open to discussion.

Discussion

Implications for prospective dirt removal

The Aula paintings were contaminated with large accumulations of loose and firmly attached dirt. While this is unfortunate for their condition and visual characteristics, the present cleaning tests and their examination proved that every prospective cleaning project involves limitations regarding both the available range of cleaning agents and their manner of application and removal.

Given their visual impact, none of the present cleaning materials and techniques was adequately good (value 6) for all the eight colours tested. The non-ionic surfactant, like the Triton-X-gel (test 4), which had the highest number of good results, did nevertheless remove original pigments on dark, diluted blue, dark, diluted red and on the semi-solid paint on the underdrawings. With minor exceptions, these colours were difficult to clean by all the tests employed. In addition, Munch's full range of colours on "Chemistry" is much wider than these eight and other tints may be more or less resistant than the tested ones.

Only the two dry mechanical cleaning methods (tests 7 and 8) released no original pigments or metal soaps. Although, the latter resulted in two uneven areas (value 3). In just a few cases, the appearances of these tests were appreciated as average or good (values 4 and 6).

Neither the two mechanical tests nor saliva (test 1) decreased the invisible zinc oxalates on the surface of "Chemistry", but the non-ionic surfactant agents (tests 2, 3 and 4) removed some salts. Only the two tests with the chelating agent tri-ammonium citrate (tests 5 and 6) efficiently removed all of the oxalates (see Fig. 8). At the same time, original pigments were also removed during the removal of the citrate solution. This is unacceptable, and even if this is desired, the invisible zinc oxalates cannot be safely removed by this cleaning agent. Since other visible oxalates have been described as insoluble and only possible to wet with varnish, the zinc oxalates may also behave similarly if allowed to form visible crusts. But varnish is not an option on the Aula paintings (see below).

Recent studies have shown that paintings must be considered as continuously changing composite systems,[45] and the metal soaps and salts detected in "Chemistry" may need many more years before becoming visible. Forthcoming environmental improvements in the Aula, like reducing accumulation of airborne dirt by changing the heating system[46] and increased control of temperature and relative humidity may minimise the growth of reaction products on the painted surfaces.[47] Placing air-tight glass in front of the paintings may also delay the process, but this is inconvenient due to the large size of the pictures, as the building is a national monument and because major areas of glass (or any similar material) will disturb the Aula's acoustics. It is likely that the best procedure to prevent the emergence of metal soaps and salts is and will be continuously enhancing the environment of the Aula by preventive maintenance and a new cleaning regime for Munch's paintings.

The sources of metal soaps and previous treatments

Invisible metal carboxylates were identified both on exposed and unexposed areas on "Chemistry". They are formed by saponification of unsaturated fatty acids (palmitate and stearic acids) in the binding media or ground layers when the protons of the fatty acids are replaced by metal ions (or they could have been added as driers in oil colours). This phenomenon is common in paintings from the 1600s to the early 1900s.[48] Usually, they become visible as white surface depositions,[49] as fading and increased transparency of colours,[50] or as thickened imprimatura.[51] Often, the appearance of the artists' colours changed strikingly.

In earlier reports on visible lead soaps on the surface of paintings, they were described as irregular bloom that did not correspond with colours or brush strokes.[52] Other examples of lead soaps are not visible to the naked eye. Microphotographs of Rembrandt's "The Anatomy Lesson of Dr. Nicolaes Tulp" from 1632 showed round holes in the paint with diameters between 100 and 200 microns.[53] The holes were either filled with whitish material of lead soaps or with darkened varnish where these inclusions were long gone. Lumps of lead soaps were also analyzed in layers of ground, not yet visible on the surface.[54] Since their discovery, many examples of clearly visible lead soaps have been observed in numerous paintings in diverse collections.

Visible zinc soaps are accordingly widespread. They were for instance analyzed in "Falling Leaves" from 1888 by Vincent van Gogh (1853–1890).[55] The painting contained whitish or transparent zinc soaps which occurred following a rather recent application of varnish. This surface defect was described as transparent material slightly elevating through the paint and standing upright through cracks in the paint layer. It was suggested that several applications of varnish including the repeated use of solvents had triggered the formation of the aggregates.[56]

From early on, the paintings from 1927–1932 by Stanley Spencer (1891–1959) at the Sandham Memorial Chapel have endured recurring surface deterioration of blanched material.[57] By 1955, they had already been treated twice to reduce blanching and were probably varnished for the first time. By 1993, white bloom had been removed at least three more times and further coats of varnish had been applied. The white deposit could easily be removed with a scalpel. Analysis suggested that the blanched material contained degradation products such as fragments of varnish, fatty acids, inorganic pigments and possibly soaps. The influence of air pollution and moisture on the blanching problem could not be ruled out, and frequent varnishing did not prevent the continual whitening. On the contrary, it may be possible that all these applications were crucial for the whitening as in the case of "Falling Leaves" by van Gogh. In experiments, applications of varnishes containing polar, oxygenated resins dissolved in polar solvents were reported to extract soluble components from oil based films.[58]

In another, although unvarnished oil painting from the twentieth century, such as "Composition Without Title" by Serge Poliakoff (1900–1969) from the late 1950s, visible surface blooming caused by the formation of metal soaps was reported in 1990, about thirty years after completion.[59] In this case, zinc soaps had concentrated on the paint surface, and an increase of the blooming with rising humidity was observed. In the late 1980s, the bloom was removed with a dry swab and the surface was subsequently impregnated with a 5% solution of beeswax in white spirit.[60] To our knowledge, no recurrence of whitening on this painting has been reported.

These few cases suggest that the growth of white surface crusts (as metal soaps and other materials) can be actuated by solvents used in conservation treatments,[61] pollutants,[62] high temperatures and relative humidity. So far, visible white crusts of metal soaps have not been analyzed in any painting by Munch, although the Aula paintings may be candidates for similar, prospective and undesired changes in texture, colour and transparency. The treatment history of the Aula paintings involved solvents (for cleaning) at least in two campaigns (1973 and 1986); more than 90 years of accumulation (six known cleanings) and relative humidity (RH) up to 80 %.[63]

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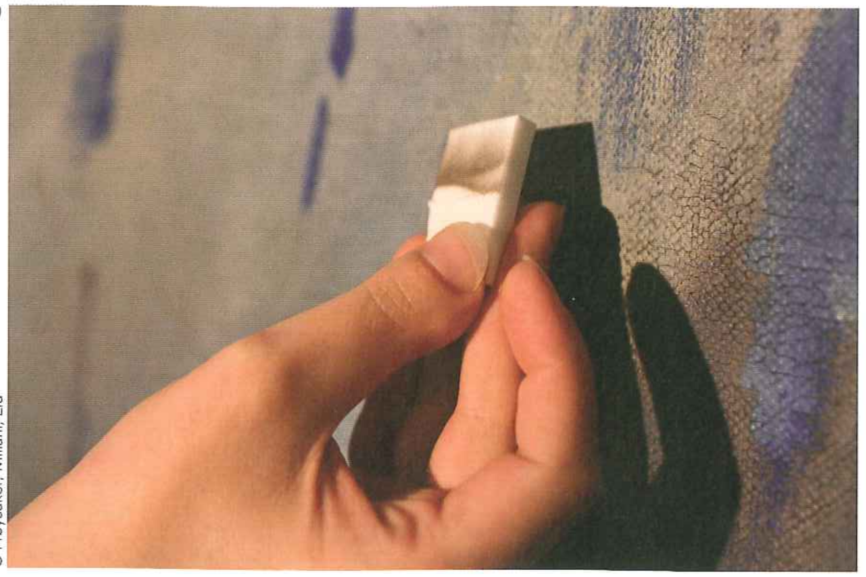

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A polyurethane sponge after being wiped over the surface twice. Courtesy of Karen Mengshoel 2009.

In addition, the paintings also have grounds containing white pigments of zinc and lead, which easily react with fatty acids in the oil. Still, neither "Chemistry" nor the other Aula paintings have any perceptible surface whitening or increased transparency; however, this may only be a question of time (see below). Since invisible metal soaps of zinc and lead were found on "Chemistry", and both on exposed and unexposed surfaces (covered by the frame), they must be related to the artist's painting materials (including possible paint driers)[64] and their ageing. This also implies that the same process is going on in every Aula painting, as well as in other similar paintings by Munch.

The sources of oxalic acid

Zinc oxalates were discovered on exposed areas on "Chemistry". They are, at present invisible and their presence was surprising because this phenomenon had never been reported. The formation of other oxalate patinas as a degradation phenomena has been widely observed and studied in the case of outdoor marble artefacts such as sculptures and monuments.[65, 66] Generally, these patinas are made of calcium oxalates ($CaC_2O_4 \cdot H_2O$), gypsum ($CaSO_4 \cdot 2H_2O$), silicates and phosphates together with iron compounds which are responsible for their characteristic yellow colour. In particular calcium oxalate may be present in two different forms, whewellite ($CaC_2O_4 \cdot H_2O$) and weddellite ($CaC_2O_4 \cdot 2H_2O$) according the number of crystallization water molecules. The fact that oxalate patinas are degradation phenomena is clear considering the reaction that is behind their formation: $CaCO_3 + H_2C_2O_4 \rightarrow CaC_2O_4 \cdot H_2O + CO_2\uparrow$.

Indeed, there is a consumption of original calcium carbonate due to the acid-base interaction with oxalic acid. On the other hand, it must be noted that once formed the oxalate patinas may act as protective layers on marble due to the lower solubility of calcium oxalate with respect to calcium carbonate at fairly low pH values.

Only in recent years, mainly thanks to exploitation of non-invasive methods for examining the surface (generally more sensitive to surface compounds with respect to conventional sampling methods), it has been observed that oxalate patinas are also present on indoor murals.[67, 68] ceramics[69] and even more surprising, also on easel paintings conserved in museums.[70] In particular, on some green polychromies the formation of copper oxalates has been proved by exploiting infrared or Raman spectroscopies.[71, 72, 73]

The origin of oxalates is strictly correlated to the origin of oxalic acid that is their precursor. Currently there are three different hypotheses on the possible origin of oxalic acid with regard to the degradation of artefacts.

1. Oxalic acid can originate from the fungi growth.[74, 75, 76]
2. Oxalic acid may be the last stage of oxidation reactions of natural organic polymers such as proteins or drying oils.[77]
3. Recent studies in the field of environmental chemistry have pointed out that oxalic acid is the most abundant dicarboxylic acid in the aerosol of urban, suburban and marine atmosphere.[78, 79]

In "Chemistry", zinc oxalates (although invisible to the naked eye and under a microscope 63x) were present in all the glossy solid or semisolid paints but not in the area under the frame. This last point suggests that zinc oxalates are the result of interaction of the painting layer with the atmosphere rather than a degradation process of the painting itself.

Zinc oxalate salts

There is no known literature on the appearance of zinc oxalate salts on the surface of oil paintings, nor on their removal. However, other metal oxalates have been analyzed on paints, especially oxalates of copper and calcium.[80] The oxalates found on murals have been described as an insoluble, obscuring, divergent and semi-opaque film. On easel paintings, the oxalates have been characterised as highly insoluble, greyish-brown surface crusts.[81] Recent SEM studies have revealed that other surface incrustations are often bound with the paint surface, thus making mechanical removal impossible.[82] In some cases, these materials have been wetted with varnish of low molecular weight resin.[83] But here, any varnish application would also involve possible organic solvent penetration into the paint structure, which may trigger further movement and protrusion of metal soaps.[84]

If the zinc oxalates analyzed on "Chemistry" becomes visible on the surfaces of Munch's Aula paintings, their removal may prove to be difficult. If and when they appear, there will be an urgent need to prevent them from producing a somewhat coloured, opaque surface layer that would distort the artist's colours including their great variety of textures and light reflections. Finally, and for the same reasons as for the formation of metal soaps, zinc oxalates may be found on the other Aula paintings and also on other works by Munch.[85]

Cleaning proposal

For the Aula paintings, dry and mechanical cleaning may represent the only cleaning option since these techniques removed neither original pigments, metal soaps nor zinc oxalates. If the zinc salt is interpreted as disposable both before it turns visible and after, it will have to be dealt with in both cases. On the other hand, if the invisible oxalates are considered acceptable aging products closely connected to original material before they become visible, it will be disputable to try to remove them before we have sufficient knowledge about their future formation and characteristics.

On other paintings, visible crusts of metal soaps have been removed mechanically or by increasing their saturation by impregnation.[86] However, it is still an open question whether filling micro fractures in surface soap deposits, for instance with a low viscosity resin solution may worsen the problem. Munch's Aula paintings are neither suitable for impregnation nor application of varnish. Furthermore, organic solvents could intensify the soap formation,[87] and any type of surface saturation will change both the artist's intent and the appearance of these paintings. Such treatments will alter the large areas of matte, opaque and porous grounds by making them transparent and by revealing the darkened canvas below. Moreover, they will diminish the tremendous variation in surface glosses.

Conclusions

In general, "Chemistry" shares the same materials and techniques with the other ten paintings in the Aula. The present study has revealed that "Chemistry" has started to form reaction products of metal soaps and zinc oxalates on its surface which are so far, detectable only with the aid of a mobile and non-invasive FTIR.

The preliminary cleaning tests on "Chemistry" demonstrated that any prospective dirt removal must be performed without the use of our selection of aqueous systems because they can remove original pigments. In the case of the Aula paintings and prior to a future development of more suitable cleaning agents for monumental and unvarnished oil paintings, mechanical cleanings seem to be the only preferable approach. Since vulcanized natural rubber gum and bread dough used on "Chemistry" only slightly removed parts of the dirt, but not pigments, metal soaps or zinc oxalates, we had to look for another mechanical tool to improve the dirt pick up. Following access to the MOLAB dry polyurethane sponges were used for this purpose on the Aula paintings. Although not tested to the same extent as the areas cleaned with rubber gum or dough on "Chemistry", the efficiency of these sponges[88] when used dry (no water or organic solvents) is apparent [Fig. 9].

Before the present renovation of the building the indoor climate of the Aula contained quite extensive amounts of dirt, and the cleaning history of its paintings clearly indicated short intervals between the previous treatments. It is hoped that the renovated hall will generate less airborne pollution when the paintings are reinstalled in spring 2011, but the prospective and extensive use of the room also implies continuous dust production. To extend future intervals between *major* cleaning of the paintings, we think it would be important to introduce *minor* cleaning each year to brush off the inevitable accumulation of loose dust (into a tip of a suitable vacuum cleaner) before it becomes firmly attached to their surfaces.[89]

Suggestions for further research

Sooner or later the soaps of lead and zinc and the zinc oxalates may occur as visible intrusions on the painted surfaces of the Aula paintings. To be able to delay this process and to prevent possible expansion of these products with proper means before any visible growth we need to gain a better understanding of their prospective aging processes. Since visible metal soaps have been reported to be removable whereas visible oxalates of copper and calcium have been characterised as insoluble, priorities should be given to gain more insight about zinc oxalate formation.

Following renovation of the building and subsequent reinstallation of the paintings, the quality of the air inside should be evaluated and a corresponding aging experiment conducted in situ on the reaction between zinc oxide and oxalic acid in the hall. The latter can be performed by a suitable arrangement close to one of the paintings with a battery of test pieces of canvas (2 x 2 cm^2) covered with a layer of lean and unvarnished zinc oxide. Each summer and prior to the suggested minor removal of dust, one of the test pieces can be taken down, sealed and sent to the MOLAB laboratory for analysis. With this arrangement we trust that the proposed monitoring, test materials and further analysis will provide reliable data to act upon if and when necessary.

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Zusammenfassung

Nicht Invasive Auswertung von Reinigungstests an »Chemie« (1909–1916)

Ein großformatiges Ölgemälde auf Leinwand von Edvard Munch

Es gibt weder Berichte über einen originalen Firnis noch wurde ein Firnis auf »Chemie«, einem von elf großformatigen Gemälden von Edvard Munch in der Aula der Universität von Oslo, nachgewiesen. Die besonderen Schwierigkeiten bei der Reinigung von ungefirnissten Ölgemälden auf Leinwand insbesondere von Munch und anderen Malern des 20. Jahrhunderts fanden im Vergleich zu gefirnissten Ölgemälden bisher wenig Beachtung. Das Gemälde »Chemie« ist beispielhaft für die Probleme, die diese Gemälde mit sich bringen. Die Oberfläche war durch luftgetragene Schadstoffe stark verunreinigt. Sie ist schwieriger zu reinigen und fragiler als vergleichbare gefirnisste Ölgemälde. Mit dem mobilen Laborservice EU-ARTECH MOLAB wurden 2008 wurden innerhalb von zwei Etappen an »Chemie« insgesamt 88 Reinigungstests durchgeführt, 80 davon vor und acht während einer Woche. Ziel war es, ein Bewertungsverfahren des Reinigungssystems vorschlagen zu können. Nicht invasive mid-FTIR spektroskopische Messungen ermöglichten die Identifizierung und Aufzeichnung von verschiedener Oberflächenverunreinigungen, wie z.B. Sulphate, Silikate, metallische Seifen und Zinkoxalate. Untersuchungen mit dem bloßen Auge und Infrarotmessungen fanden Anwendung, um die Effektivität und die Risiken der Reinigungstests zu evaluieren.

Keywords: ungefirnisste Ölgemälde, luftgetragener Schmutz, Reinigungstests, nicht invasives FTIR, Blei- und Zinkseifen, Oxalsäure, Zinkoxalate, Reinigungsvorschlag.

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[16] Including "New Rays", "Women Harvesting" and "The Fountain". Frøysaker and Liu 2009 pp. 47–58 (see ref. 5).

[17] In fact, zinc was found in all XRF measurements and it also seems to be one of the elements in the reverse side preparation of the canvases from 1925–26.

[18] The review was defined by the following preferances: the cleanings had to be on genuine oil paintings from the 20th century with no varnish, and that the reported cleaning agents had to be easy available as well as applicable for 220 m².

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[27] Personal discussions with Dr. Biljana Topolova-Casadiego at the Munch-Museum.

[28] Yellows were omitted for three reasons, some areas were too small to accommodate all the tests, others had already been cleaned when the glue paste was removed and the remaining ones were located too far away from the edges of the painting.

[29] Frøysaker and Liu 2009 p. 64 (s. ref. 5).

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[85] Interestingly, in 2009, MOLAB also found invisible zinc oxalates on the surface of Munch's "Puberty" (1894–95). This painting contains no ground layer. Zinc was however found in all XRF measurements and suggests the use of zinc-based white pigments during a previous lining of the canvas. Personal discussions with Dr. Biljana Topolova-Casadiego at the Munch-Museum.

[86] See e.g. the treatments of Stanley Spencer's paintings in the Sandham Memorial Chapel as well as the treatment of Serge Poliakoff's "Composition Without Title".

[87] van Loon 2008 pp. 196–197 (see ref.48).

[88] http://www.scribd.com/doc/28005037/Painting-Dry-Cleaning-Table (accessed 9. December 2010).

[89] Minor dirt removal could be performed each summer when there are no concerts or festivities in the Aula. This should also include yearly reports on the paintings' condition to raise the level of maintenance.