

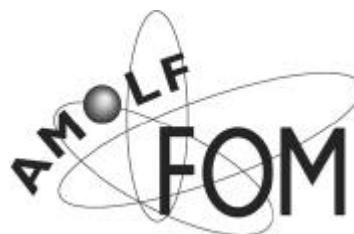


**A multidisciplinary NWO PRIORITEIT project
on
Molecular Aspects of Ageing in Painted Works of Art**

Final report and highlights

1995-2002

Edited by Mark Clarke & Jaap Boon



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The MOLART participants

This list includes the principal participants and organisations that contributed to the MOLART projects at any time. Those people that have been supported by funds from the project are marked with (M) or (M-fellow).

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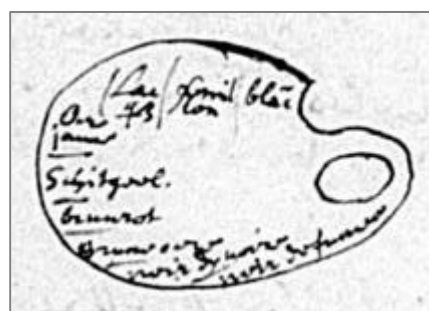
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Palette, from the de Mayerne manuscript



The MOLART experience

Jaap Boon

A big challenge for MOLART has been to stay in contact with prevailing ideas in conservation and material science of art while exploring new areas of research that would elucidate dynamic molecular processes during the life time of works of art. Residual molecular signatures, distributions of particles and organic constituents in the build up of the painting carry information about the history of the works and the materials available to their makers. MOLART sought new eyes to explore these vistas and often found that the resolution in its field of vision was still too limiting. Many burning questions remain unanswered. Partly because time erased the slate, partly because sampling is too limiting or impossible, partly because samples are so small that traditional organic analytical chemistry can not cope. Little did we realise that our questions were nanoanalytical in nature. Interface chemistry that completely changes the composition of the original biomaterials used to make paintings – vegetable oils, tree resins, egg lipids and proteins, polysaccharide gums – creates new materials with a surprising solidity and sturdiness. Such insights were hunches when the project was conceived. Now they have become believable.

The committee for selection of Priority projects of NWO believed in 1995 that a lot was to be gained from a molecular approach in studying ageing processes in paintings. New knowledge that might improve the risk assessment of conservators. New information about the making process and materials gleaned from documentary sources. New insight sprouting from the joining of some many fields and disciplines involved in the keeping of paintings. This multidisciplinaryity was unique for the study of paintings in the Netherlands. MOLART had a very fundamental streak. Input was received from J.R.J. van Asperen de Boer, E. van de Wetering, R. de la Rie, A. van Grevenstein, A. Phenix and many museum conservators with a paint material interest here and abroad. Rather than studying existing ideas in poorly documented and scattered sources, MOLART attempted to see with new eyes bringing young academics and practitioners together from physics, chemistry, conservation science, art history, practical restoration and conservation. This innovative approach, the MOLART formula, also drew unfriendly fire: How could we ignore earlier work? We didn't. But we wanted to see first and then compare our notes with literature sources and the opinion of established experts. New, fresh looks with modern technology could well lead to new insights. And it did! Over the years the contribution by van Asperen de Boer has been extremely valuable and deserves special mentioning. He was the person that had the patience and perseverance to keep bringing important concepts and details to everyone's attention. He freely shared his love of art and lifetime experience with studies painting in the many working group meetings and so contributed greatly to the success of the MOLART formula.

MOLART funds supported the people while AMOLF provided a lot of laboratory support using the already existing instrumentation in our biomolecular mass spectrometry group. MOLART has directly or indirectly supported ten PhD studies, several master and bachelor theses, and five project leader positions. The institution of fellowships for study of specific topics coming up during the project proved to be a flexible tool. It allowed the study of several conservation oriented research projects such as the Pettenkofer treatment (Schmitt), the effect of solvent cleaning (Sutherland), computer modelling of cracking (de Willegen), and

reconstruction of 19th century oil pretreatment and paint making processes (Carlyle). Some laboratory support was received from sponsoring organisations such as Shell, AkzoNobel and ICN. New instrumentation was also acquired by MOLART to improve the imaging of the spatial distribution of paint materials in tiny paint chips analysed as cross sections and to perform advanced infrared reflectography. Instrumental development work at AMOLF was supported with funds from FOM and NWO that made it possible to build a laser desorption ion trap mass spectrometer (SLIM). The instrumental concept however also had some unforeseen limitations and was very difficult to integrate with other more imaging oriented approaches that became available in the course of the project.

Over 120 scientific publications and about 280 public presentations of research work at conferences has given MOLART public exposure. Further public exposure was realised by participation in radio and TV shows (VPRO Noorderlicht, NOS-Teleac, National Geographic Channel) and articles in newspaper and magazines. Knowledge transfer was also achieved by courses given via the SRAL organisation in Maastricht and laboratory support given by MOLART to many SRAL students during their external training in museums.

What has been achieved is expressed in the contributions to this final report and highlights written by the researchers of the MOLART team. Contributions are organised under the headings of core research, pigments, organic constituents and media, new physical analytical techniques, conservation-oriented studies and multidisciplinary painting studies. None of this work could have been achieved without the tremendous help of museum staff in the Netherlands and abroad. They were the eyes in the field and they posed interesting and challenging questions. I thank them for their patience because many answers were not easy to get and took a lot of time.

Some very daring problems were put forward on the instability of oil paint that expressed itself as metal soap inclusions and protrusions, increased transparency, efflorescence and blooming. All these phenomena relate to the ageing process of oil paint leading to metal carboxylate ionomeric networks that sometimes fail to develop properly. The reasons for these failures are manifold and range from poor quality materials or painting technique to traditional conservation methodology at elevated temperature or improper storage in the past. Research into the mechanism of the failure to form a stable oil paint is ongoing in the De Mayerne Program. The initial work was inspired by the efforts of Petria Noble from the Royal Cabinet of Paintings the Mauritshuis in the Hague who contributed greatly to the recognition of metal soap aggregation in a number of paintings of their collection and those elsewhere. Many conservators have seen the damage caused by metal soap aggregation in paintings ranging from the 15th to the 20th century. The studies of mobile and stationary phases in oil by MOLART have provided a first theoretical framework for the explanation of metal soap formation but a lot remains for future studies.

The topic of metal soaps is just one example of the many interesting research projects that have taken place during the project. MOLART wanted to attract and inspire young scientists and art historians to studies of paintings. The MOLART report series of PhD theses are proof of their MOLART experience and evidence of the success of the MOLART formula and the project. Finally, I wholeheartedly thank all the individuals and organisations that contributed so much to the success of MOLART.

Reflections on MOLART

J.R.J. van Asperen de Boer (invited contribution)

The MOLART project was essentially due to the initiative of Professor Dr. J.J. Boon (Chair of Molecular Palaeobotany, University of Amsterdam, and a mass spectrometrists). In view of the requirement that proposals for the Priority project of NWO (Netherlands Organization for Scientific Research) should have “societal relevance” he sought from the early stages collaboration with restorers, art historians and conservation scientists in the Netherlands, interested in multidisciplinary work. Some Dutch museums were also approached. The final and accepted proposal reflected the various interests and back-ground of their supporters. A fairly broad range of research topics was the result but they were all limited in essence to easel paintings. The proposal being primarily embedded in the AMOLF (FOM Institute for Atomic and Molecular Physics) laboratory there was - understandably - strong emphasis on research related to molecular aspects of e.g. ageing and conservation and a preference for mass spectrometric techniques.

In retrospect a limitation of the number of research projects would have been more efficient. The selection of topics to be studied demanded from the beginning considerable effort from the participants to reach sufficient mutual understanding. This required time. The specialized language used by the participating scientists is not easily grasped by restorers and art historians, not even by the present writer trained as an experimental physicist. On the other hand it was not always realised by those new to this field that there is a vast, albeit scattered, body of conservation literature and that it is relevant to be acquainted with some of it if only to recognize and avoid difficulties and pitfalls.

The materials encountered in artistic works - and certainly in highly individual easel paintings - are a function of their genesis and vicissitudes. It is not self-evident that their complexity can be easily reproduced in the laboratory. Thus physicists and chemists cannot *per se* define their own parameters and use known materials in experiments.

During the project several multidisciplinary frames of collaboration developed and some of those are maintained in the present De Mayerne project, the successor of MOLART.

It proved a great advantage that in this NWO Priority project the various research topics did not have to be spelled out in great detail. All too often - especially in the humanities - it is required to guarantee the result almost before a proposal can be granted. This, together with the considerable material and personnel support from AMOLF, has allowed flexibility and permitted changes of direction within the programme during the project period.

It is obvious that in experimental science one must try something first and see what the results are. Setbacks are normal. Thus the SLIM instrument designed and developed to improve the identification of organic materials notably in paint cross-sections by imaging techniques did not for various reasons perform as expected. A more recent instrument using SIMS (acquired by AMOLF with NWO support) is now also used for such work in the de Mayerne project and seems more promising than SLIM.

The results of research carried out within the MOLART project have produced a number of theses and numerous other publications. Through lectures and also during

conferences in the conservation field a number of publications have appeared in the regular conservation literature. The audience which would most benefit from the MOLART research has thus been addressed.

It would seem, however, that more earmarked financial support and attention could have been given to produce tangible results from the application of MOLART research. Two examples may illustrate this.

1: In research on molecular changes of aged pine and larch resins the index for the degree of oxidation (IDOX) was measured, This represents in principle a dating method for such samples from actual paintings. It would be most helpful in conservation and technical studies in art history not only for wax-resin relined canvas paintings but also for paintings with suspected original or intermediate varnish layers. Elaborating such a dating method is probably too much applied science for those used to work in fundamental science. It may be fundamental however for the other disciplines.

2: One of the MOLART results was a proposed model of the oil paint polymer network: "Molecular aspects of mobile and stationary phases in ageing tempera and oil paint films" (Boon *et al.* 1996). It provides "... the basis for further analysis of potential effects of conservation treatment and cleaning procedures" and would also allow an explanation of the observed behaviour of certain pigments such as orpiment and vermilion in oil paintings leading to deterioration. So far this more applied research has not been undertaken nor does it seem to have been elaborated elsewhere.

MOLART research, so closely associated with the suprauniversital and at least post-graduate AMOLF laboratory, has introduced in the Netherlands a quality level for such studies, which previously was virtually non-existent. It was certainly not stimulated by ever-diminishing research possibilities at Dutch universities. This shows how crucial NWO financing is. The very fact that the successor of MOLART, the de Mayerne project, arose out of the collaboration of the NWO sections on humanities, sciences and chemistry and is again supported for the period 2001-2008 is proof of the important role the MOLART project has played.

The contributions of MOLART to conservation and technical studies of paintings

*Aviva Burnstock, Department of Conservation and Technology,
Courtauld Institute of Art, London (invited contribution)*

It is a challenge to select from the many MOLART projects those that have been most relevant and useful to the technical studies of paintings and their conservation. Technical studies of original painting materials and their deterioration, including the characterization of non-original material and artists' reworking provide data that is crucial for facilitating the decision-making processes involved in any conservation intervention. MOLART projects that may inform conservation practice therefore include material studies (of pigments, binder and coatings, for example) as well as the information gathered from the technical study of important paintings, or groups of works by one artist. These include, for example, Groen *et al.* study of paintings by Vermeer [1] and Rembrandt [2]; Noble *et al.* [3]; Henriks *et al.*, of Frans Hals' Civic

Guard series [4]; Jones *et al.* of Joshua Reynolds [5] and several other studies of this kind. Links with conservation practice led to the MOLART studies of the condition of resin used in wax-resin mixtures for lining paintings [6] and investigation of paintings subjected to the Pettenkofer process [7]. Technical studies carried out under the MOLART banner, for example, of the deterioration of yellow and orange arsenic sulphide pigments (Wallert *et al.*) and of copper green paints (by M. van Eikema Hommes and K.J. van den Berg) may assist conservators' and art historians' interpretation of changes in materials, thus facilitating a notion of the outcome of the passage of time on the appearance of paintings. The combination of these analytical studies with historical context provided by documents and treatises related to the historic use of pigments by artists is exemplified in the useful thesis by Margriet van Eikema Hommes in her chapter that focused on indigo, and in her earlier publication on colour changes [8,9].

A focus of the MOLART group, on the condition of drying oils used for painting, including work by J.D.J van den Berg and colleagues [10], and latterly, on the interaction between oil and inorganic pigments [11] has provided data on the condition of oil paint on curing, and in the first stages of ageing. This led to a revised chemical model of aged paint, that has been used to explain phenomena such as efflorescence in paintings and other kinds of deterioration, and may have implications for cleaning paintings [12]. Data from studies on drying oils, and those focusing on di- and triterpenoid resins used in paint and coatings [13,14] has contributed significantly to the body of knowledge in the field outlined by Mills and White [15].

MOLART's contribution to the development, or adaptation of analytical methods for the study of small samples from paintings, in particular those that offer the opportunity for spatially resolved characterisation of painting materials in paint cross-sections, has benefited the conservation scientist and hence the conservator. Non-invasive methods for characterization of painting materials are in general limited by poor spatial resolution, and detailed chemical analysis of organic materials from single layers in paintings is problematic, both for sample taking and interpretation. The application of non-destructive analytical techniques to stratified samples that may be available from the damaged areas of paintings has been enhanced during the MOLART program. The application of new methods such as TOF-SIMS [16] and the adaptation of software for FTIR imaging and analysis of paint cross-sections [17,18] generates new data that shows promise for future material studies of samples from paintings.

Cleaning of paintings, that may involve the removal of surface dirt, resin, oil-resinous and sometimes proteinaceous coatings from the original paint surface, is frequently part of a conservation campaign. Understanding the chemical character of the coating material, and its relationship, both physical and chemical, to the original paint beneath is crucial to the conservator. Characterization of the class of material used for a varnish, its polarity and mechanical properties, directly influences the choice of method for its removal, and the risks associated with a particular method may be assessed. MOLART projects that have contributed to this understanding (in addition to the fundamental material studies discussed earlier) include the study by Kenneth Sutherland of solvent extractable components of oil paint films [19]. Sutherland's thesis includes data that suggests that fatty acids are selectively removed (according to polarity) from young oil paint even in the course of varnishing using solvents of relatively low polarity. The experiments were carried out on both relatively "young" paint films and naturally aged material of significant age, and these findings may have significant implications for treatment of paintings, including the

application of temporary varnishes during cleaning [20]. Arguably, these studies could help to formulate risk assessments for first cleaning of oil paintings. As part of risk assessment for cleaning, and the continued importance of solvents for cleaning paintings, Phenix's review of solvents and solubility is significant, and a prelude to his ongoing research in the field [21].

Publication of papers from the MOLART group in international journals in the conservation domain, such as *Studies in Conservation*, the *American Institute for Conservation* and *Zeitschrift für Kunsttechnologie und Konservierung*, and the group members' contributions to working groups and conferences of the International Committee for Conservation (ICOM-CC) and to the International Institute for Conservation (IIC) has raised the profile of the group and its findings. Dissemination of information from technical analytical studies to the conservator is crucial if the results of MOLART studies are to have influence on further research in conservation, and possibly eventual influence on practice. Communication of data from fundamental material studies to conservators, many of whom are expert art historians or practitioners rather than scientists, continues to be a relevant consideration. The importance of the focus of MOLART on fundamental studies of materials should not be underestimated - in recognition of significant gaps in understanding how paintings materials change from the moment the artist has finished a painting and arguably even before. The data gathered from experimental studies and reconstructions may offer statistical reliability where studies of samples from paintings are hampered (in the scientific sense) by limited availability and lack of reproducibility. Variability reflects the nature of painting practice, and recognition of this is of vital importance in the interpretation of analytical data from paintings.

The relationship between natural ageing and the material changes that occur in artificially aged samples remains an issue for different materials used for painting, especially for composites. These problems highlight the importance of caution in making deductions about the visual or practical significance of data gleaned from the application of new analytical techniques for the study of individual painting materials or "reconstructions".

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Core research projects

These projects were relevant to all components of the MOLART programme.

Documentary sources on oil painting techniques

Documentary sources are an essential ingredient in multidisciplinary studies of painting materials and historical painting technique. Several studies have been performed and documented in the PhD thesis of Margriet van Eikema Hommes. One of the highlights is the study of the copper green glazes. Many of these glazes have undergone severe colour changes on the paintings. At the start of the project on Copper glazes many questions existed with respect to their preparation and fate.

Sources on copper green glazes Margriet van Eikema Hommes

The study of a large group of historical recipes and application instructions has established a clear picture of the way glazes of the historical pigment verdigris were used in oil paintings. This knowledge assists in the interpretation of data obtained from scientific research into transparent copper-green paints in Renaissance and Baroque paintings and contributes to a greater insight into the changes to which these paints have been subject in the course of the centuries.

First and foremost, there turns out to be little basis for the assumption that copper-green glazes consist of a *copper resinate* made by artists by dissolving verdigris in heated varnish. In fact, the instructions show that the glaze was made with finely ground verdigris mixed in a cold oil or varnish medium. The historical term *verdigris* related to a range of green and blue-green corrosion products of copper, which painters rarely differentiated. In addition, the various methods for distilling unrefined verdigris resulted in products of varying degrees of purity. These processing methods explain the frequent presence of copper chlorides and copper sulphate pigments in the glaze.

To date, analyses of binding media of glazes have generally revealed linseed oil to be the main component. The oil often appears to have been heated to a high temperature. A small amount of resin is also regularly identified. The old texts discuss the preparation and function of these media. Artists boiled the oil to obtain a thicker and more fluid medium that provided a more uniform and glossy glaze. To accelerate the thickening of the oil, a little lead-white or lead-oxide was often added. This may explain why in copper-green glazes with no lead pigments nevertheless sometimes a small amount of lead is identified. The addition of varnish was intended to improve the glazing properties of the paint.

The earliest description of the production process of verdigris in Montpellier, in a manuscript by De Mayerne



The ancient instructions show that verdigris could be mixed with a variety of pigments, while the composition of the underpainting could also vary. Analyses of copper-green glazes often indicate the presence of chalk. The historical texts show that this may be due to various reasons. Painters may have added a yellow lake with a substrate of chalk to the verdigris. The verdigris may also have been adulterated with chalk. The purification methods employed by artists appear not to have removed the substance.

From the early 16th century painters referred to the lack of colour-fastness in verdigris glazes. On the other hand, they praised the durability of the paint. Sources show that the colour-fastness depended on the purity of the pigment and the skill with which the specific painting technique for this particular pigment was employed. To prevent discoloration in the pigment, some varnish was regularly added to the oil medium, sometimes verdigris was mixed in pure varnish, although it was more usual to apply a layer of varnish over the glaze as soon as it had dried. At present, brown layers that contain no copper, which seem to be a part of the original paint layer, are regularly found on copper-green glazes (fig.2). Modern researchers have assumed that these layers are the remnants of a yellow lake that was applied over the verdigris to create a warmer green. However, the historical texts reveal that these layers are just as likely to be the remains of the local varnish.

Key publication: M. Van Eikema Hommes *Historical Painting Recipes and their Relevance for the Scientific Study of Paintings* (2002) PhD Dissertation.

Accelerated light ageing

Within the framework of the MOLART Project a climate room with an installation for accelerated light-ageing was designed and constructed at the Stichting Restauratie Atelier Limburg (SRAL) in Maastricht.

The MOLART light ageing facility René Hoppenbrouwers

The accelerated light ageing facility has been designed in order to support several MOLART projects studying the ageing properties of different historic binding media and paint systems such as natural resins, paint additives, egg tempera paints, indigo paints, lead white paints made according to 19th century recipes, asphalt and bitumen, arsenic sulphides, and ‘copper resinates’.

Instead of using set-ups used for industrial purposes such as the fadeometers or weatherometers it was decided to build an open construction with banks of fluorescent lights in order to create a less intense form of accelerated light ageing. A rack with four compartments of about 1 square meter each, and containing a total of 48 fluorescent tubes (36 W Philips Colour 96) was placed in a small room that could be air-conditioned and humidified. By means of transparent Lexan® polycarbonate sheets ultraviolet radiation could be filtered out. The rotation of the samples at set time intervals guaranteed an average illuminance of 10.000 Lux for all samples under study during a specific experiment.

The MOLART set-up ensured an ambient condition of 20 to 24 degrees Celsius and a relative humidity between 40 and 44%. As distinct from closed lightboxes with fluorescent lighting that are frequently used in conservation science, the temperature of the air surrounding the samples could be kept relatively low and the relative humidity fairly close to the museum environment. This prevented a drop of the absolute moisture content in the samples to low levels, and prevented the migration of volatile components of the sample materials. Therefore, compared to natural ageing, the physical and chemical degradation processes during accelerated ageing were influenced as little as possible.

Light ageing chamber at SRAL



A comparison of light-accelerated ageing regime of five conservation institutes in the United Kingdom with the that of SRAL / MOLART pointed out several differences in performance of the lighting cabinets, therefore making the reproducibility of experiments in different set-ups impossible (Saunders and Kirby, 2001). Nevertheless performing comparative accelerated light-ageing studies using one and the same facility under controlled and monitored conditions is still a valid method contributing to our understanding of the ageing properties of historic paint materials.

See: David Saunders and Jo Kirby, 'A comparison of light-accelerated ageing regimes in some galleries and museums', *The Conservator*, (25) 2001, pp.95-103

Development of advanced analytical instrumentation

The input from AMOLF physicists made it possible to explore several new avenues of scientific examination using, adapting and integrating technologies that could provide information on chosen microscopic areas of interest. In the early stages of the project laser-aided spot analytical methods were emphasized and applied with some success. New technologies that became available during the project changed the emphasis toward imaging analytical approaches.

Chemical imaging *Ron M.A. Heeren*

Chemical imaging techniques developed for the analysis of the spatial distribution of organic molecules have provided new insight into the organic chemistry of embedded paint cross-sections. In the framework of this MOLART core project innovative instrumentation and methodologies in the field of mass spectrometric imaging, FTIR imaging spectroscopy, UV/VIS imaging spectroscopy and Infrared reflectography have been developed. FTIR imaging microscopy is used to deliver a two dimensional image of the functional group distribution, revealing chemical aspects of the binding medium in each individual paint layer or the alteration products of the molecular constituents of the paint materials as well as their ageing products. Secondary ion mass spectrometric (SIMS) imaging of the paint cross-section surface provides the molecular identity of chemical compounds in the layer. The development of spatially resolved mass spectrometric imaging instrumentation has expanded the chemical imaging capabilities in elucidating the molecular structures at surfaces. It is a very powerful extension of the range of organic analytical methods already available. The integrated application of FTIR and SIMS imaging reveals the presence, spatial distribution and molecular identity of various organic and inorganic compounds. From the many experimental results it can be concluded that a single chemical imaging technique, however powerful it may be, usually only provides part of the chemical information embedded in the cross-section. A careful approach, considering several advanced analytical techniques, is imperative to unravel the complex picture of organic and inorganic surface chemistry of paint cross-sections.

The strength of the combination of these chemical imaging techniques is demonstrated in the characterisation of the organic constituents found in microscopic protrusions in the surface of the painting *The Anatomy Lesson of Dr. Nicolaes Tulp* by Rembrandt van Rijn, 1632. (left) During the period of examination and investigation that preceded its restoration at the Mauritshuis, it was observed that the entire surface of the painting is covered with small, crater-like holes, with a diameter ranging from 100 to 200 micrometers. Generally the holes are completely round and regular, although due to abrasion some have a flatter profile and a more irregular contour. Some are filled with darkened secondary material, but most contain whitish substance which protrudes through the paint.

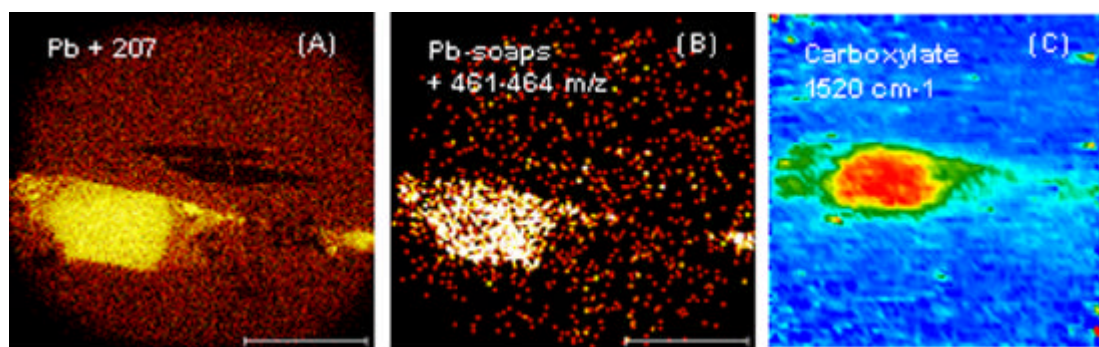
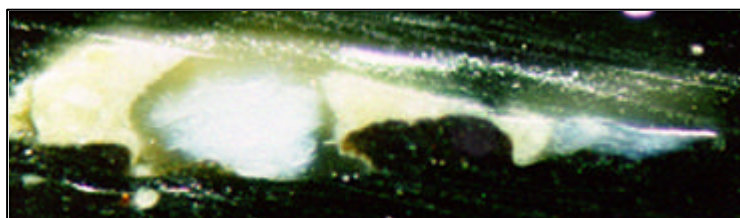


Cross-sections with a variety of protrusions were subjected to FTIR imaging and mass spectrometric imaging. FTIR chemical imaging, showed a distinct spectral difference in the chemical constituents of the inclusion compared to that of the

surrounding ground layer. The main organic functional groups identified in the inclusions are carboxylates with absorbencies in the range around 1500 cm^{-1} and 1700 cm^{-1} as well as a strong C-H absorbance around 2900 cm^{-1} . The samples were also subjected to spatially resolved secondary ion time-of-flight mass spectrometric imaging in order to obtain a molecular signature of the carboxylates. This study revealed among others the strong presence of lead and lead-carboxylates in the protrusion. The images below show the mass spectral lead (A) and lead-carboxylate (B) distributions as well as the infrared carboxylate band spectral distribution (C). This study confirmed the molecular identity of the transformation products found in the protrusion and has shed new light on their formation process.

These analytical techniques combined provided experimental evidence on the combined presence, distribution and identity of various (lead-)soaps in protrusions. The developed methodology has been used further for the rapid identification for similar protrusion phenomena in a variety of paintings, which is now a core study subject in the De Mayerne program.

Cross-section of a protrusion



SIMS images for Lead (A) and lead-carboxylate (B) distributions, and the infrared carboxylate band spectral distribution (C).

Key publications: (1) R. M. A. Heeren, J. J. Boon, P. Noble and J. Wadum (1999) "Integrating imaging FTIR and secondary ion mass spectrometry for the analysis of embedded paint cross-sections." *Preprints of the ICOM-CC 12th Triennial Meeting*, 29 August - 3 September, Lyon. Ed. by J. Bridgland, p.228-233; (2) P. Noble, J. Wadum, K. Groen, R. Heeren and K.J. van den Berg: "Aspects of 17th century binding medium : Inclusions in Rembrandt's Anatomy Lesson of Dr Nicolaes Tulp". In: *Art et Chimie, la Couleur : Actes du congrès*, edited by J. Goupy and J.-P. Mohen. (CNRS Éditions, Paris, 2000) p. 126-129.

Pigments

A selection of pigments has been studied as constituents of paints in which they were thought to fade, react with other materials or have a severe effect on the chemical and physical properties of the paint.

Indigo used in easel paintings

Margriet van Eikema Hommes and Ella Hendriks

Research carried out in the indigo project indicated that the colour-fastness of indigo paint can differ considerably, due to differences in the chemical and physical composition of the indigo pigment preparation. Furthermore, it appeared that in an oil or egg yolk medium, degradation of indigo's colouring component *indigotin* proceeds much faster than in gum, or glue media.

It also appeared that the way the indigo paint was applied, i.e. the painting technique, plays an important part in its preservation. Factors that accelerate the discoloration of indigo paint include the presence of a high percentage of lead-white, a thin indigo paint layer, low pigment to volume concentration, and a light paint layer underneath. An overview of the use of indigo as a pigment in oil painting and the factors influencing the fading of indigo paint is provided by Eikema Hommes (2002).

In the 17th century, painters used indigo almost exclusively for depicting folds in drapery, the tonal range of which required them to vary the composition of the paint.



Johannes Verspronck, Portrait of a girl dressed in blue (signed and dated 1641). Where the paint of the girls' dress has been shielded by the frame (lower edge) the blue colour has been preserved.

Therefore, one indigo passage can exhibit widely different degrees and types of discoloration. Consequently the modelling as conceived by the artist may have flattened or, on the contrary, may have become more pronounced resulting in the loss of specific material effects. In the indigo project the various optical changes caused by the degradation of indigo were investigated. This knowledge offers the possibility to theoretically deduce the paintings' original appearance and thus gain insight into the intentions of the painter.

That indigo fades more when mixed with higher proportions of lead-white implies that the lighter tones of the drapery fade faster than the darker passages. Therefore the middle and light tones of the young girl's dress painted by Verspronck, today approach the same tonal value. The contrasts between these passages and the deep shadows are now stronger than originally intended.

That indigo fades faster when applied thinly, implies that in 17th century paintings a more pronounced loss of indigo colour usually takes place in the middle tones, since according to 17th century systems for modelling these passages were often applied in one layer while the shadows and lighter tones were more thickly applied. Therefore, in Frans Hals' *Officers and Sergeants of the St. Adrian Civic*

Guard (c.1633) (*below, top*) the middle tones and reflections reveal to a greater extent the ochre coloured underlayer, and now appear more greenish blue than was originally intended. That indigo paint may fade faster when applied on a light layer and because faded indigo paint also appears better preserved on a dark layer due to the turbid medium effect helps the paint appear more blue, and affects the modelling of indigo passages. That in the 17th century shadow areas were usually underpainted with dark colours while these were absent in the lighter zones, implies that fading is faster in the lighter tones. The indigo tablecloth in Verspronck's 1641 *Regentesses* portrait, was painted directly on top of the light pink ground layers. Notably, the paint appears bluer in an area where the indigo paint overlaps the dark brown deadcolour of the dress of one of the Regentesses.



Detail: indigo blue sash of Captain Johan Schatter. For rendering the effect of warm reflections, Hals used the brown colour of the ground through thinly applied indigo layers.

That indigo fades faster when it is mixed with a high proportion of oil has often led to indigo paint areas with a spotty appearance. In Verspronck's portrait the indigo paint was used medium rich. Only around the contours of a hand lying on the tablecloth, the colour was filled in separately, with paint richer in pigment that was brushed out thinly. Originally, the colour of this paint may not have deviated strongly from the colour of the rest of the cloth. Today, since it has faded less severely than the rest of the cloth, the impasted paint stands out as a dark halo around the hand.



Right: Johannes Verspronck, Regentesses of the St. Elisabeth's Hospital (signed and dated 1641). overlap of indigo with black dress. Detail of pentimenti during varnish removal. Photo: Conservation studio, Frans Halsmuseum, Haarlem.



Left: Verspronck Regentesses detail dark halo of indigo around the hand. Detail of tablecloth before restoration.

Key publication: M. Van Eikema Hommes *Historical Painting Recipes and their Relevance for the Scientific Study of Paintings* (2002) PhD Dissertation.

Asphalt, mummy and Kassel earth pigments

Georgiana M. Languri

Complex organic pigments from geological sources such as asphalt, mummy, and Kassel earth, some almost 200 years old, have been identified and characterised at a molecular level. Asphalt and Kassel Earth were identified in oil paints after accelerated aging and tentatively in 19th century paintings. The influence of these pigments on the oil network especially in the initial drying period was also examined. These results were achieved using complementary DTMS and Py-(TMAH)-GC/MS techniques and multivariate analysis of data (PCA, DCA). On column GC/MS, ESI-FTMS, SEC, FTIR, light microscopy and SEM-EDX were used additionally.

Each of the pigments contains marker compounds that retain information about their origin in their chemical structure. Kassel earth is a subbituminous coalified material also called lignite that keeps many molecular characteristics from the original tropical wet depositional environment. Markers indicative for coalified leaves (fatty acids and long chain montan wax esters) and woody plant polymer remains (lignin derived alkyl substituted phenols, methoxyphenols, dihydroxybenzenes and vanillic acid), tropical-subtropical plants substances (e.g. cladanes, diterpenoids, triterpenoids) were found. Unique for Kassel earth pigments seems to be the montan waxes with molecular weights between 600 and 800 and the lignin derived compounds. Asphalt is a cross-linked petroleum derived product. Long chain aliphatic hydrocarbons, aromatic compounds such as alkyl substituted benzenes, naphthalenes, anthracenes and phenantrenes and sulphur containing aromatic compounds such as alkyl-benzothiophenes and alkyl-dibenzothiophenes are typical for asphalt. In addition carbon skeletons are found reminiscent of fossilised plant derived steroids and bacteria derived hopanoids [1]. Mummy pigment was embedded in a megilp-like substance made from mastic and oil. Branched chain fatty acids, a series of shorter and longer chain fatty acids (C6-C18) were clearly additional components assigned to be of possible mummy derivation. The complex pigment will be difficult to

discriminate from other components in oil painting.



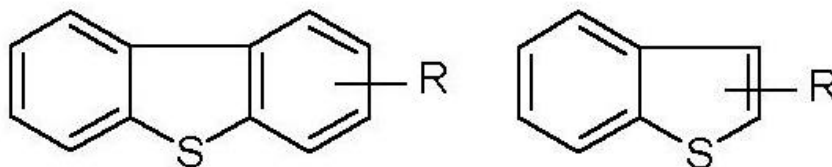
Alone in the world Joseph Israels, c.1880, Mesdag Museum, Den Haag. As early as 1911 it was suggested that the drying cracks and darkening in this painting may have been caused by the use of asphalt. Analysis detected Kassel Earth, but not asphalt.

The effect of these pigments on the composition of the oil paint was investigated for Kassel Earth (K) and Asphalt (A). The drying of A-K containing oil paint was monitored using the azelaic acid:oleic/elaidic acid ratio as indicator for retarding antioxidant activity (< 1) or accelerating oxidation activity (> 1). Asphalt was shown to prevent the drying of oil (ratio $\ll 1$), while the genuine Kassel earth

tested acted similarly as lead white used in the same oil paint matrix (ratio >1). Ivory (carbon) black pigment, a possible substitute of A-K pigments showed some antioxidant activity but less than asphalt.

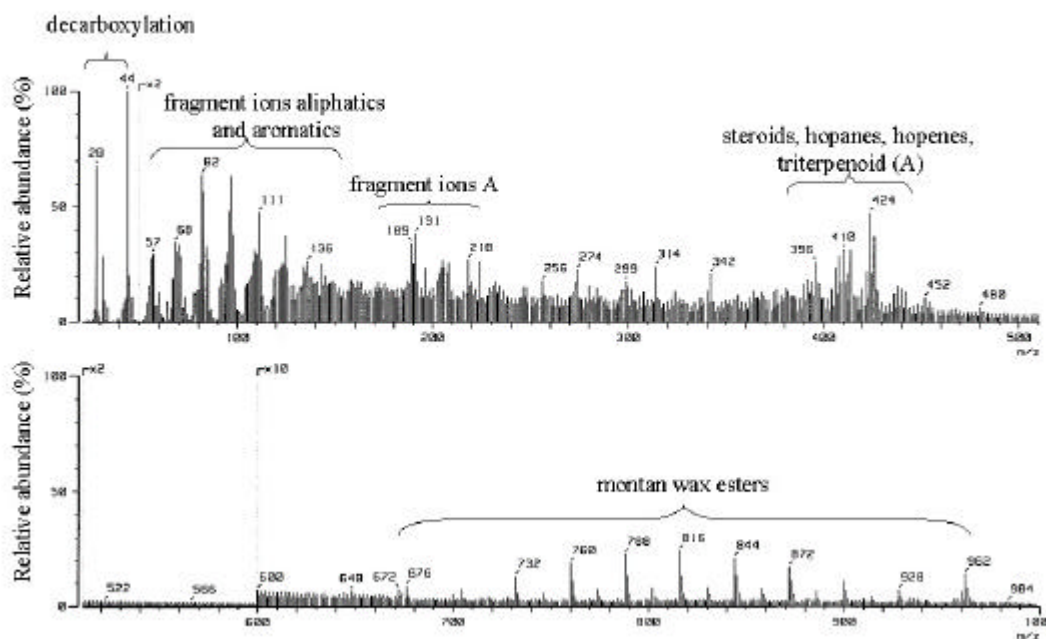
Markers of asphalt:

Left: alkyl-dibenzothiophenes,
Right: alkyl-benzothiophene



There are indications for the adulteration or substitution of these pigments. We have found natural asphalt, various Kassel earth-like pigments sometimes adulterated and asphalt containing mummy pigment in the 19th century Hafkenschied pigment collection. Other A-M-K historic pigments were genuine (Asphalt-Harrington Brothers, Asphalt-Roberson & Co, probably Mummy-Swillens collection) or not (Asphalt-Swillens, Asphalt-Winsor & Newton).

Key publications: (1) Languri, G.M., van der Horst, J., and Boon, J.J., ‘Characterisation of a “unique” asphalt sample from the early 19th century Hafkenschied painting materials collection by analytical pyrolysis MS and GC/MS’, *Journal of Analytical and Applied Pyrolysis*, 2002, 63, 171-196 (and erratum in vol. 64, 123-124). (2) Languri, G.M., and Boon, J.J., ‘Between myth and reality: on the “asphaltic” composition of the mummy pigment from the 19th century Hafkenschied Collection. A mass spectrometric approach’, *Studies in conservation* (submitted March 2003). (3) Languri, G.M., and Boon, J.J., ‘Mass spectrometry as a micro-analytical method to study Kassel earth (Vandyke brown) pigments in oil paint’, *7th International Conference of Non-destructive Testing and Microanalysis for the diagnostics and Conservation of the Cultural and Environmental Heritage*, Antwerp, Belgium, 2002.

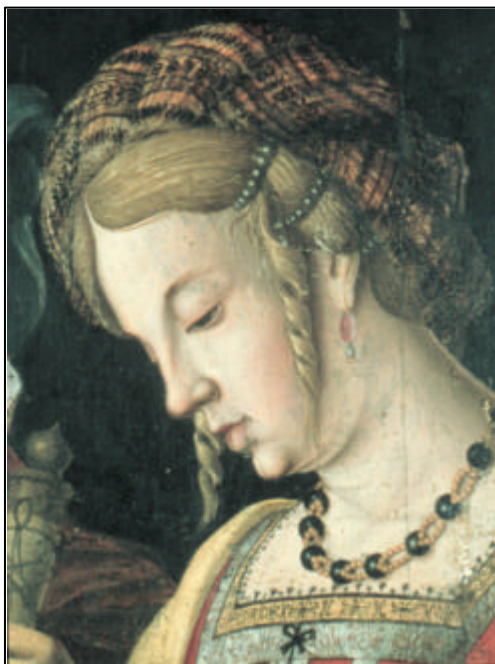


DTMS of the Hafkenschied Kassel earth pigment

Copper green glazes

Klaas Jan van den Berg and Margriet van Eikema Hommes

Copper-based green glazes were used by many 15th-17th century painters, for depicting foliage and drapery. The glazes were often applied over opaque underlayers of green paint that contained verdigris with lead white and/or lead tin yellow or azurite and lead tin yellow. In a number of cases, these colours changed from green to brown thus changing lush green into somber brown landscapes. A multidisciplinary study of a selection of paintings, historical recipes and paint reconstructions was undertaken to increase our knowledge of how copper greens were made and what the chemical reactions and resulting colour changes were that took place in drying and ageing of the paint.



Marco Marziale, Christ and the Woman taken in Adultery (c.1505) Detail of head-dress of the adulterous woman. The copper green glaze used for the patterns in the head-dress has turned brown.

It was established from documentary sources and experimental data that the still commonly used term 'copper resinate' is a misnomer. Copper resinate is a term referring to green paint or pigment in which copper resinate salts have been formed on purpose by mixing verdigris or another copper salt in warm solution of pine resins.

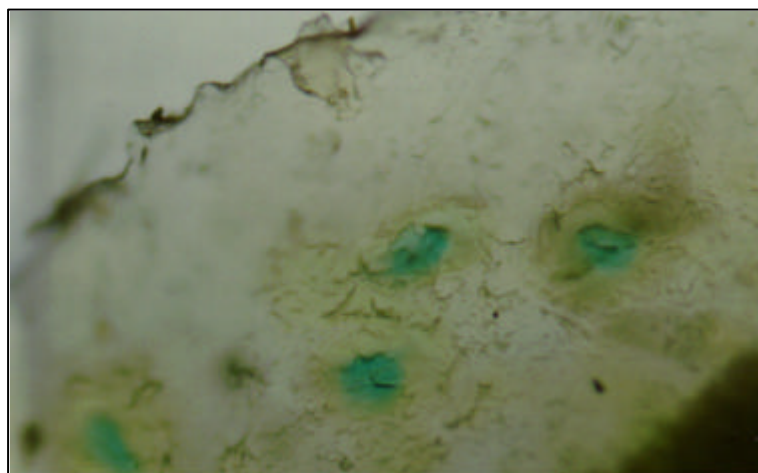
Extensive study of European 15th to 19th century treatises and workshop handbooks indicated that recipes for copper-based glazes for easel paintings only advise to mix verdigris in a medium of oil and/or resin *without heating*. The 'green varnishes' obtained in this way are still bluish but could be modified into every green colour with yellow lake. Heating verdigris in resin-containing media appears to have been common practice for producing lacquers for furniture, glass and metal foil *only*.

Paintings studies of some 20 paintings (Netherlandish, German, Italian, ca. 1450-1550) showed in several instances calcium on top of green glazes indicating the presence of a (faded) yellow lake. In addition, resin was found in only low amounts in the glazes, which mostly consisted of oil.

The low amounts of copper chlorides and copper sulfates found with X-ray diffraction studies in some glazes were interpreted as remnants and by-products of the production of verdigris, in which the main copper acetate part had dissolved.

A number of paint reconstructions with verdigris showed that the copper acetate particles can dissolve in the course of time after making and applying the paint (*below*) resulting in a more greenish paint than before. It was found that the shift from bluish green to green is not only due to the initial yellowing of the oil but is mainly due to the dissolution process described above.

The dissolution or loss of birefringence of the verdigris was fastest in verdigris oil paints. The higher the amount of resin in the medium, the slower the shift to green. This indicates that the copper in the verdigris is also involved in the chemical drying process. We speculate that in this reaction both ‘copper oleate’, an ionomer involving the oil network, and ‘copper resinate’ moieties are formed depending on the relative amount of the reacting organic species. Exploratory desorption mass spectrometry studies have shown that it is possible in principle to distinguish between copper oleate and copper resinate complexes. More fundamental chemical studies are necessary to establish the role of coordination chemistry in the composition of the green glazes. The transition from green to brown is still not fully understood on the molecular level.



Paint reconstruction: The copper acetate particles can dissolve in the course of time, resulting in a more greenish paint than before

Key publication: K.J. van den Berg, M.H. van Eikema Hommes, K.M. Groen, J.J. Boon and B.H. Berrie. “On copper green glazes in paintings”, in *Art et chimie, la couleur*. Actes du congrès, J. Goupy and J.-P. Mohen (Eds), 2000, CNRS Editions, Paris, p. 18-21.

Orpiment: deterioration of arsenic sulphide pigments

Arie Wallert

Orpiment and realgar are some of the most common yellow pigments. These pigments are natural mineral compounds based on arsenic and sulphur. Orpiment was already used in Egyptian wall-paintings dating from the 18th dynasty in Tell el Amarna. It is mentioned in Greek and Roman literary sources. The Hellenistic *Leyden papyrus* described its use for late Egyptian painting, as does the *Mappae Clavicula* for early mediaeval painting. The pigment has been described in various other mediaeval manuscripts dating from the 12th to the 15th centuries. Documentary evidence suggest that it was still in use in the 17th and 18th centuries in spite of the fact that there were some reasonably good alternatives, such as lead-tin yellow, Naples yellow, weld lakes, and the ochres.

From historical documents we learn that in painting three different arsenic yellow colours were recognised. Observations on many paintings tell us that all these pigments suffer from the same defects; they are poisonous, they do not dry very well in oil, and they change colour.

The aim of this study, done at the Shell (SRTA) laboratories, was primarily directed towards a better understanding of the mechanisms of decay, light induced and otherwise, within the As - S system. Structure, crystal characteristics, and particle morphology were studied with the use of XRD, and the scanning electron microscope equipped with an energy dispersive spectrometer (SEM-EDS) . These examinations helped to gain a better understanding of the crystallinity of the materials. The variability in cell dimensions of some specific types, as well as the amorphous characteristics (although contaminated with crystallites) of other types were especially notable.

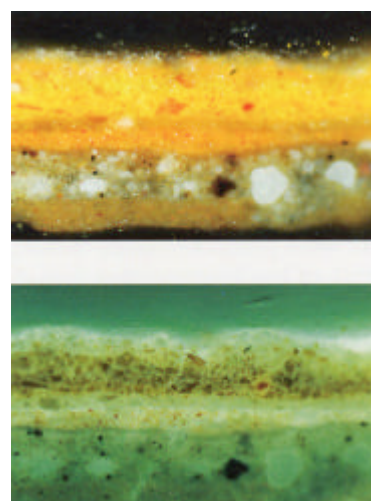


The problems of corrosion in orpiment or other substances of the As - S system is evident. The As-S system, generally described as the “realgar” group, consists of many chemically similar substances with different crystal lattices, sometimes belonging to different crystal systems. Polymorphism is a recurrent problem in the study of arsenic sulphides.

Left: Jan Davidsz de Heem Festoon of Fruits and Flowers (1606-1683/84), Rijksmuseum Amsterdam Detail, showing discoloured lemon.

In the natural mineral specimen many of these occur concurrently, and often together with intermediate phases, making their identification extremely difficult. There is orpiment (As_2S_3), dimorphite I ($\alpha\text{-As}_4\text{S}_3$), dimorphite II $\beta\text{-As}_4\text{S}_3$, alacrinite (As_8S_9), uzonite (As_4S_5), duranusite (As_4S), pararealgar ($\alpha\text{-AsS}$), realgar ($\beta\text{-AsS}$), ‘phase κ ’, and so called realgar “high” ($\beta\text{-AsS}$). All of these components may oxidise to the monoclinic arsenolite (As_2O_3) or to the cubic claudelite (As_2O_3). Conversion of these minerals is not limited to oxidative corrosion. Realgar especially also seems to have a tendency for light induced phase inversion to yellow and white polymorphs.

Right: Cross-section of highlight in the lemon, 500x, normal and ultraviolet light. The broken surface of the paint layer of orpiment shows the white ‘clouds’ of arsenic trioxide.



In paint mixtures on easel paintings, the conversion of the mineral phases of the pigment resulted in either a brownish-red discoloration, or in ‘invisibility’ due to the closeness of refractive indices of the newly formed product to those of the surrounding medium.

Understanding of these mechanisms proved helpful in the interpretation and conservation of paintings by Tintoretto, Titian, Maarten van Heemskerck, Jan Davidsz. De Heem and others.

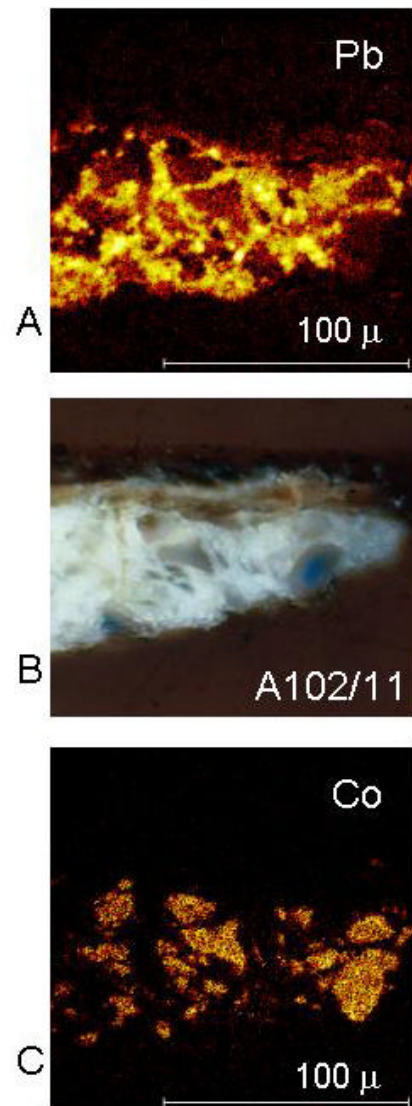
Key publication: A. Wallert (1999) “Methods and materials of still-life painting in the seventeenth century” in: *Still lifes: Techniques and style, the examination of paintings from the Rijksmuseum*, ed. A. Wallert. Zwolle/Amsterdam:Waanders / Rijksmuseum, pp.7-24.

Smalt

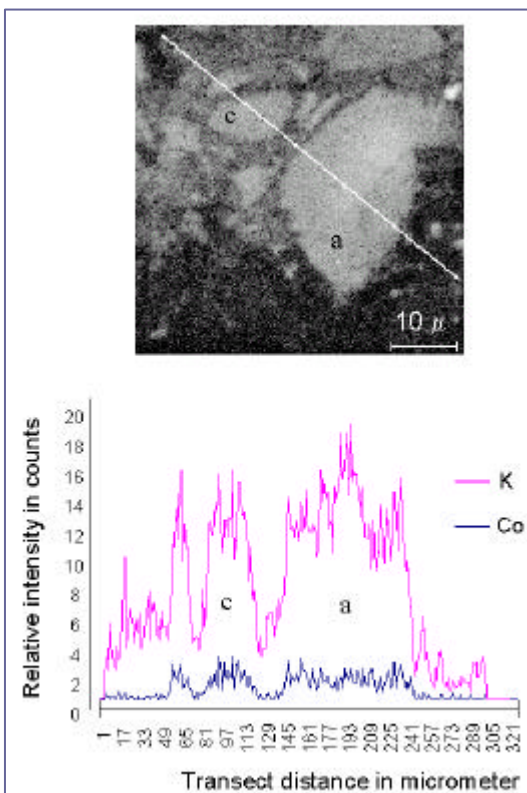
Jaap Boon, Muriel Geldof and Katrien Keune

Smalt, the cheap blue glass substitute for ultramarine and azurite that became popular after 1650, is unstable when it is made into smalt oil paint. In the beginning, sources point out that there were complaints by painters saying it affected the flow and drying of the paint. About a century later colour changes become more apparent. The discoloration of smalt leads to an increased transparency of the paint layer often showing colours that are used deeper in the painting. This can lead to grey or brownish tones in skies where blues were present before. Overall the effect will be that the atmosphere in the painting will become much more sombre. Studies were performed on paint cross-sections of 16th century paintings by Dirck Barendsz (1534-1592) and Pieter Aertsen (1509-1575). Reflection light microscopy demonstrated the presence of partially discoloured smalt particles with a blue core remaining. Semi-quantitative SEM-EDX analysis revealed that the potassium abundance was relatively low in all discoloured smalts, while cobalt remained at an approximately constant level. Imaging SIMS demonstrated that cobalt has a uniform distribution in the glass particles.

Right: SIMS maps of Smalt in lead-rich medium: The SIMS lead map shows presence of medium; the lead-free dark areas indicate the presence of smalt particles. B: Visible image of cross-section showing smalt particles. (Note the preserved blue core of one smalt particle) C: The SIMS cobalt map shows the presence of the smalt particles.



Potassium however shows relatively high levels in the oil paint matrix around the discoloured glass particles and an uneven distribution in smalt particles with a blue core. These blue cores always have a higher K level compared to the discoloured rim of the particle. (left)



The blue cores have a higher K level compared to the discoloured rim of the particles. The K level around the particles is high due to migration out of the glass matrix.

The loss of potassium from smalt in oil paint is interpreted as a leaching process, which lowers the basicity of the glass below a critical level for colour maintenance. The colour change of the smalt is thus an indicator of a change in alkalinity inside the glass. The alkalinity of glass is tested nowadays by the blue colour when cobalt is used as probing tracer. A critical level in 16th century potash glass appears to be near a K:Co of about 1:1 on the basis of semi-quantitative SEM-EDX data. Imaging SIMS clearly shows higher levels of potassium in the oil paint matrix

around the smalt particles. The migrated K is thought to be accommodated on the many fatty acid groups of the mature oil network ionomer in the aged oil paint. We speculate that this will make smalt oil paint layers more hydrophilic and water sensitive. The observation that the varnish is often affected above smalt oil paint layers might be related to this as well. Ca was observed in a number of blue remaining smalts, which suggests a role in preservation of the blue glass or retardation of the leaching process. The differences in potassium levels in the remaining blue part of the smalt particles point to variability in the quality of the base potash glass in the 16th century. Analysis of partially discoloured smalt from two panels of a triptych by Dirck Barendsz demonstrated that he had access to two chemically different smalts. Further collaborative studies using microXANES have shown that cobalt in the coloured and discoloured parts of the smalt particles has the same oxidation state. Our study demonstrates that earlier hypotheses like loss of cobalt from the glass and a change in oxidation state can be discarded. Loss of potassium from the glass matrix affects the coordination chemistry of the cobalt, which changes to from tetra to octahedral symmetry. Under these conditions the colour changes from blue to light pinkish.

Key publication: J.J. Boon, K. Keune, J. van der Weerd, M. Geldof and J.R.J. van Asperen de Boer: "Imaging microspectroscopic, secondary ion mass spectrometric and electron microscopic studies on discoloured and partially discoloured smalt in crosssections of 16th century paintings." *Chimia* 55 (2001) 952-960.

Organic constituents and media

Molecular studies not only revealed the many mechanisms of chemical change in paintings but also demonstrated that the organic and inorganic worlds are intimately linked.

Fresh and aged triterpenoid varnishes

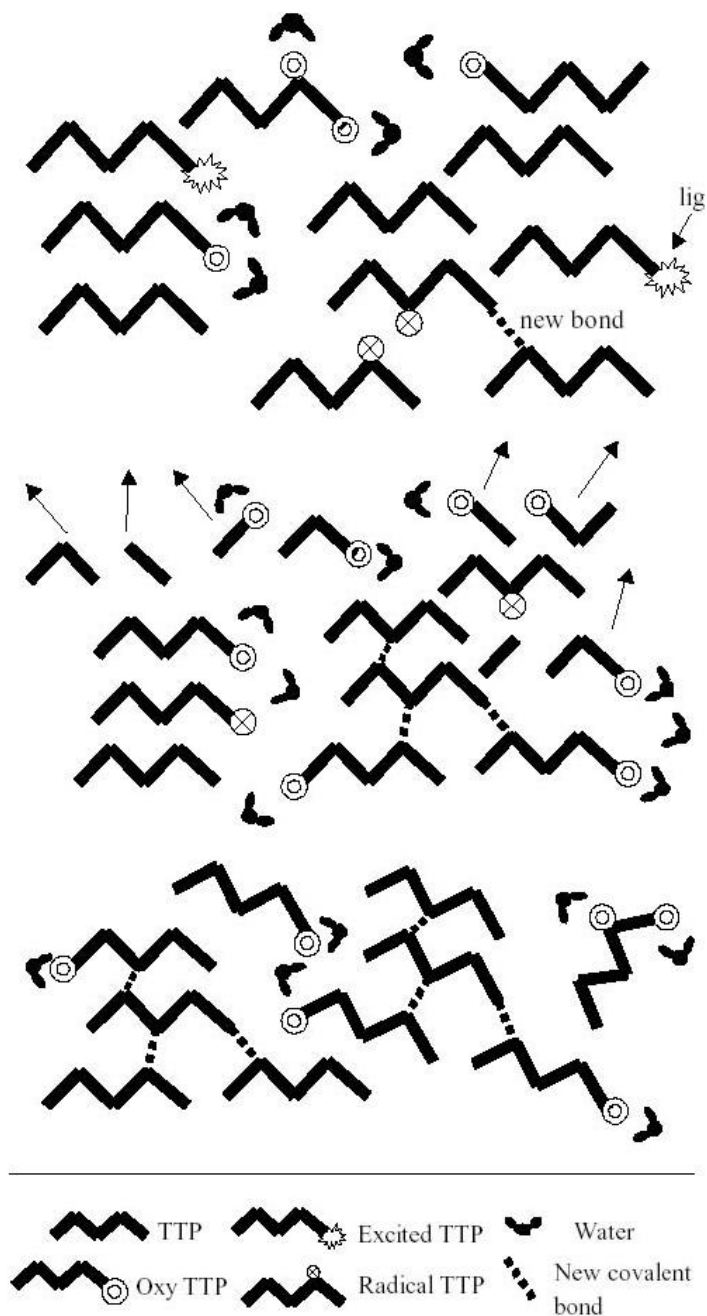
Gisela A. van der Doelen

The removal of degraded varnishes constitutes one of the major activities of painting conservators. Analysis of over a hundred varnish samples from paintings under restoration showed that more than 80% was of triterpenoid origin (predominantly dammar and some mastic). Prompted by how little was known about the chemistry involved, we have been studying the ageing process of these resins.

Analysis of both fresh and aged triterpenoid varnishes led to the formulation of a descriptive model on the molecular ageing of a triterpenoid varnish. The composition of the varnish changes radically over time with the appearance of a diverse range of triterpene oxidation products and higher molecular weight compounds. A key question is what happens to the higher molecular weight fractions during solvent cleaning. Tests proved that they are less soluble in solvents commonly used for cleaning. This finding may have implications for the practice of partial cleaning. Advocates of partial cleaning suggest that varnish layers can be removed independently from each other leaving a last “protecting” layer behind. In our view some degree of separation of varnish constituents may take place. It is possible that microscopic layers of cross-linked varnish remains are concentrated on the paint surface. Our work on solvent aged varnishes indicates that these higher molecular weight fractions are important in the yellowing of the triterpenoid varnishes.



Artificial or accelerated ageing tests are often used in the field of conservation science to study the changes in physical properties, e.g. yellowing and solubility, of painting materials. Unfortunately, these investigations never consider the changes on the molecular level. As physical changes are usually a consequence of chemical changes it is important to investigate whether the chemical changes induced by artificial ageing are the same as those that occur “naturally” on paintings.



Schematic model showing different stages of ageing of triterpenoid varnishes.

Upper: Oxidised varnish: Oxygen uptake. Formation of excited and oxidised triterpenoids.

Centre: Oxidised varnish: loss of C-skeletons; emission of volatile oxidation products; further cross-linking and O-insertion.

Lower: Highly degraded varnish: H and O loss. Polar and apolar regions in polymer fractions.

Dammar and mastic reconstructions were aged with commonly used ageing regimes (using a xenon-arc device and fluorescent tube devices) and analysed. The UV proportion of the light used for ageing was found to induce the production of specific molecular species, which were not observed at all in the many samples from real paintings analysed. These species all contain an additional acid group, which is likely to effect the solubility behaviour and the mechanical properties of the varnish film. Clearly, artificial ageing did not simulate natural ageing in

this case. When the UV component was filtered by means of filters the same oxidation products were formed as found on paintings. This study clearly demonstrates the importance of research on the molecular level.

A new method was introduced to study the ageing process of materials: ageing in solution. Ageing of samples in solution has several advantages over the ageing in the solid state: faster (no kinetic limitations compared to the relatively low diffusion rates encountered in varnish films), possibility of ageing single pure triterpenoid species (no necessity to form a film), higher control over the production of reactive species, and higher homogeneity of an aged sample (ageing is not predominantly a surface phenomenon as in the solid state).

Dissolution in the solvents dichloromethane and acetone and subsequent light exposure induced similar molecular changes in triterpenoid substances as found on paintings during “natural ageing”. This technique can therefore be successfully used to study the oxidation processes in painting materials and might be useful for industrial purposes.

Key publications: (1) Gisela A. van der Doelen. *Molecular studies of fresh and aged triterpenoid varnishes* (1999). PhD Dissertation. University of Amsterdam, Amsterdam, 25 March 1999 (2) G. A. van der Doelen, K. J. van den Berg, and J. J. Boon (1998). “Comparative chromatographic and mass spectrometric studies of triterpenoid varnishes: Fresh material and aged samples from paintings” *Studies in Conservation*, 1998, p.249-264 (3) G. A. van der Doelen and J. J. Boon (2000). “Artificial ageing of varnish triterpenoids in solution” *Journal of Photochemistry and Photobiology A: Chemistry*, Vol. 134, p.45-57.

Diterpenoid resins in paintings

Klaas Jan van den Berg

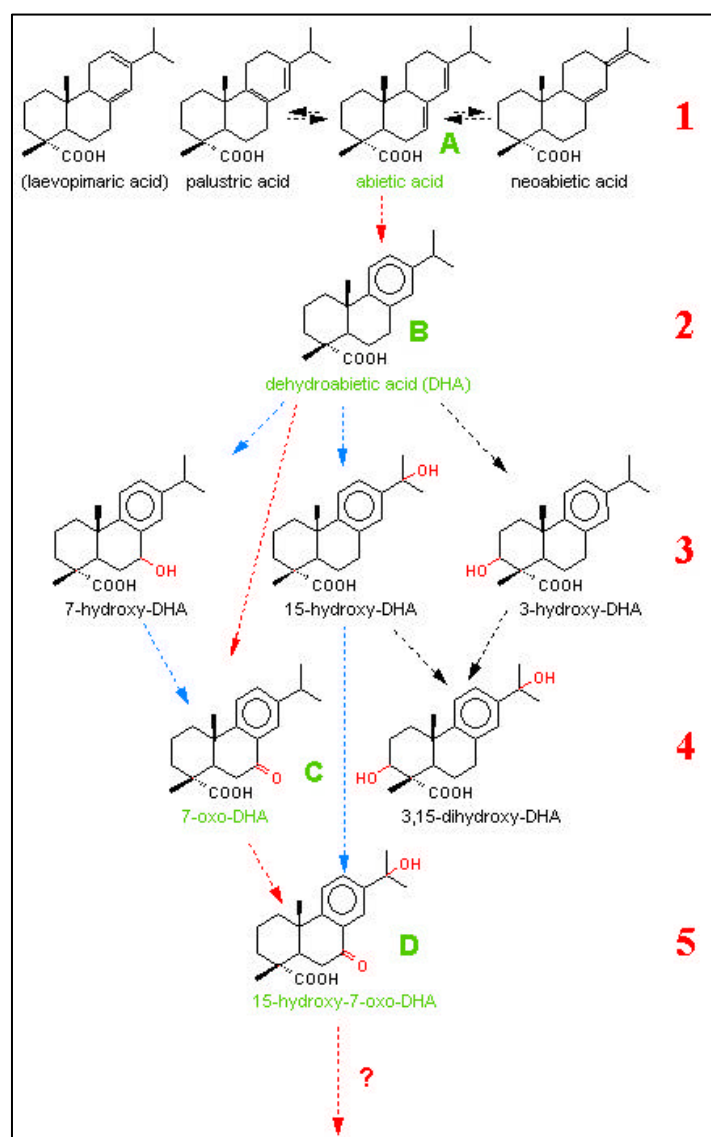
Diterpenoid resins are exudates from a large variety of trees throughout the world. Most of these resins originate from conifers (*Coniferae*) and tropical flowering plants from the *Leguminosae* family. Artists have used the resins extensively in and on works of painted art, as varnish, as additive to paints (e.g. glazes, lacquers), as pigment (copper green glazes) and mordant (adhesive for e.g. gold leaf). In addition, resins may have been introduced by conservators, e.g. as varnish or for regeneration purposes or as wax/resin lining paste for repairing and strengthening of the support.

In this project we focused on the best-known and most often used diterpenoid resins which belong to the conifer subfamily *Pinaceae*. This family comprises e.g. pine (*Pinus*), a tree which produces non-amber-forming balsams as a result of the large amounts of diterpenoid acids, particularly abietic acid and its isomers. In the course of ageing chemical changes take place, which is reflected in the physical properties of the resins.

Especially pine resins, turpentine and colophony have always been very cheap. Indeed, we found (often highly oxidised) abietic acids in a number of cases, as cheap adulteration of more expensive balsams, such as copaiba balsams. In addition, we found that old turpentines, distillates from pine balsam, also often contain abietic acids. As these turpentines were often used as diluent or solvent for other resins such as gum mastic, as well as oil paints, finding small amounts of these compounds may be explained by the use of these solvents.

We studied the molecular changes of the resins during ageing with a number of mass spectrometric techniques, including DTMS, several GCMS techniques and LCMS. We confirmed earlier work of Mills and White, who found that the abietic acids oxidise to a number of different species, but also discovered a new, highly oxidised compound, i.e. 15-hydroxy-7-oxo-dehydroabietic acid. This compound, which may be present in high amounts in some aged varnishes, wax-resin linings etc. is probably soluble in water (although this has never been checked!), and explains very well why sometimes proposed ‘regeneration’ of wax/resin adhesive on a painting is bound to fail; by heating the mixture the wax will melt, but no longer dissolves the now more polar oxidised resin. With the discovery of this compound a scheme could

be drawn which shows five levels of oxidation, the newly discovered species being the most oxidised species 'D'. To date, we have not found evidence for further



oxidation although further oxidation would compromise the carbon skeleton thus changing the chemical properties possibly beyond recognition

With the different levels of oxidation drawn in the scheme, we can now derive information on the degree of oxidation of the abietic acids, and as a result make inferences about the strength of the oxidative environment of the medium in which the resin is present. We found that, apart from time and oxygen, light, the method of dispersion of the resin (in solution oxidation is much faster), and certain metals such as copper in some pigments can accelerate the oxidation.

Key publication: K. J. van den Berg, J. J. Boon, I. Pastorova, and L. F.M. Spetter (2000). "Mass spectrometric methodology for the analysis of highly oxidised diterpenoid acids in Old Master paintings" *Journal of Mass Spectrometry*, Vol. 35, p.512-533

Resin macromolecules

Klaas Jan van den Berg and Jerre van der Horst

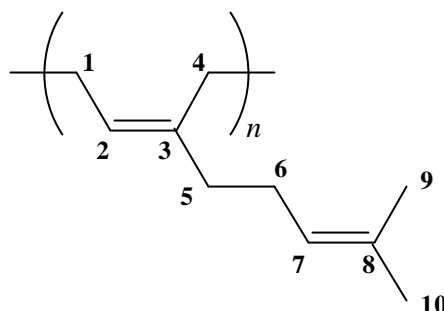
Macromolecules play an important role in the physical properties of resins. This fraction, for example, gives a varnish good working properties. Moreover, in addition to the oxidative processes (also mentioned in the chapters on diterpenoid molecules and triterpenoid varnishes), condensation of lower molecular weight molecules to polymers of high molecular weight plays an important role in the ageing process of resins. The project began by obtaining more information on the chemistry and molecular changes of polymer fractions in resins and yielded two significant results: a) the discovery of the polymer fraction of gum mastic, and b) a new methodology to trace copal and copal-related resins in small paint samples.

a) The discovery of *cis*-1,4-poly-**b**-myrcene

The polymer of gum mastic, a resin harvested from *Pistacia lentiscus* L., was the only important resin from which the polymer was still unknown. In MOLART we set out to study this polymer by isolating the polymer in a series of steps, using size exclusion chromatography to assess the purity of the polymer. In a series of experiments including DTMS and Py-GC-MS we found that the polymer predominantly contained C₁₀H₁₆ units, probably from a monoterpene. ¹³C-NMR as well as ¹H-NMR analyses finally characterised the polymer as *cis*-1,4-poly- β -myrcene. This structure, which is a double isoprene from which one part is involved in the polymerisation, is also the first reported naturally occurring polymer of a monoterpene.

The structure of cis-1,4-poly-b-myrcene

β -myrcene is the one monoterpene that is naturally present in the balsam that exudates from the tree, which can polymerise under sunlight. This assumption has since been confirmed by a group of Swiss researchers (P.Dietemann PhD thesis forthcoming) who found that mastic balsam kept and dried in the



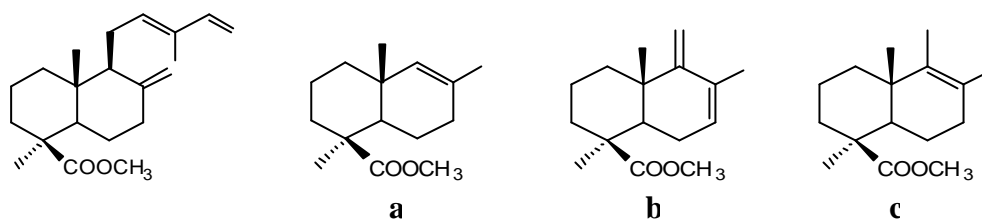
dark does not form a polymer fraction. The polymer fraction was found to contain some oxygen-containing molecules that may have been formed from low molecular weight impurities, as well as by oxidation of the polymer. The polymer proved to be relatively unstable after isolation when no precautions to avoid degradation were taken which was indicated by the rapid decrease of solubility of the polymer after isolation. This suggests that, due to the large number of unsaturations, oxidation and/or cross-linking takes place rapidly. This rapid degradation explains why poly- β -myrcene is not identified in, for example, aged varnish samples from paintings.

b) New methodology to trace copal and copal-related resins in small paint samples.

Copal and related resins were extensively used in the 19th Century, in resin-oil varnishes, as well as in resin-oil paints. Yet only very few times has analytical evidence been found for the actual presence of these resins. During the MOLART project, we realised that one way to overcome this gap between contemporary descriptions and analyses might lie in the analysis of the polymer fraction, which is amply present in copal resins and related resins such as sandarac and ambers.

A new approach using a two step py-GCMS with *on-line* derivatisation with tetramethylammonium hydroxide (TMAH) was applied. In this procedure, in the first step performed at around 220 °C, derivatisation of the sample takes place and analysis of the monomer fraction. In the second step, pyrolysis and analysis of the residue takes place.

The analysis of the copals in this procedure yielded large amounts of communic acids (or ozic acid in the case of African copals) in the second step, as well as dicyclic fragments.



Methyl communate and its fragment marker molecules

Analyses of copal/oil varnishes indicated that characteristic copal components had disappeared to a large extent, both in the manufacturing process (which includes strong heating of the resin) and during ageing. However, some characteristic marker compounds could still be found. At a later stage in the project, actual analytical evidence was indeed found in a number of paint samples in paintings by the pre-Raphaelite brotherhood, as well as a number of 19th Century varnishes on other paintings.

Key publications: (1) van den Berg, K.J., van der Horst, J. Boon, J.J. and Sudmeijer, O. *Tetrahedron Letters*, 39 (1998) 2645-2648. (2) van den Berg KJ, van der Horst J and Boon JJ, in Preprints *ICOM Committee for Conservation 12th Triennial Meeting, Lyon, France, 29 August-3 September 1999*, pp 855-861. James & James, London (1999).

Mobile and stationary phase in traditional aged oil paint

Jorrit D.J. van den Berg

Most of the paintings from the 15th to early 20th century produced in Northern Europe are made with drying oils (linseed, poppyseed and walnut) as the binding medium. The ageing of this medium after application leads to chemical changes, which strongly influence the physical behaviour of the paint film. Examples are blooming (efflorescence of "mobile" material), shrinkage and crack formation and chalking of the paint film. Due to the autoxidation processes of the unsaturated fatty acids of these oils, the initial triacylglycerols cross-link ("chemically dry") and form larger molecular complexes, a so-called stationary phase. Oxidative degradation processes also play an important role in the formation of volatile matter and the formation of (oxidised) fatty acids and diacids. In this stage we envisage the formation of an elastomeric polymer-like substance with associated oxidised non-cross-linked fractions that may act as plasticising materials. It is our assumption that chemical drying in oils and paints made with oils follow similar chemical pathways.

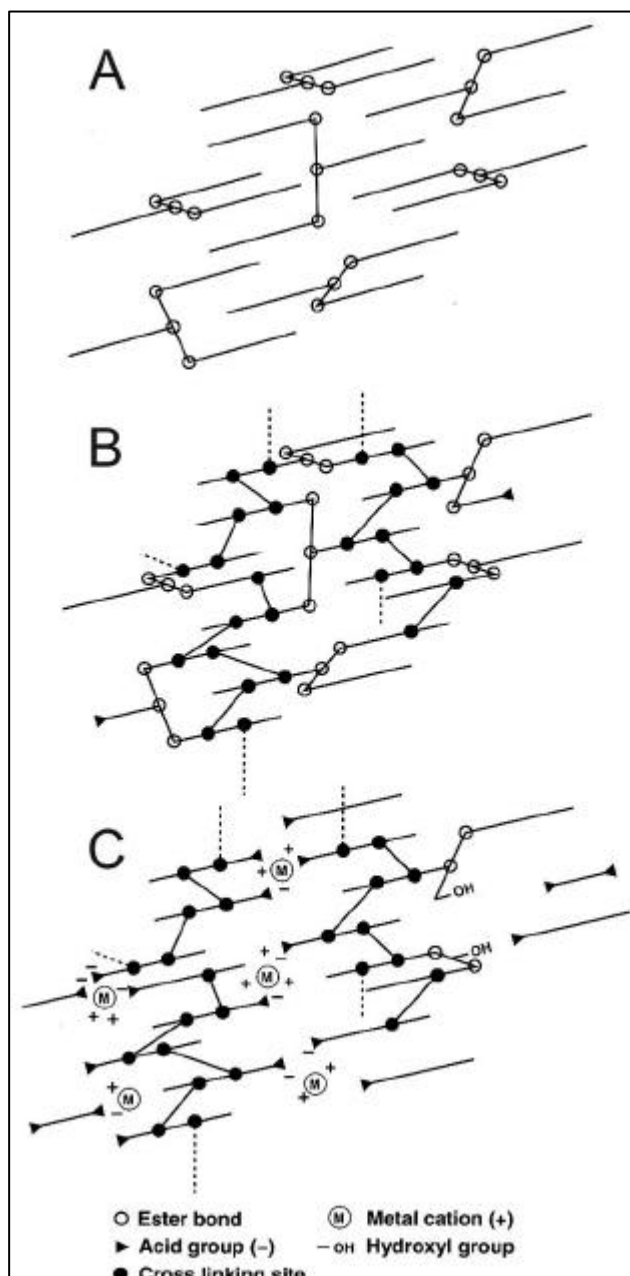
Hydrolysis of the initial ester bonds of the triacylglycerols contributes to the formation of free fatty acids. During ageing, most of the initial ester bonds are hydrolysed and part of the free fatty acids will form metal carboxylates by reaction with the metal containing pigments. Apart from the already free non-cross-linking compounds, these newly released free fatty acids are potentially "mobile", which implies that they can be moved or removed by solvents during cleaning procedures. They can also become redistributed by diffusion processes or lost by evaporation from the painting. Recall the formation of fatty acid films on glass paned paintings.

Drying oil polymer networks may be found in the various layers of a painting, e.g. the ground, the paint layers themselves, intermediate/isolating layers and even the varnish. They consequently determine most of the organic chemistry of the painting. The most important change discovered during the MOLART project is the transformation from an apolar polyester-type polymer to a polymer system which is kept together by ionic forces (metal-carboxylate interaction) to form an ionomeric structure. These changes depend on many factors often not known in detail, such as light, relative humidity, nature of the (transition metal), availability of oxygen, and possibly the restoration history. A schematic model of the transition of fresh oil paint to an aged oil paint as developed within MOLART is shown *right*.

Within the course of the MOLART project several micro-analytical techniques have been investigated and further developed, which were aimed at the identification of both the mobile and the stationary phase of oil paints. These techniques proved very useful when the

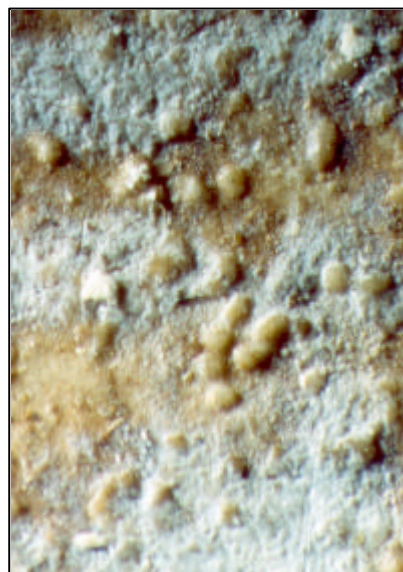
question comes into play why certain phenomena are observed on works of art, e.g. bloom formation, efflorescence or protrusion formation. To get a better insight into these undesirable physical and chemical changes, the molecular chemistry of the oil paint has to be investigated in detail. At present, the identification of the constituents of mobile phase does not pose a problem. The structure of the ionomeric networks, however, is still a problem that is much harder to tackle but is directly relevant for elucidation of the physical behaviour of the paint.

A good example of the use of analytical techniques are my studies directed towards the origin of bloom formation. The formation of patches of whitish, crystalline material observed on two unfinished canvasses formerly in the possession of the American painter F.E. Church (1826-1900) and two works of modern art by Frank Stella (1936-) were investigated. Two types of materials are observed. Bloom on the surface of the canvasses consisted of apolar long chain C16 and C18 saturated fatty acids and their lead soaps, independent of the type of paint and pigment present



at the surface. This material is formed in an oil rich ground layer and fatty acids have migrated to an underpaint pigmented with lead white. Formation of concentrated regions of lead soaps, so-called protrusions, are also observed in this layer.

A number of panels painted by F. Stella show disturbing white patches. A red and blue paint, consisting of alizarin crimson and cobalt blue respectively, are sensitive to bloom formation. The crystalline bloom was identified as free C16 and C18 fatty acids, derived from the oil medium. The exact mechanism of their formation and migration within the paint is not entirely clear. A more detailed study performed on one of the paints revealed a degree of hydrolysis of the paint of 40%. The absence of reactive pigments to trap free fatty acids, the high ratio of oil to pigment and a non-absorbing (magnesium metal) support lead to an excess of potentially mobile fatty acids that can migrate to the surface of the oil paint.



Right: Bloom and efflorescent crusts found on preprimed canvas (c.1865) formerly used by F.E. Church



Left: Bloom on a panel of F. Stella. Note the white streaks of bloom on the crimson red following the brush strokes.

Key publication: MOLART Report 6: Jorrit van den Berg *Analytical chemical studies on traditional linseed oil paints.* (2002) PhD Dissertation, University of Amsterdam.

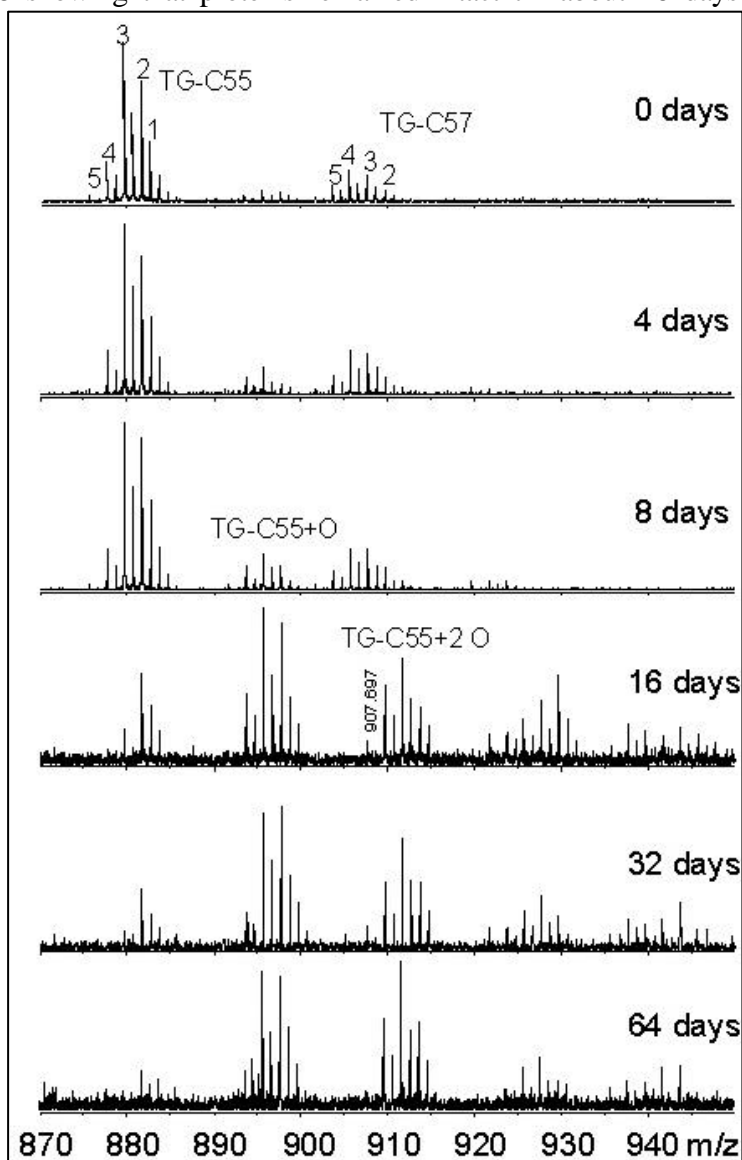
Lipids in egg tempera paint films

Oscar van den Brink and Jaap Boon

Knowledge of the chemical composition of aging egg tempera paint films is a spin-off from the studies of the chemistry of egg tempera paint dosimeters that were developed to monitor the museum environment. The egg tempera paint dosimeter films consisted of egg yolk, the egg whites without the high molecular weight mucopolysaccharides and mastic to stabilise the film. Lipids were studied by various mass spectrometric techniques (GCMS, DTMS, DTMSMS, MALDI-TOFMS, MALDI-FTMS and Electrospray FTMS). Non-lipid components were analysed by DTMS and MALDI-TOFMS showing that proteins remained intact till about 16 days of accelerated aging. Prolonged light aging introduced more acid and sulphonic acids groups in the proteinaceous material.

Cholesterol, that constitutes about 5% of the lipid fraction, rapidly oxidises under light exposure to 3-hydroxycholes-5-en-7-one, 5,6-epoxycholestan-3-ol, several cholestan-3-ol isomers and other oxidation products. The integrity of the cholesterol skeleton is thus severely compromised at an early stage leaving doubt about the use of cholesterol as a marker for centuries old egg tempera paint films.

The acylglycerols lipids in egg yolk consist of phospholipids (mainly phosphatidylcholine with 42 and 44 carbons) and triglycerides (C51:0 to C57:6). Fourier transform MS studies made it possible to observe the incorporation of more than one oxygen atom into these lipids under light ageing conditions and conditions of NO_x exposure.



Above: Incorporation of oxygen in triacylglycerols of the egg fats as a function of age is demonstrated by the increments of 16 in MALDI-FTMS spectra of light-aged egg-only tempera samples.

Interestingly, dark thermal aging for 3 weeks did not lead to appreciable oxygenation. Triglycerides rapidly incorporated oxygen in the first two weeks of light exposure leading to a strong increase in mono-, di- and tri-oxygenated compounds. Apart from oxygenation of the aliphatic chain, chain shortening to C9 diacids could be observed on remaining oxygenated triglycerides. The high resolution mass measurements of the FTMS were extremely useful in the unravelling of the complicated mixture of compounds that resulted from light aging in the presence of various types of mineral pigments (lead white, azurite, vermilion). There is a general decrease in the relative amount of phospholipids and triglycerides with aging time. Apart from oxidation as depletion mechanism, cross linking of lipid fractions could be demonstrated by size exclusion chromatography of the lipid extract. Aging for 16 and 64 days led to molecular weight fractions of about 10 000 Dalton (calibrated against polystyrene). Interestingly, the MS data of the proteinaceous extraction residues showed that acylglyceryl lipids were incorporated in this fraction as well.

From this work a picture emerges on the nature of the egg tempera binding medium. The proteins form a prepolymeric network with the very stable polyamide backbone although denaturation and new formation of three dimensional networks can not be precluded. This network reacts with lipids forming hydrophilic and hydrophobic domains. It is possible that the cross-linked lipid fraction interacts and binds with van der Waals forces to some of these domains. Gradually, as aging progresses, lipids become more polar and proteinaceous materials become enriched in acidic groups. Both phenomena lead to a stronger interaction with metals from the pigments. It is unclear whether the glycerolester bonds in egg tempera paint films survive in the long term. Hydrolytic processes will probably affect these bonds but will leave the amide backbone unaffected. Compared to oil paint we therefore expect some ionic character mainly involving lipid moieties but a large part of the plasticity of the film might be preserved because the amide bonds resist hydrolysis. Overall a stronger binding is expected to the mineral surfaces because of the increasing polarity of the organic fractions creating ample functional groups for coordination chemical bonds. Indeed, the extraction of egg tempera macromolecular fractions has been very difficult in our hands and requires conditions that break these kinds of chemical bonds.

Key publication: MOLART Report 4: Oscar F. van den Brink *Molecular changes in egg tempera paint dosimeters as tools to monitor the museum environment (2001)* University of Amsterdam.

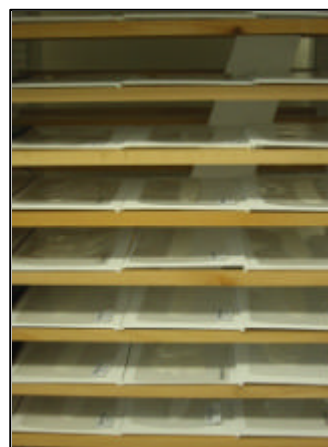
Historical reconstructions of artists' oil paint, an investigation of oil processing methods and the use of medium-modifiers

Leslie Carlyle (MOLART Fellow)

The aim of this project was to evaluate the long-term effect on oil paint of lead-based driers in linseed oil and the use of lead-based mediums which were added to the paint. Recipes for oil clarification and for driers were collected from early 19th century British sources to ensure that concentrations of lead compounds used in the project reflected actual use in the past. Organically grown linseeds were pressed to extract the oil, so that the same seed lot and conditions of extraction were maintained for all the sample paints.

There were several surprising outcomes in the initial stages of this project. It was discovered that the method of oil processing and the use of different lead compounds had a profound effect on the handling qualities of the paint (rheological properties). Another unexpected outcome was the effect of these variables on the colour of the lead white paint. For example, lead acetate, a common ingredient in British nineteenth century oil paint, caused significant yellow colour development, whereas litharge (lead II oxide), another common ingredient, shifted the colour slightly towards red. These colour developments were measured with a Minolta spectrophotometer, and have been published in the thirteenth triennial meeting of ICOM-CC 2002, along with an evaluation of the light/dark response of these paints.

The paint samples continue to be useful; naturally and artificially aged materials from this project are now available as reference samples and for the investigation of the development of lead carboxylates, as well as the influence of the oil and its processing on the lead white pigment itself. Work is likely to continue on these samples for many years to come, and eventually they may provide answers to the original question: how are paint films affected by a variety of lead compounds used in oil processing and medium formulations.



Key publication: *L. Carlyle, N. Binnie and Elzbieta Kaminska* "The Yellowing/bleaching of oil paintings and oil paint samples, including the effect of oil processing, driers and mediums on the colour of lead white paint" in *ICOM, Committee for Conservation 13th Triennial Meeting, Rio de Janeiro, 22-28 September 2002*, p.328-337

New physical analytical techniques

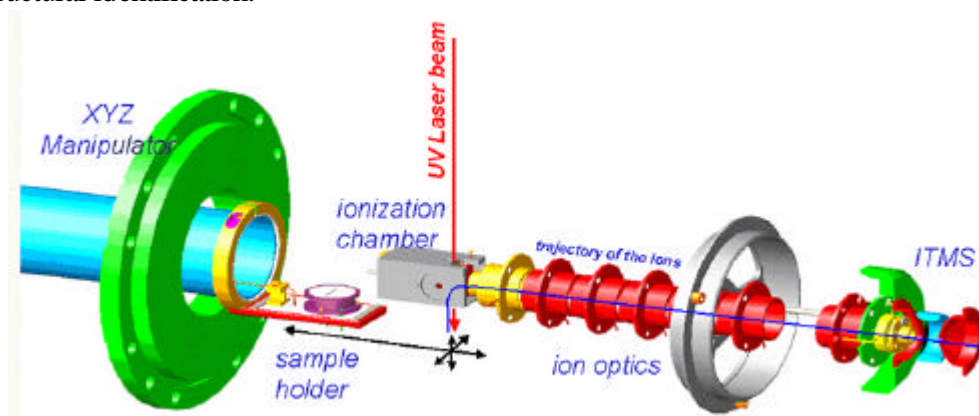
Several new techniques were explored and tested for their potential to answer questions on the spatial and depth distribution of organic and inorganic paint components. This was high risk research with an unknown final outcome.

Exploratory research on artist's pigments with laser desorption mass spectrometry

Nicolas Wyplosz

This project examines the use of laser desorption mass spectrometry (LDMS), as a novel technique of analysis in the investigation of natural and synthetic organic pigments encountered in easel paintings. Two mass analysers were utilised for LDMS, namely an Ion Trap Mass Spectrometer (ITMS) and a Time-of-Flight Mass Spectrometer (TOF-MS). The key advantage of using LDMS for the study of paint materials is the possibility to perform spatially resolved surface analysis of minute amounts of materials with a lateral resolution down to 10 micrometers. LDMS was applied for the first time to the investigation of cross-sections.

Two ionisation techniques were examined for the LDMS analysis of flavonoid and anthraquinone reference materials, namely LDI and MALDI. LDI is the direct laser desorption and ionisation of the sample surface, whereas MALDI involves the added use of a strong chromophore (a matrix) to assist the LDI process. These experiments revealed that substantial amounts of ions are obtained with low laser power densities in LDI. This particular feature makes it possible to perform multiple-stage analysis (MS^n) with the ITMS set-up. This was proved to be particularly advantageous in cases where MS^n experiments added analytical information essential to structural identification.



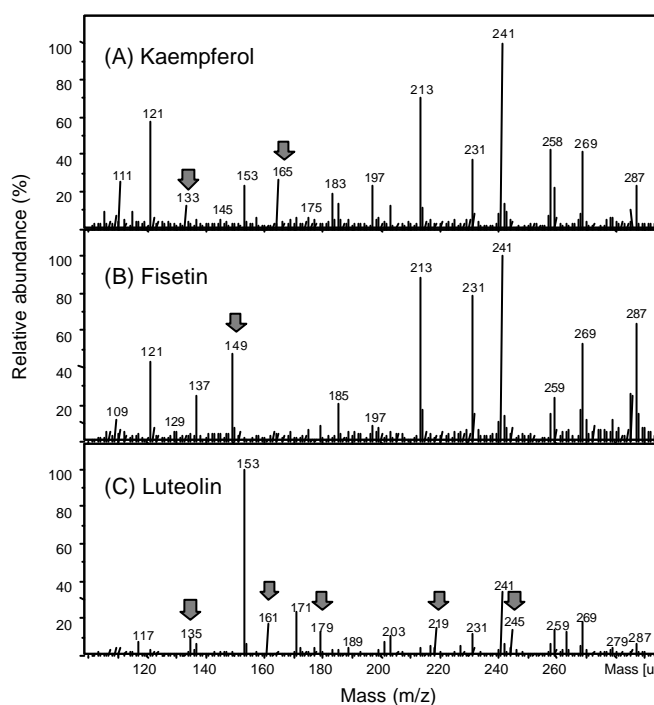
The AMOLF external ion-source Ion Trap Mass Spectrometer. (1) The sample can be positioned under the laser beam with micrometric precision. (2) Ions are produced from the surface of the paint sample (thin film on a metallic probe or embedded cross-section) and transferred to the ITMS for MS or tandem MS analysis.

LDMS was established as a valuable tool in the analysis of a number of flavonoid and anthraquinone pigments. MS/MS was proven to be capable of differentiating between three flavonoid isomers, luteolin, morin and kaempferol. Complex forms of alizarin (alizarin lakes) were successfully investigated with LDI. The spatially-resolved capacities of a focussed laser beam for LDMS sampling was proven to be successful for the analysis of a flavonoid at the surface of a dyed wool fibre but direct analysis of dyes in paint layers was less successful. LDMS proved to be a suitable technique for the differentiation between natural and synthetic indigo. Spatially-resolved analysis was successful with the direct identification of indigo on dyed fibre and within cross-sectioned paint samples. In mixtures of dyes in lead white, the LDI spectra revealed a strong contribution of lead related species that suggests a chemical reaction between indigo and lead white in the condensed phase under the action of the UV laser.



The effectiveness of the LDMS was demonstrated successfully for the analysis and characterization of modern pigments. Mass resolution of the TOF-MS analyser is sufficient to afford molecular formula determination of multi-chlorinated and brominated species by assigning their different isotopes. Analysis of quinacridone pigments shows that it is possible to simultaneously identify different compounds in a mixture (multi-component analysis). In MALDI experiments additives to the paint in trace amount were detected, which shows that the technique is suitable for interrogation of the purity of the samples.

The new possibilities offered by LDMS to investigate organic pigments by mass spectrometry directly from the surface of paint cross-sections were proved to represent a significant improvement. Spatially-resolved experiments at the surface of paint cross-sections showed that it is possible to positively identify the presence of a pigment – or a mixture of pigments – in an individual layer of circa 10 micrometers.



Above: Low-energy CID spectra of the protonated molecular ion of the three structural isomers kaempferol, luteolin and fisetin in an MS/MS experiment with the ITMS. Diagnostic fragment ions marked with an arrow make it possible to distinguish the three isomers.

Key publications: (1) N. Wyplosz, R.M.A. Heeren, G. van Rooij and J. Boon (2001) Analysis of natural organic pigments by laser desorption mass spectrometry (ldms): A preliminary study to spatially resolved mass spectrometry. *Dyes in History and Archaeology* 16/17 187-198. (2) Wyplosz, PhD thesis forthcoming.

Secondary Ion Mass Spectrometry maps binding medium in a paint cross-section

Katrien Keune

A paint cross-section provides a lot of information about the build up of the paint layers in a painting and about the paint materials themselves. The thickness and colour of layers, the shape and distribution of the pigment particles and their elemental composition in the layers, and the presence of medium rich layers can be deduced from a paint cross-section with microscopic and micro-spectroscopic



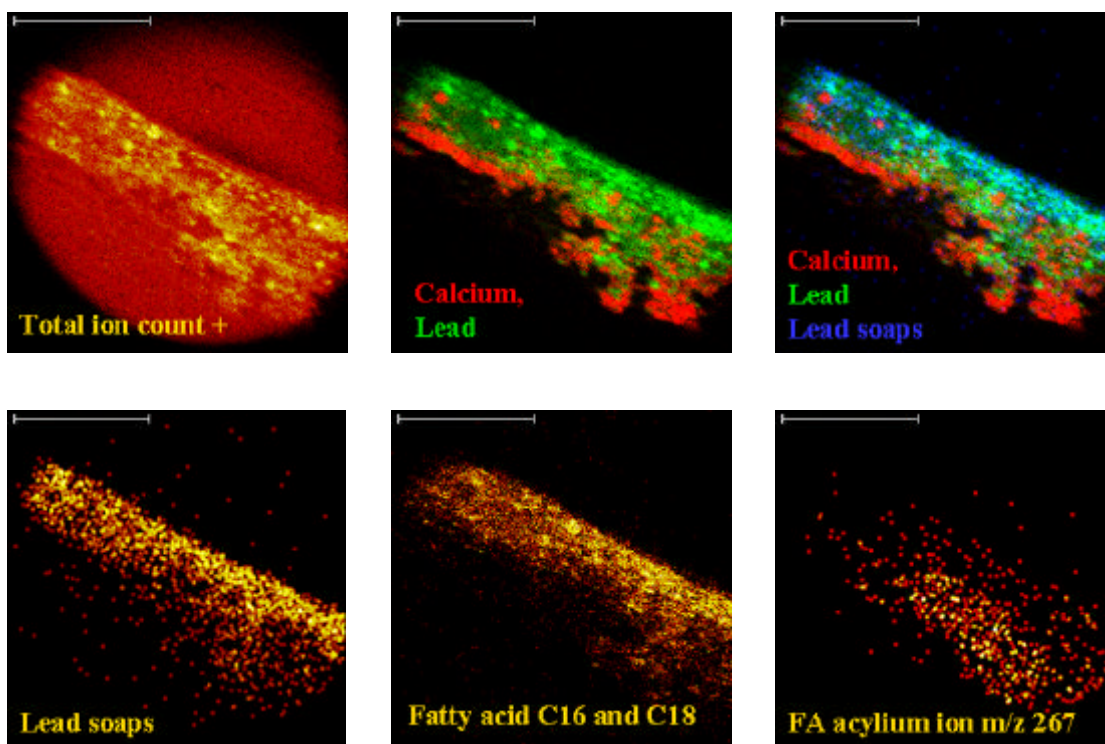
techniques. Secondary Ion Mass Spectrometry (SIMS) has been found to be a non-destructive additional technique to acquire information about the nature and distribution of pigments as well as binding media in a single measurement of a cross-section. SIMS is a highly surface sensitive technique, which uses a high-energy primary ion beam to generate

secondary ions. SIMS gives information about elemental composition as well as small and larger organic and inorganic fragments that can be related to molecular structures in the paint. An image is acquired by scanning of the focused beam over the surface, measurement of the mass spectra and reconstruction of the molecular distribution from the distribution of the ions.

SIMS images, visible in the figure, were acquired from a paint cross-section taken from a cream colored commercial 19th century pre-primed canvas from the estate of Frederick E. Church (1826-1900). Many paintings by Church and others from the Hudson River school are affected by disturbing paint defects such as ground staining, efflorescence and protrusion formation. FTIR studies in parallel demonstrated that these defects are related to the presence of lead carboxylates or lead soaps formed by trapping of mobile free fatty acids by lead ions.

The SIMS images illustrate that the primed canvas is build up of three layers. Calcium, a marker for chalk, is present in the ground and positioned in the lowest part of the paint cross-section. Lead, present as lead white, is observed in high concentration in a thin preparatory top layer. In between, a thick layer composed of a mixture of lead white (lead) and chalk (calcium) is visible. Lead soaps (the image is a sum image of lead soaps with FA C16 and C18) are present in higher concentrations in the top layer and in fact on the surface. A similar distribution is observed for the fatty acids, C16 and C18, which are derived from free and ester bonded fatty acids and lead soaps. In the calcium-rich ground mainly acylium ions of fatty acids, originating from ester bonded fatty acids, are visible. This indicates that in the lower part of the paint cross-section an oil polymer network is present with fatty acids that are still ester bonded.

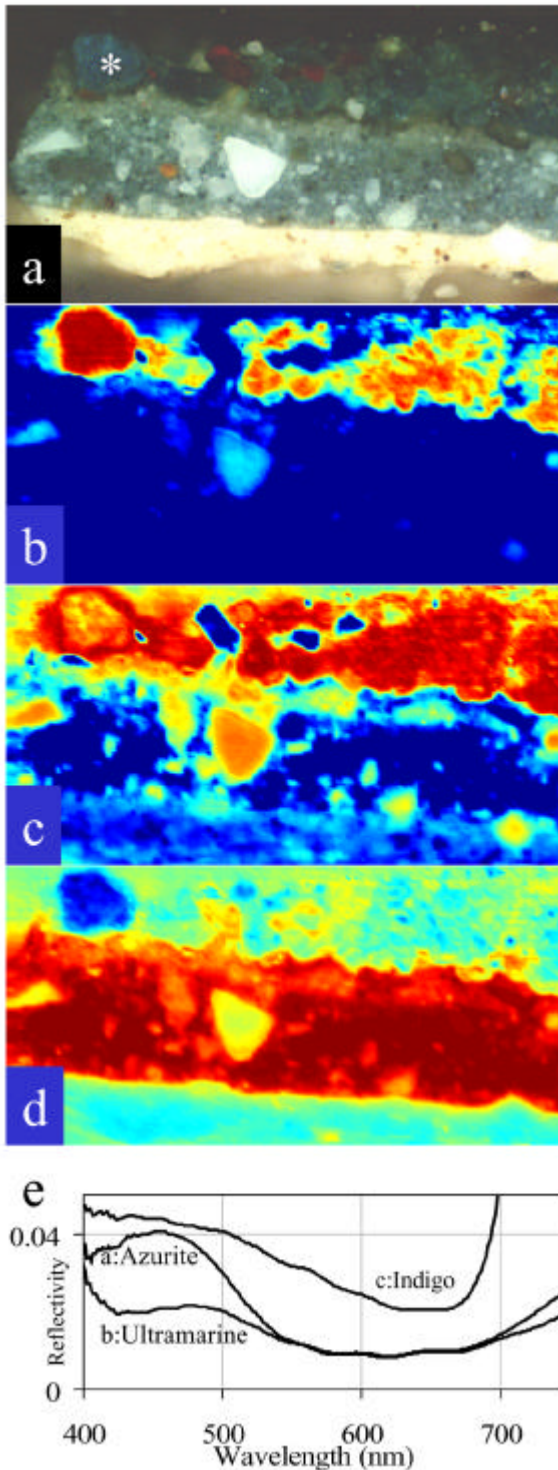
Although these distributions of fatty acids in various forms are only a snapshot, we presently believe that fatty acids are released in the lower layers of the ground which is likely to be too medium-rich. Part of these fatty acids migrates to the canvas itself and another part appears as bloom and efflorescence at the surface. The preparatory lead white paint is possibly saturated with lead soaps, because we see the formation of lead soap aggregates in this layer. These unique molecular images from SIMS provided vital additional information that supported the interpretation of the FTIR and DTMS data.



Key publications: (1) MOLART Report 6: Jorrit van den Berg *Analytical chemical studies on traditional linseed oil paints*. (2002) PhD Dissertation. University of Amsterdam. (2) MOLART Report 7: Jaap van der Weerd *Microspectroscopic analysis of traditional oil paint*. (2002) PhD Dissertation. University of Amsterdam.

Imaging microspectroscopic FTIR and VIS analysis of paint cross-sections

Jaap van der Weerd



Microspectral analysis of embedded paint cross-sections has played a key role in the MOLART project. Samples of various traditional paintings were investigated by advanced micro-spectroscopic imaging techniques in the infrared and visible wavelength ranges. Key to these techniques is that they analyse a large area of the surface of a cross-section instantaneously through the use of multichannel detection techniques (i.e. a camera). This approach provides combined spatial and spectral resolution without the need to move sample or analysis area. As a result the spectral image quality improved dramatically, as did the time needed for cross-section analysis.

The two techniques, FTIR and visible spectral imaging provide strongly complementary information. Visual light imaging for example has been used for identification of the pigments present in the cross-section. We were able to identify different blue pigments, entirely on the basis of their spectral properties. FTIR imaging on the other hand provides information on the organic materials present in the cross-section. In traditional paintings, the binding medium is the main source of this organic material.

VIS-imaging data of paint cross-section taken from the painting Vijf Muzen (1650) by Jan Lievens. (a) Optical microscopic image, (b-d) false colour score plots showing the localisation of azurite, ultramarine and indigo respectively, and (e) averaged reflection VIS-spectra from the highlighted regions.

A new sample preparation method, based on the old-fashioned method of pressing KBr pellets was developed to obtain thin sections that gave much more informative FTIR spectra.

It has become clear from the FTIR microspectroscopic imaging that the pigment-binder interactions play a far more important role than was anticipated. The imaging measurements were supplemented by a large series of normal FTIR measurements of different materials as these interactions had hardly been studied for aged traditional paints. The most dramatic result obtained is the observation that pigments such as lead white and zinc white can dissolve in and react with constituents of the binding medium. In this process, pigment particles are replaced by a gel-like substance with a much higher volume. The gel-like masses were found to consist of metal carboxylates or metal soaps. These materials were found to be present in paintings from Rembrandt, Vermeer, Steen, Hals, Cuyt and others. The metal soap aggregates were named 'protrusions', as they tend to protrude through the surface of a painting when they are formed in ageing paint-layers. Similar materials were found in a 19th century commercial primed but unused canvas from the estate of the painter F.E.Church. Here metal soaps appear at the surface as efflorescent crusts but on closer inspection slightly protruding metal soap aggregates are present as well. Since the canvas is not lined, it shows that metal soap can migrate through paint layers and organise themselves in liquid crystal-like form simply by diffusion.

Key publication: J. Van der Weerd (2002) *Microspectroscopic analysis of traditional oil paint*. (2002) PhD Dissertation. University of Amsterdam. MOLART Report #7, Chapters 7-8

Infrared reflectography with a European PtSi-camera

J.R.J. Van Asperen de Boer

Earlier European panel paintings were covered with a white ground. As a first stage in the creative process an underdrawing was applied on this ground – usually in black aqueous paint or black chalk – both containing carbon and subsequently covered by paint layers. Since the 1930's infrared photography using films responsive to c. 0.9 μ wavelengths were used to make such underdrawings visible again. This was only successful in reddish, brownish and whitish areas – blue and green paint not being penetrated and showing black. Earlier paintings are rarely signed and art historians have thus attributed paintings on stylistic grounds based on the painted surface. An underdrawing - not meant to be seen – can be important – in checking or modifying attributions and unraveling workshop practices.

Infrared reflectography was further developed in the 1960's by J.R.J. van Asperen de Boer [1] to improve upon infrared photography in revealing underdrawings. His research showed that paints including blue and green are most transparent when 'seen' in reflected radiation around 2 μ . At wavelengths beyond 3 μ emitted radiation becomes predominant. Infrared-to-visible image translation systems used in infrared reflectography are essentially instruments designed for military purposes. The first Barnes Infrared Camera was a modified research thermograph normally used to study images of emitted radiation (aircraft, tanks, soldiers, etc.). The second was a closed circuit television system provided with an infrared vidicon responsive up to 2.5 μ . the best vidicons were developed by Hamamatsu of Japan for

the Vietnam war. Such infrared reflectography systems became widely used in museums over the last decades for the examination of paintings. Although not cheap they are affordable and reliable. The main drawbacks are geometric distortion and inhomogeneity in the resulting reflectograms. Reflectograms are not as sharp as photographs and in order to be read only a small area on the painting can be registered



at the time. Reflectograms were photographed from the monitor screen in the first twenty years and the individual reflectograms printed on paper had to be pasted together. Since the late 1980's images are usually digitized with frame grabbers and pasted together with various computersoftware programmes such as Adobe Photoshop, PanaVua and VIPS.

Reflectography of a triptych by the Master of Moulins

After the dissolution of the Soviet Union in 1991 much military data was declassified. A new class of infrared detector systems became available for peaceful applications: infrared focal plane arrays. Their advantages are notably: low geometric distortion, high homogeneity, a resolution which can be better than the vidicon systems and higher responsivity.

In 1993 Walmsley *et al.* (National Gallery, Washington D.C.) reported the use of a 640 x 486 and a 512 x 512 pixel Thermal Imaging Camera for infrared reflectography [2] Part of the MOLART programme was to acquire such a new class infrared reflectograph – too expensive for most museums – as the first in Europe. A problem was that there is a ban in the USA on exporting infrared focal plane arrays of more than 100.000 image points. In Germany a 640 x 486 platinum silicide focal plane array camera was commercially available from AEG Infrarot Module, Heilbronn. This was purchased but required considerable modifications to operate properly as a reflectograph (3).

A number of paintings in Dutch museums previously examined with an infrared vidicon system was reflectographed again with the new instrument in order to asses the differences. Two paintings by the Master of Moulins of c.1500 were reflectographed for the first time in Autun and Moulins (*above*) and the results published [4]. Apart from its cumbersome weight (10 kg) making operating on tripods not easy, the main drawback of the instrument proved to be its lack of reliability. It broke down too often. When it functioned properly it produced beautiful reflectograms (*below*).

*Infrared reflectogram
assembly of a detail in the
right hand outside shutter of
the Moulins triptych*



References: (1) J.R.J. van Asperen de Boer, *Infrared Reflectography. A Contribution to the Examination of Earlier European Paintings*. Dissertation, University of Amsterdam, Amsterdam 1970.
(2) E. Walmsley, C. Metzger and J.K. Delaney, 'Evaluation of Platinum Silicide Cameras for Use in Infrared Reflectography', *Preprints ICOM Committee for Conservation, 10th Triennial Meeting*, Washington 1993, 57-62.

Key publications: (3) J. van der Weerd, R.M.A. Heeren and J.R.J. van Asperen de Boer, 'European 640 x 486 PtSi Camera for Infrared Reflectography', in: *Colloque XIII pour l'étude de la technologie et du dessin sous-jacent dans la peinture*, Bruges 1999, Leuven 2001, 231-243.
(4) Albert Châtelet, Jean Prévost. *Le Maître de Moulins*, Éditions Gallimard 2001.

Conservation oriented research projects

Research conservators were actively pursuing solutions for practical conservation problems in collaboration with the team.

Effects of cleaning procedures on solvent extractable components in oil paint films and paintings

Ken Sutherland

The extraction of soluble organic components from oil paint films was studied as part of an investigation into the risks associated with the exposure of oil paintings to solvents in cleaning and other conservation treatments. Organic solvents are the most widely used materials for cleaning paintings, that is, the removal of discoloured and deteriorated varnish and overpaint, as well as in other painting conservation treatments such as varnishing, inpainting and consolidation. A detailed understanding of the potential effects of solvents on paint layers – including the leaching of soluble components from the paint, which has been associated with changes in mechanical and optical properties – is important to provide a theoretical base for cleaning practice, to help conservators to predict, and minimise, the risks inherent in solvent treatments.

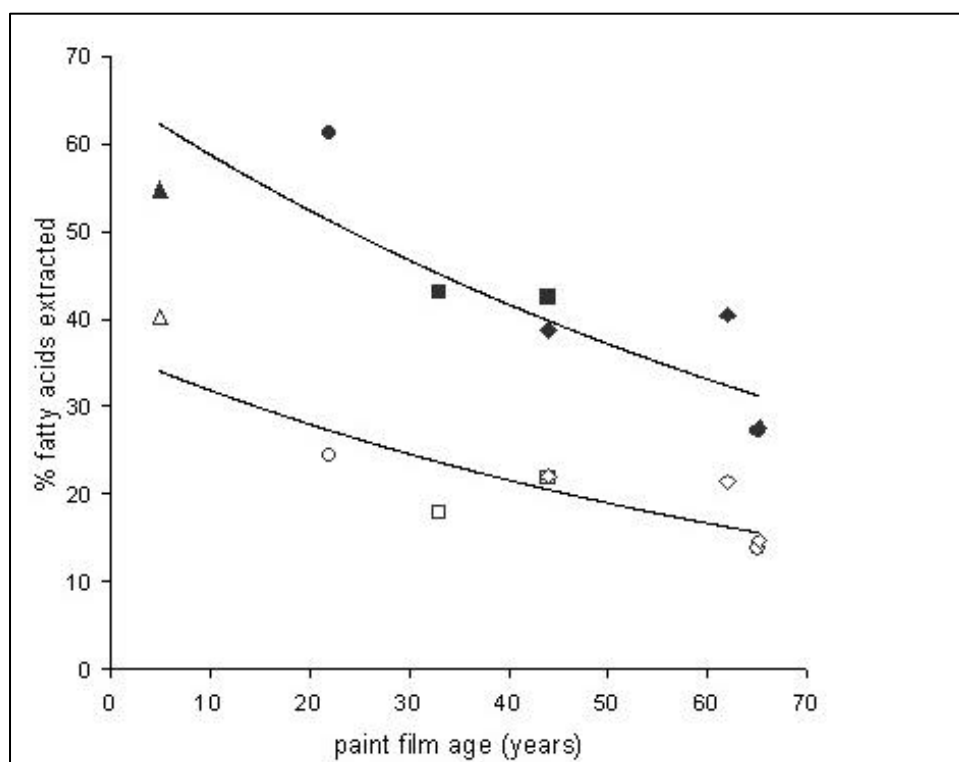
The study included gas chromatographic (GC) measurements of fatty acids in paint samples, and in solvent extracts from samples. Fatty acids in drying oils, such as palmitic and azelaic acid, are convenient markers for extraction studies as they can be readily converted to derivatives (methyl esters) which are amenable to quantitative GC analysis. The graph below shows a plot of quantities of palmitic and azelaic acids extractable from a series of linseed oil films pigmented with lead white, ranging in age from 5 to 65 years old, by immersion in acetone. The data indicate a trend of decreasing proportions of acetone extractable material with age, relating to ongoing chemical changes which occur in the paint films over extended ageing periods. Reactions such as continuing polymerisation of the drying oil, and interactions between organic and inorganic components of the paint film, may have contributed to a progressive chemical stabilisation of these paint films over time. Despite this general trend, however, significant quantities of extractable fatty acids were still found in older samples, including samples from paintings as much as 700 years old.

Experiments designed to investigate the potential leaching effects of more “realistic” exposures to solvent, as in cleaning treatments, were also carried out, for comparison with the more theoretical immersion studies. These treatment-based experiments, conducted on paintings dating from the seventeenth to nineteenth centuries, aimed to measure the quantities of fatty acids extracted from paint layers as a result of local solvent cleaning treatments.

Paintings examined included several large-scale seventeenth century oil paintings in the Oranjezaal, Huis ten Bosch, den Haag. In the tests, a small, but measurable, proportion of soluble fatty acids was found to be removed from the paint layers in some cases, whereas in others the effect was too small to be reliably

determined. This was despite the use of polar solvents, and prolonged exposure to swabs in most cases. The results indicate that although a considerable amount of soluble material can remain in very old paint films, extractable by immersion in solvent, in the examples tested only a very small proportion of this appeared to be removed by superficial exposure to solvent as in a single cleaning treatment. It is worth noting that these experiments are based on individual cleaning treatments, and do not account for the possible cumulative effects of repeated treatments over time.

Key publications: (1) Sutherland, K., 'The extraction of soluble components from an oil paint film by a varnish solution', *Studies in Conservation* **45** (2000) 54-62. (2) Sutherland, K., 'Solvent extractable components of linseed oil paint films', *Studies in Conservation* (in press).



Proportions of palmitic acid (solid figures) and azelaic acid (hollow figures) (sum of free acid + glyceride ester forms) extracted from lead white paint samples after 24 hour immersion in acetone, expressed as percentages of total fatty acids in samples.

The conservation-restoration history of the wax-resin lining procedure

Mireille te Marvelde

Conservation-restoration history as a discipline is still in its infancy. I choose the history of the wax-lining procedure as a focus within the more general research into the history of conservation-restoration, because it was feasible to integrate it into a multidisciplinary process. Important progress was made within MOLART by developing a research methodology for the discipline, which takes the investigation of the paintings themselves as the starting point while supporting the outcome with documentary and analytical research. In this way information that was believed impossible to obtain could be gathered, and information from written sources that seemed not explicit enough could be linked and interpreted in a bigger framework that provides possibilities for further development of a more detailed history of conservation-restoration.

A first tangible result of the wax-resin study is the clarification of when, where, by whom and why wax-resin lining was invented. Moreover, for the first time the early international dissemination of the method could be followed in detail, and the initial intentions with every phase of the method, the choice of materials, and the different developments of the method, could be traced. Knowledge on the behaviour of the materials was increased in some respects.



The adoration of the kings by Salomon de Koninck, *Mauritshuis*, showing the 1861 lining of Nicolaas Hopman.

It was proven that wax-resin lining was invented in the middle of the nineteenth century by Nicolaas Hopman sr. in the Netherlands and not earlier elsewhere in Europe as was asserted by Volker Schaible in 1992. From the Netherlands the method was first introduced to Germany in the 1870s, and came to Austria near the end of the century. The subsequent introduction of the method into the U.S.A., England and France could also be traced. Slowly the wax-resin lining

method was changed and developed. It became an important topic of discussion in the international conservation-restoration literature.

Several old existing linings were found and subsequently investigated, attributed to various restorers, dated and chemically analysed.

Until recently it appeared that Hopman sr.'s last extant documented wax-resin lining had been removed when Rembrandt van Rijn's *Nightwatch* was restored in 1947. However, during studies in the context of MOLART a remaining wax-resin lining applied by Hopman in 1861 was identified on the *Adoration of the Kings* by Salomon de Koninck in the Mauritshuis in The Hague.

Recently another wax-resin lining was discovered that can also be attributed with high certainty to Hopman sr. on a painting by Cesar van Everdingen in the Frans Hals Museum in Haarlem that has recently been re-attributed. Over the last 140 years this painting was considered to be of little importance and stayed mainly in the depot. The quality of the lining is today still superb and does not require removal.

The finding these earliest existing wax-resin linings and the possibility of studying them was always thought to be no more possible. The existence of these old linings is of high importance for our knowledge of the history of conservation-restoration of paintings and a means to investigate the ageing properties of wax-resin mixtures. The results of these studies are highly relevant for the preservation of paintings.

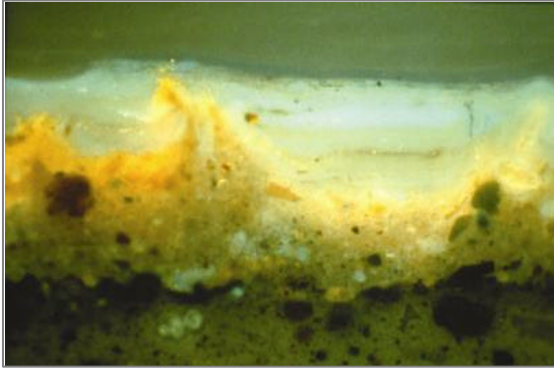
Key publications: (1) The forthcoming book on the Oranjezaal (see p.65.) (2) Marvelde, M.M. te. "Research into the history of conservation-restoration: Remarks on relevance and method", in 12th Triennial Meeting, Lyon 29 August – 3 September 1999 (ed. J. Bridgeland), ICOM-CC, vol 1, 194-199, James and James, London. (3) Marvelde, M.M. te. "How Dutch is 'The Dutch Method'? A History of Wax-Resin Lining in its International Context", in Past Practice-Future Prospects, The British Museum Occasional Paper 145 (ed. A. Oddy and S. Smith), London 2001, p.143 -149.

The effect of the Pettenkofer regeneration process on paintings

Sibylle Schmitt (MOLART fellow)

In this MOLART-fellowship project the multidisciplinary research approach proved to be most effective. All aspects of MOLART were involved and enriched fruitfully the knowledge about the effects. The genesis of this historical conservation method and the background of its reception in Europe were studied in detail. A new and appropriate terminology was developed in German, English and French for the discovered and classified migrations in paint layers and varnishes, thus helping an international professional discussion.

A microscopic field study in various museums in several parts of Europe was carried out, revealing similar treatments and corresponding dramatic migrations of original paint particles up into the varnish (*below, top*). We acknowledge the National Gallery in London (UK), the Royal Cabinet of Paintings the Mauritshuis in Den Haag (NL), the Staatliche Gemäldesammlungen in Kassel (D), the Bayerische Gemäldesammlungen in München (D), the Gemälde-galerie in Berlin (D), the Herzog-Anton-Ullrich-Museum in Braunschweig (D), and the Stadtmuseum in Köln (D).



Detailed archive study revealed notes on about 150 paintings that had been involved with regeneration treatments. Pettenkofer used alcohol and copaiba balsam as regeneration substances. Stable markers of copaiba identified by Inez van der Werf et al. were used to trace the use of the balsam as a conservation compound in the aged paint matrix. Evidence for copaiba balsam was found in samples taken from a number of regenerated paintings and also as additive in the paint by Van Gogh used in the Nuenen period.

The effects in paintings could be reconstructed in historically accurate test systems and successfully photo-documented in 3-D-laser-microscopy, normal light, ultra-violet (marble-like migrations, *left, lower picture*). These images will be combined in a forthcoming research report with

comments on the restoration history of works investigated. This information will form a didactic catalogue of defects. This 'BILD-Atlas' will be directed to colleagues as a manual, transferring actual research results immediately into the field of practice. The first mechanical measurements of test fields realized are very promising. Reconstruction tests, analytical work and microscopy of layer structure will be continued in the next few years as part of a PhD study in Germany to elucidate the causes for the effects observed. The migrations are perceived as 'mobile phases' activated by three factors, aging of the paint, possible use of poor quality materials combined with a weak painting technique and later conservation treatments. Preliminary thermomechanical studies supported by MOLART and performed in collaboration with Dr. M. Odlyha (Birkbeck College London) revealed that copaiba balsam and alcohol vapour significantly increased the plasticity of the paint in a fragment from a 18th painting available for study. The PhD thesis work will focus on the overlap of the two disciplines science and conservation looking back on Pettenkofer's initial interdisciplinary activity and realising actual evaluation of this conservation method by making use of the modern instruments of both disciplines in order to support preservation of regenerated paints in the future.

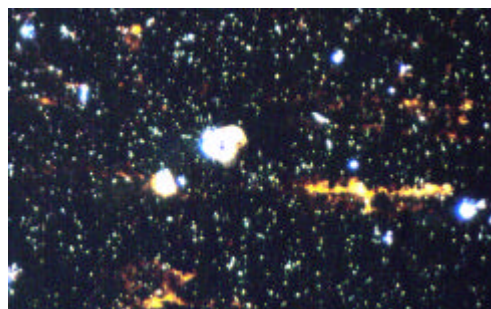
Key publications: (1) Schmitt, S. The Reprint of Professor Max von Pettenkofer's 1870 publication 'Über Ölfarbe', ICOM Kongressakten 1999, Lyon, Vol. I, p.188 - 193; (2) Van der Werf et al. 2000 (*Studies in Conservation*) (3) Schmitt (2001) (*in press*); Schmitt, S. "Progress in research of the Pettenkofer Process", In: 'Art et Chemie - La couleur' Congressakten Paris 1998, Sonderdruck der Zeitschrift 'Techne', CNRS Edition Paris 2000, p. 137 - 140, Fig. XI

Drying problems in nineteenth-century paintings and the role of bitumen in the darkening of paint

Rene Boitelle

One of the most interesting events of this project was the examination of *La Descente des vaches*, (Museum Mesdag, The Hague) by Théodore Rousseau (1812-1867). The examination involved closely linking art historical, technical and analytical data (Presented in 2001, see below). Forty years after its completion, *La Descente*, created to impress the Parisian art world in 1834-35, was recorded to have darkened severely. As well as darkening, the paint surface shows dramatic drying defects (youth cracks and areas of contracted paint) to such a degree that the painting has become almost unreadable. The starting point for the examination of *La Descente* was a chapter in Sensier's biography of the artist (1872), listing the techniques and materials said to be at the root of the painting's problems. Apart from a better understanding of Rousseau's complex use of colour and build up of paint layers, close examination of the painting, cross-sections and binding media analysis revealed that most of Sensier's explanations for the defects were correct. In addition to this, the discovery of a manufacturer's stamp on the reverse of the support provided an important clue to the understanding of Rousseau's painting.

Reconstruction of bituminous paint - Semi transparent protrusions and cracking lead white ground layer showing through a bituminous paint layer after artificial ageing.



This manufacturer Brullon advertised “bituminous products” from a village in Eastern France in 1835. Mapping different bituminous and asphaltic reference materials by means of mass-spectrometric analytical techniques prior to this examination, provided a proper reference to interpret the results of the analysed dark brown paint samples of *La Descente*. More specific than labelling “bitumen” or “bituminous products” as the main cause for darkening and bad drying, it could be established that the defects on this painting were (at least partially) caused by the careless use of lignite containing colours – probably similar to the ones found in Eastern France at the beginning of the 19th century. Furthermore, examination of this painting in the context of the 19th century painting project established that many paint layers are extremely medium rich, contain complementary coloured particles and lack the necessary opacity to reflect light.

In collaboration with SRAL Maastricht, reconstructions of bituminous paints based on 19th century sources were prepared for artificial ageing in order to understand the development of drying defects.

Drying defects: Youth cracks in dark, medium rich paint layers on Th. Rousseau's Descente des vaches (1834-35). The transparent red brown underlying paint layer has become visible.



These reconstructions were made on glass tiles, with two water and oil based grounds, three bituminous materials and using three different recipes that were considered to be representative of late 18th century and 19th century practices. Although this project could not be brought to a final stage, some of the paint surfaces started to show small deformations (wrinkles in the brown paint layers). Surprisingly, some of the lead white grounds also started to protrude and crack. Also, semi-transparent yellowish/greyish lumps, probably having been formed in the these same grounds, had migrated towards and through the surface of the bituminous paint layers on top within eight months after having been applied. These lumps are not unlike the phenomena that are part of ongoing research into metal soap formulation in oil paintings.

Key publication: Boitelle, R., Van den Berg, K.-J., Geldof, M. and Languri, G.M., “Descending into the details of Th. Rousseau’s *La Descente des vaches* (Museum Mesdag, The Hague) – Technical Research of a Darkened Painting”, *Deterioration of Artists’ Paints: Effects and Analysis. Preprints of the Joint Meeting of ICOM-CC Working Groups 1 & 2 and The Painting Section, UKIC*, London 10-11 Sept. 2001, 27-31.

Dosimetry of the museum environment using egg tempera paint strips

Oscar F. van den Brink

Paintings change and decay slowly as a result of continuous interactions with their environment. In order to increase the life-time of the paintings, and thus preserve the cultural heritage for future generations conservation departments of museums pay a lot of attention to the control and monitoring of the museum environment.

The indoor environment of museums and galleries is subject to fluctuations in parameters such as relative humidity, temperature, light intensity and levels of pollutants such as nitrogen oxides. The indoor conditions in museums and galleries are studied by monitoring of a number of these factors. However, it is important not just to measure these variables, but also to investigate the actual damage that is caused by each of these factors. And even more important, to investigate the damage resulting from their synergistic action which may well differ from the damage caused by the individual factors. Individual paintings are themselves dosimeters of damage incurred upon exposure to their environment. The most effective way of assessing the overall damage to works of art in a given environment would be to measure changes with time in the objects themselves.

The idea was conceived in the ERA project (EC’s Environment Programme EV5V-CT-94-0538) to produce mock paintings of known composition and history which function as paint-based dosimeters that can be analysed by a variety of destructive and non-destructive techniques in order to determine the integrated effect of their exposure to the environment. A twin-track exposure of the paint-based dosimeters to predefined laboratory conditions and to the environmental conditions of five selected European museums was carried out. The paint-based dosimeters consisted of an egg tempera based binding medium mixed with a variety of organic and inorganic artist’s pigments. The museum-exposure sites included rooms where environmental factors were controlled as well as rooms with uncontrolled

environments. After exposure the paint-based dosimeters were analysed by a variety of techniques which includes thermo-analytical and mechanical testing (Birkbeck College, University of London, England) and visible spectroscopic analysis (CNR-IROE, Florence, Italy). A semi-quantitative comparison of the molecular changes in the exposed paint-based dosimeters involved mass spectrometric techniques and the multivariate technique of discriminant analysis (DA).

Oxidation and hydrolysis were identified as the most important processes that take place upon exposure of the test systems. Results obtained on the dosimeters that were exposed to laboratory conditions of light, thermal and NO_x/SO_2 concentrations clearly show that different environmental factors affect the paint systems in different ways. In all cases a positive correlation of the degree of chemical change with the duration of light exposure was observed. Each of the paints has its own response to the environmental conditions in the museums where they were exposed.

Results obtained on the paints with inorganic pigments classify the Tate Gallery (Clore gallery) as the best field site in terms of environmentally induced change. This is attributed to a better air quality. At the time of exposure the Tate Gallery was the only site where carbon filters were used to remove air pollutants from the inlet air of the air-conditioning system. In many cases, the uncontrolled environments of the Alcázar (Segovia, Spain) and Sandham Chapel (Burghclere, England) were found to show the largest chemical differences compared to the unexposed controls. In all cases the changes in the field-exposed dosimeters were much greater than would be expected on the basis of the reciprocity principle for light ageing (which states that the extent of damage on a system is proportional to the product of light intensity and exposure time). The dosimeter that was exposed in the Depot “Oost” of the Rijksmuseum (Amsterdam, NL), which is an almost dark site, consistently showed a significant change in the chemical composition. This is another indication that other factors than light alone contribute significantly to the molecular changes of the tempera paints. The fact that the dosimeters are strongly affected by exposure to nitrogen oxides and sulphur oxides in the dark further confirms this observation. The work carried out in the project has been reported in a PhD thesis.

Photograph of a test painting (in circle) on a storage rack in the Rijksmuseum Depot “Oost”.

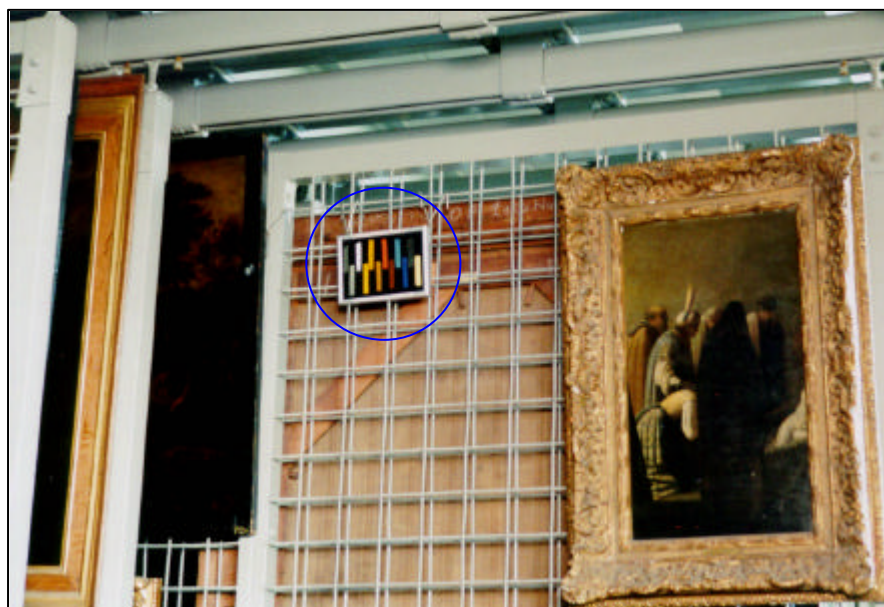
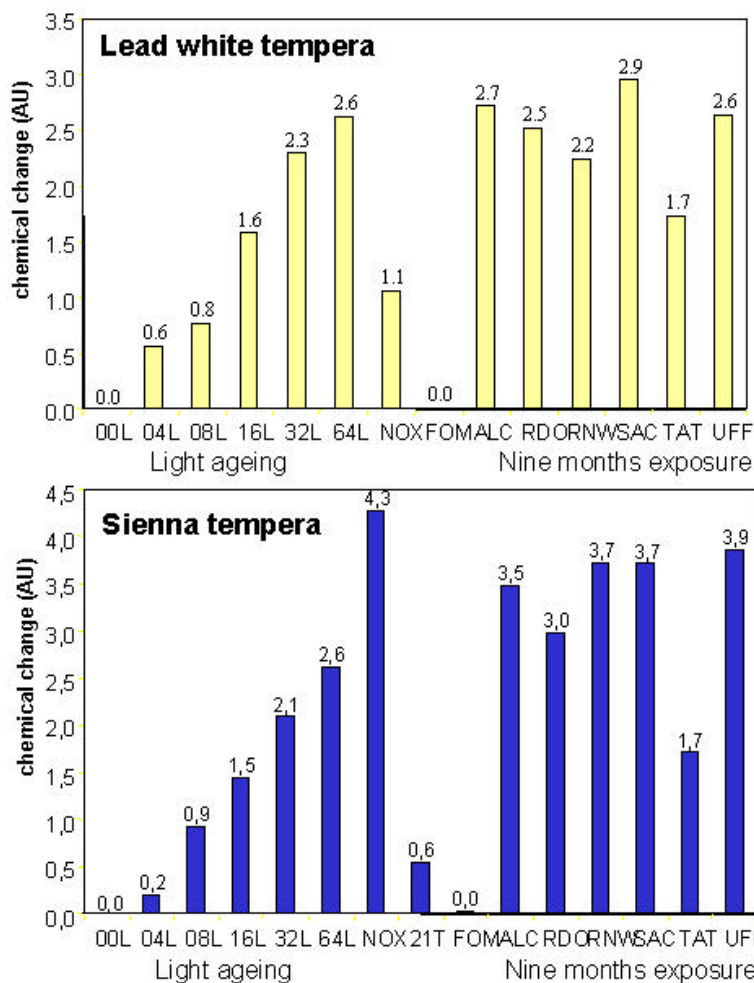


Figure showing the degree of chemical change in sienna and lead white dosimeters reflecting the difference in response to environmental conditions

The last chapter of the thesis gives recommendations for improvement and further testing of the principle of paint-based dosimetry. It must be noted that some of these recommendations are currently being applied in a new project which aims at further development of paint-based dosimetry (MIMIC, Microclimate Monitoring in Cultural Heritage Preservation). In addition, paint-based dosimeters which are similar to the ones used in the

ERA project have been used in a study of the effects of laser cleaning of paintings in the framework of the European CRAFT-project “Advanced workstation for controlled laser cleaning of artworks”.



Key publication: (1) MOLART Report 4: Oscar F. van den Brink *Molecular changes in egg tempera paint dosimeters as tools to monitor the museum environment* (2001) University of Amsterdam. (2) O.F. van den Brink, G.B. Eijkel and J.J. Boon (2000) Dosimetry of paintings: Determination of the degree of chemical change in museum-exposed test paintings by mass spectrometry. *Thermochim. Acta* vol.365, p.1-23.

Multidisciplinary painting studies

MOLART supported several multidisciplinary studies of paintings applying the newly acquired fundamental knowledge. This posed new challenges for the team of scientists, conservators and art historians.

Painting techniques in the Oranjezaal

Lidwien B.J. Speleers

The restoration of the Oranjezaal in Huis ten Bosch (1998-2001) was an opportunity for a study of the 40 paintings in this seventeenth century ensemble. Because the paintings had never been closely studied before, the restoration revealed a lot of new data on the paintings and their production. The conservators had a strong desire to learn more and compare the techniques of the twelve painters that worked in the Oranjezaal



Lidwien Speleers and Gisela van der Doelen were given the opportunity to start a research program on this subject in the MOLART program in 2000. Since January 2002 it continues in the De Mayerne program 'Comparative Studies of Paintings in the Oranjezaal' with Margriet van Eikema Hommes and Lidwien Speleers.

MOLART was already involved in the Oranjezaal project via studies on the varnishes, wax-resin linings and cleaning. Whereas during MOLART our study was limited to painting technique, in the De Mayerne program the relation of technique and style is further explored. For the study of painting techniques and materials in the MOLART program the paintings were studied, sampled and paint cross-sections were made and interpreted.

One of the paintings studied is *Frederik Hendrik triumphant* by Jacob Jordaens (1652). It is the largest painting in the Oranjezaal and measures circa 7.5 by



7.5 meters. Its size raises the question how the painter could have fitted the painting in his Antwerp studio. The canvas consists of several strips of canvas sewn together. One of the seams is clearly visible. A study of the area revealed that the painting was prepared and painted in two separate parts and joined in the Oranjezaal.

The canvas consists of several strips of canvas sewn together. Here the seam is clearly visible.

To reduce the height of the painting, Jordaens rolled the painting. As a result the canvas has left imprints in the wet paint. Also from the painting itself it is clear that the painter worked in zones. The two columns that flank the scene over the entire height change colour once or twice

and both columns have different capitals.

To match the two halves of the painting in the Oranjezaal a ground layer was applied over the seam and the parts were painted together. The brushstrokes on the seam are painterly and freely done and resemble the brushwork elsewhere in the painting and the painter took the liberty to work a bit further on the areas around the seam. So it seems that Jordaens or a good assistant travelled to the Hague to finish this part of the painting.



By careful study we can thus learn a lot about the painting and how it was made. It also makes us realise how difficult it was to paint this large picture. As Jordaens said in a letter: *“Het valdt in het groot ongelyck moeyelycker, als gegist hadde, ende het aldermoeyelyckxste, dat ick t niet en can geheel in mijn huijs wtspannen, soodat ick t meer met het concept in t hooft dan met het gesicht heb moeten wtvoeren om sijn moeyelycke groote”* “The actual scale makes painting disproportionately more difficult than estimated, and

most difficult is not to be able to stretch it out completely in my house, so that I had to execute it with the concept in my head rather than visually because of its difficult size.”

Key publications: (1) The forthcoming book on the Oranjezaal (see p. 65.) (2) These results were presented on the symposium ‘Art history and technical investigation; the genesis of 17th century paintings’, 29-30/10/2002 RKD Den Haag as ‘Het valdt in het groot ongelyck moeyelycker, als gegist hadde...’ Making the large-scale paintings for the Oranjezaal. [unpublished]

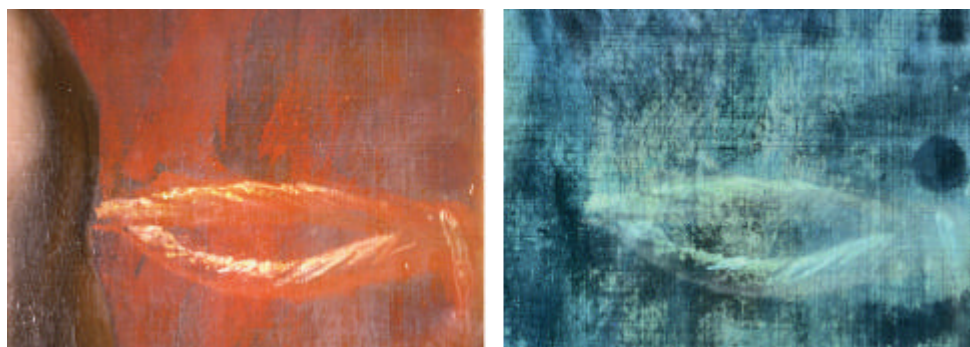
Research into the conservation-restoration history of the paintings in the Oranjezaal

M. te Marvelde, L.B.J. Speleers and G. van der Doelen

One of the results of the research into the conservation-restoration history of the paintings of the Oranjezaal was that different varnish layers, related to the subsequent treatments, could be identified. Remains of varnishes that had been removed from the paint surface in the past were found behind the frames. The identification of the varnishes using scientific methods allowed us to correlate them with the archival data that had been collected by Paula van der Heiden. As a result we were able to find out who applied which layer of varnish, and in what period each of these layers was removed. These were unique results as in general it is impossible to obtain this kind of information in the field of historical painting.

The information gathered adds to our knowledge of historical varnishes and to a more detailed knowledge about the people who applied and removed various layers. But this new knowledge also is of a further significance. It provides a better understanding of the condition of the paintings on a molecular level.

An example: The red dress of a child at the right edge of the painting *Part of the triumphal parade, carrying along war booty* by Pieter de Grebber has turned locally, irregularly, into dark gray. The red pigment was identified as vermilion, which led to the conclusion that the paint layer suffers from the so-called ‘vermilion disease’. The color change is caused by a chemical reaction within the mercuric sulfide pigment, that is promoted by exposure to light, humidity and salts (in, for instance, dirt).



Detail of Part of the triumphal parade, carrying along war booty by Pieter de Grebber, Oranjezaal, Paleis Huis ten Bosch, The Hague. Photograph SRAL. The photographs show a detail of the red dress of a child at the right edge of the painting, in normal light and in ultraviolet light. In ultraviolet light varnish remains are visible, some of them showing a yellow fluorescence. Below this yellow fluorescent varnish remains the paint layer did not discolor from red to dark grey, as it did in many other areas

Studying the dress in ultraviolet light showed that the areas that had kept their red color were covered with remains of a strong yellow fluorescent varnish. This varnish, identified as a diterpenoid resin, was the most clearly identifiable varnish on the Oranjezaal paintings, because of its differently colored fluorescence compared to that of the other varnishes. The latter varnishes show a green fluorescence, as we normally observe when we study old varnishes and they are most of the time made of triterpenoid resins. On the basis of various arguments, the yellow fluorescent

(diterpenoid) varnish could be identified as the one applied in 1767 by Jan van Dijk (c.1695 – 1769). It is extremely rare to find remains of a varnish that was applied at such an early date. Its surprising deviation in fluorescence must be related to its material properties but is still without explanation.

From the visual analysis in ultraviolet light we could draw the conclusion that the red paint had not turned grey in those areas where Huijbrechts, the restorer who removed Van Dijks varnish in 1806, had left some remains of that varnish. The color change in the vermilion must have occurred as a result of Huijbrechts treatment. On the basis of this understanding it was decided, during the recent conservation-restoration of the Oranjezaal, not to remove the remains of Jan van Dijks varnish, in order not to risk the the discoloration of the preserved red paint.

Key publication: The forthcoming book on the Oranjezaal (see p. 65.)

Still Life Paintings in the Rijksmuseum: techniques and style

Arie Wallert

Still life painting was practised by the most prominent masters of the seventeenth century. In conjunction with a major Dutch 17th century exhibition, a selection of important still life paintings from the Rijksmuseum's own collection were restored. The restoration campaign presented an opportunity for technical examinations of the pictures. During restoration, when dirt, yellow varnishes and overpaint may be removed, the objects are more readily accessible for close examination and analysis. Thus, we were able to address a number of questions regarding the relationship between style and technique.

Dutch seventeenth and early eighteenth century still life painting is marked by significant stylistic developments. Various types of still life pictures, differing in composition, style and use of colour, can be distinguished throughout these centuries. Beside stylistic changes, still life painting also showed significant technical changes. These changes are related to materials as well as to working methods. What did it take to produce such works? Did all still life painters of the seventeenth century use the same methods and materials? If so, why are there so many striking differences? If they used different materials, or the same materials in a different way, can we relate this to differences in style?

These questions were explored in a small catalogue in an introductory essay covering the characteristics of the materials, the equipment, the working properties of pigments and the like. The study dealt with the typical materials and working methods in the seventeenth century painter's workshop.

Aside from the information to be culled from the paintings themselves, there is a wide range of knowledge about material aspects and working procedures to be gained from seventeenth century technical documents. These contain recipes for the preparation of paints; sometimes comment on the characteristics and uses of various colours, their working qualities, their ageing properties, and techniques for refining; or focus on the proper arrangement and combinations of certain pigments. In an essay on technical sources, attention was also given to prescriptions for the rendering of

shadows and highlights on round surfaces, to the composition, and even to the general organisation of the studio.

The paintings that were examined in the conservation studio were discussed in a number of technical 'entries'. In these entries, questions regarding the making of the paintings, problems of preservation, and the relationship between the application of various materials and methods and the historical technical sources are discussed.

Questions like these can best be addressed in an interdisciplinary study. This is easily facilitated in a situation where paintings conservators, scientists, art historians and museum curators collaborate in such a way that the practice and knowledge of the different disciplines enrich one another. In this case the staff, conservators and curators of the Amsterdam Rijksmuseum and participants of the MOLART project collaborated in order to answer these questions. An objective of MOLART is the determination of the present chemical and physical condition of works of art in comparison to the chemical and physical state of the original, unaltered state of the works. Towards this aim, studies of historic paint manufacturing and workshop practice gave insight into the nature of the painter's materials and the original painting technique.



Still Life, Balthasar van der Ast (1620-1) Tiny traces of red lake (preserved below the frame) show that the tablecloth has completely discoloured, and would have originally been purple-red.

Key publication: *Still Lifes: Techniques and Style The Examination of Paintings from the Rijksmuseum*, ed. A. Wallert, Waanders, Zwolle, 1999.

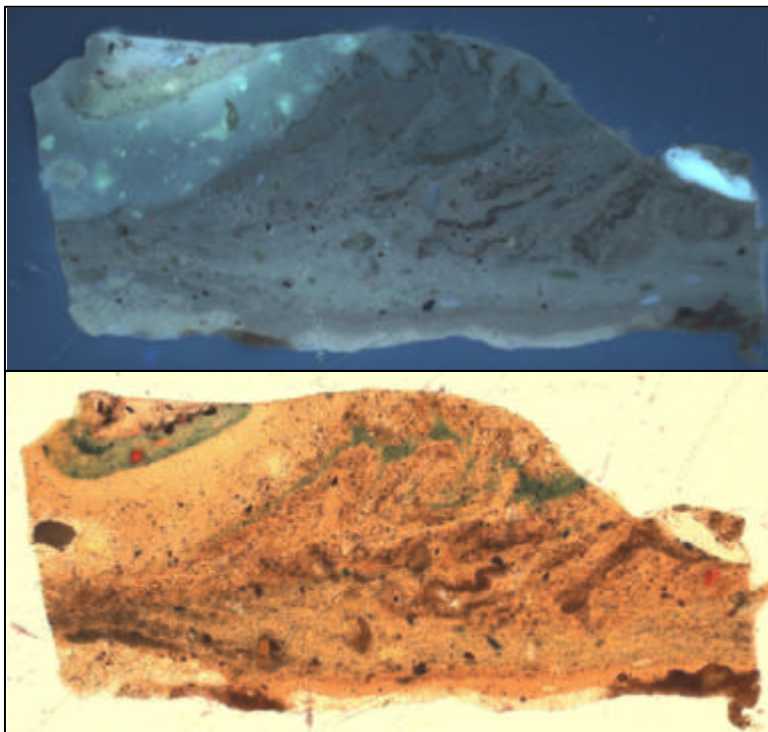
Colour changes in nineteenth century paintings

Klaas Jan van den Berg

The 19th Century project was carried out from 2000 to 2002 at the ICN in co-operation with several museums in the Netherlands. In this project, the presumed darkening of nine 19th and early 20th Century paintings from Theodore Rousseau, Jozef Israels, Jacob Maris, Willem Witsen and Floris Verster was studied through extensive technical research. This information was combined with documentary research in to letters and articles, which describe the painting techniques and use of materials of the artists, in combination with information on the original appearance of the works of art.

This broad and interdisciplinary research brought about much information on the working methods of the artists and the relation with the appearance of the paintings. As expected, both painting techniques and use of materials by the artists varied widely. Nevertheless, some general patterns could be found.

In several darkened paintings, severe surface degradations such as premature cracks were observed as well. It was established that these deformations are mostly associated to the painting technique of the artist, who applied many still relatively wet paint layers on top of each other. In contrast, darkening was associated mostly with the use of materials, especially transparent and medium-rich linseed oil paints. In my view the darkening in these paintings seems to be associated mostly with the yellowing of the paint medium.



Cross-section of Rousseau's La Descente des Vaches in reflection UV-fluorescence, showing medium-rich layers.

Thin section photographed in transmission, showing the large number of very binding medium-rich paint layers.

Key publication: K.J. van den Berg, M. Geldof, S. de Groot and H. van Keulen. Darkening and surface degradation in 19th- and early 20th century paintings an analytical study. in *ICOM Committee for Conservation 13th Triennial Meeting*, Rio de Janeiro, 22 -28 September 2002, 2002, 464-472.

Protrusions: metal soap aggregates in oil paintings

Petria Noble

A serious deterioration phenomenon in lead pigmented paint layers was identified in numerous paintings spanning the 16th to the 20th centuries. Far more widespread than first suspected this has now been recognised in hundreds of paintings. Often the entire surface of a painting shows protruding light-coloured lumps that are perfectly round, and regular in size, hence the name protrusions. Exhibiting a diameter between 100 and 200 micrometers their presence can easily be revealed using a binocular stereomicroscope or by close scrutiny of x-radiographs where the less dense masses show up as dark spots in the x-ray picture.

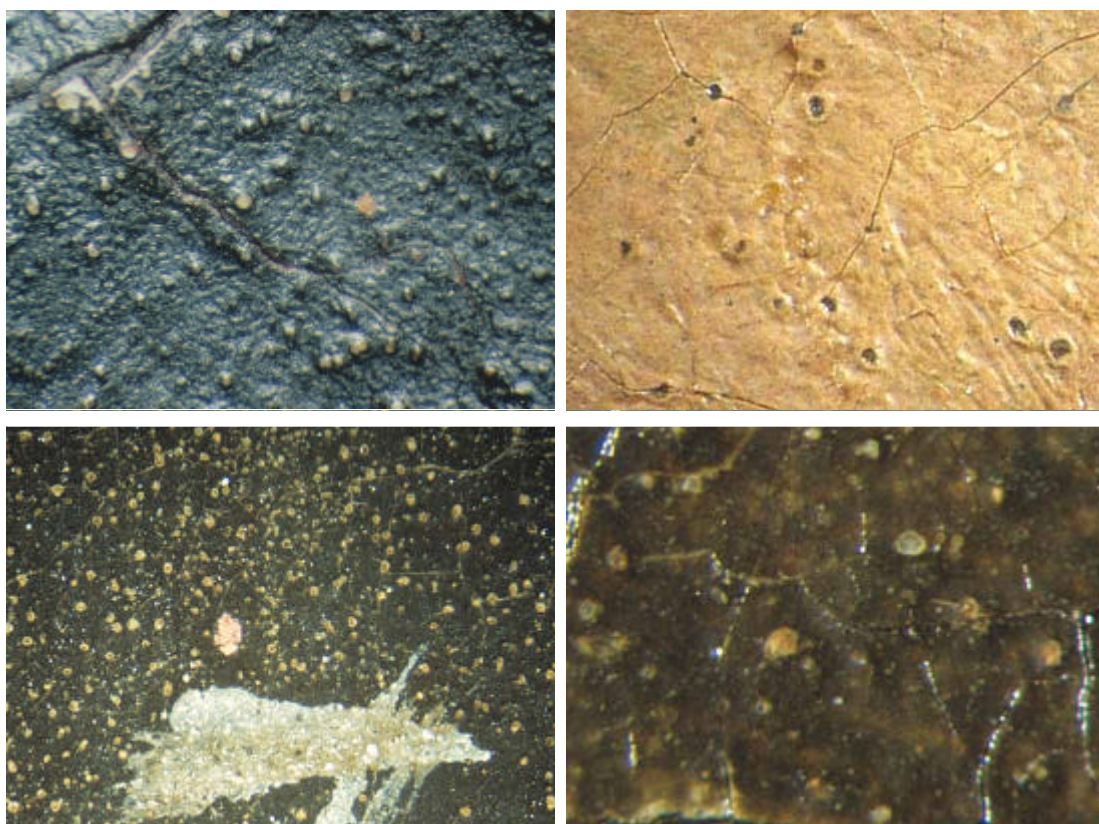
Where the lumps are partially lost, small crater-like structures that sometimes contain residual opalescent material is often all that remains. When dirt / varnish residues collect in the craters they can appear as masses of black spots. Analyses of cross-sections with Imaging FTIR, SEM/EDX, Imaging SIMS and micro-spectroscopy, in collaboration with the MOLART team, so far tells a consistent story. The protruding material, consisting of metal carboxylates (soaps) and newly formed inorganic compounds in an organic-rich network, are related to chemical changes in the composition of a particular ground or paint layer. It appears that the original particulate pigment matter of the paint has "dissolved" and the normally dispersed metal soaps have undergone damaging aggregation, expansion and re-mineralisation processes resulting in large firm lumps that protrude up through the paint surface causing the crater-like structures and paint loss observed on the surface. The microscopic characteristics have been described in several of the articles given in the references. Subsequent abrasive surface treatment may cause the crater-like structures to become enlarged resulting in a very disfiguring surface appearance.

Where these lumps lie just below the surface of a painting a bubbly surface texture can result that is not the original intention of the artist. A similar process has also been found to occur in zinc pigmented paint layers from 19th century paintings where firm lumps, but often also softer material exudes through the cracks in the varnish.



*Decapitated protrusion, topped with varnish layer
cross-section Rembrandt van Rijn's
The Anatomy Lesson of Dr. Nicolaes Tulp.*

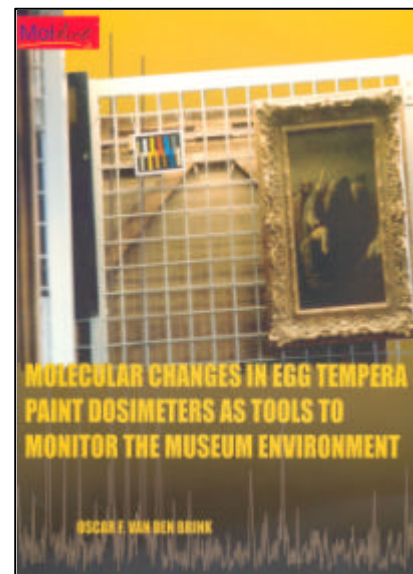
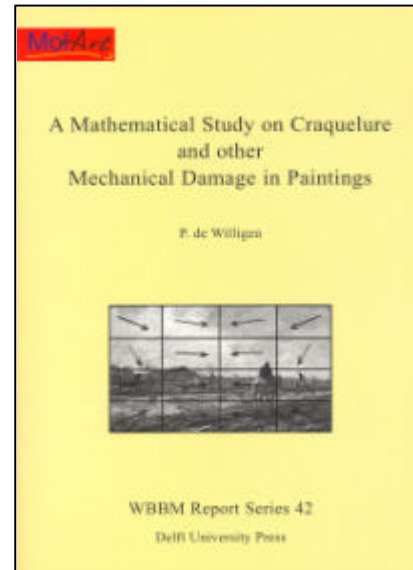
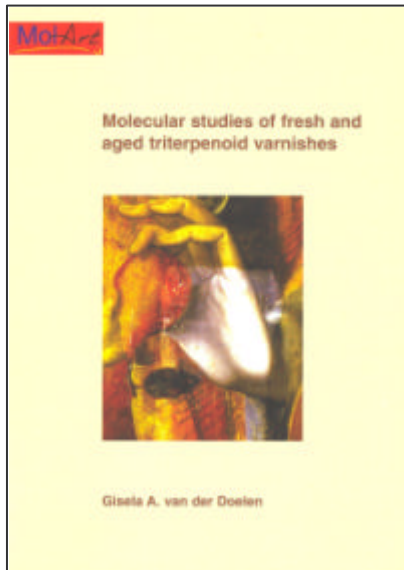
The causes for the deterioration and the driving forces are presently unknown. We presently think that, in some cases, the process is still on going. Research is being continued in the context of MOLART II, the De Mayerne Research Program (2002-2006) where the dynamics of dissolution, soap formation and protrusion are being studied. A questionnaire has been prepared and sent out to museums worldwide in order to increase the awareness, and to better gauge the extent and diversity of this deterioration phenomenon.



Macro and microphotographs of protruding lumps / associated craters

Key publications: (1) P. Noble, J.J. Boon and J. Wadum (2002) Dissolution aggregation and protrusion: lead soap formation in 17th century grounds and paint layers. In: *Art matters : Netherlands technical studies in art*, edited by E. Hermens. (Waanders, Zwolle) vol.1, p. 46-61. (2) J.J. Boon, J. van der Weerd, K. Keune, P. Noble and J. Wadum (2002) Mechanical and chemical changes in Old Master paintings : dissolution, metal soap formation and remineralization processes in lead pigmented ground/intermediate paint layers of 17th century paintings. In: *13th triennial meeting Rio de Janeiro 22-27 September 2002 : ICOM committee for conservation*, edited by R. Vontobel. (James & James, London, 2002) (Preprints, 1) p. 401-406.

MOLART Reports

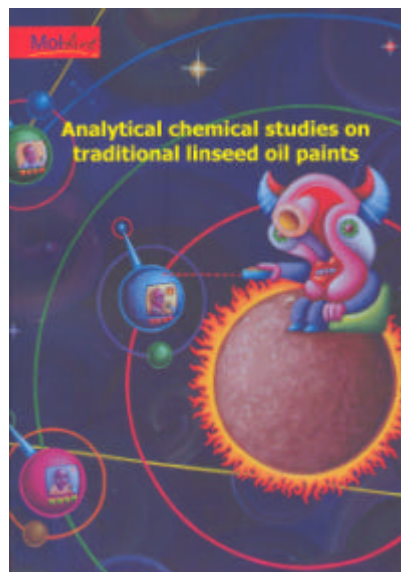
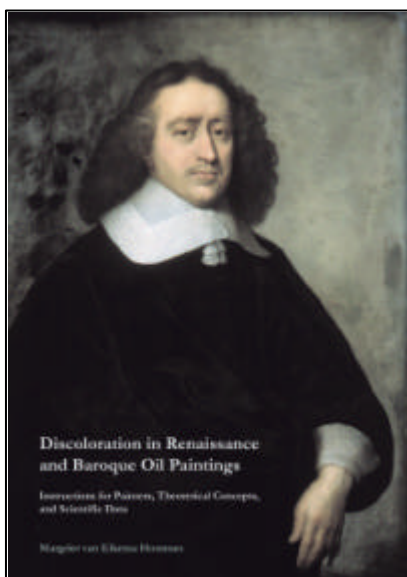


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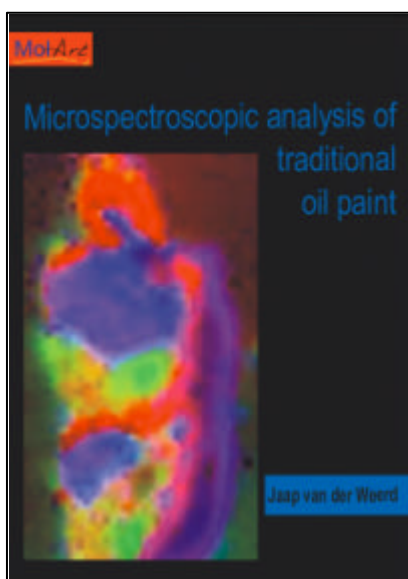
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10: Klaas-Jan van den Berg *Diterpenoid varnishes: with an atlas of Mass Spectra*, forthcoming

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- Part 1: Publications 1995-2002
- Part 2: Theses, books and other reports
- Part 3: Forthcoming publications
- Part 4: Presentations 1995 - 2002
- Part 5: Contributions presented at MOLART meetings

MOLART reports

The MOLART project produced theses and other books, among which the yearly cumulative *MOLART Research Projects and Progress Report*. That report is available for free at the MOLART secretariat at the FOM Institute for Atomic and Molecular Physics. It can also be viewed and printed via the menu on the MOLART homepage:

www.amolf.nl/research/biomacromolecular_mass_spectrometry/molart/molart.html

This book, *MOLART Highlights 1995-2002*, is available at http://www.nwo.nl/nwohome.nsf/pages/NWOP_5MBJWD?OpenDocument

Forthcoming book on the Oranjezaal

The RKD (Rijksbureau voor Kunsthistorische Documentatie / Netherlands Institute for Art History) is preparing a book on the Oranjezaal, which will include the results of the recent restoration (1998-2001) and of the research of the MOLART and De Mayerne projects. Chief editor is Dr. R.E.O. Ekkart, director of the RKD. The publication will be in English. It is hoped that the book will be published in 2004.

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J.J. Boon (lecture). Seminar on Mass Spectrometry of Art.

ASMS conference on Mass Spectrometry and Allied Topics, Atlanta, United States of America, May 21-26, 1995

J.J. Boon, K.J. van den Berg, J. Pureveen, G. van der Doelen, K. Groen, J. van Och and A. van Grevenstein (poster). Direct temperature resolved mass spectrometry (DTMS) as a tool to study painted art: a case study of "De Eendenfamilie" by W. Maris a 19th century Dutch impressionist painter.

R.M.A. Heeren, G.J. van Rooij, M.C. Duursma, C.G. de Koster en J.J. Boon (poster).

Characterisation of electrosprayed polymers with MALDI-FT-ICR-MS.

Historical Painting Techniques, Materials, and Studio Practice, Leiden, June 26-29, 1995

J.J. Boon (invited lecture). Molecular aspects of ageing in art: MOLART.

G.A. van der Doelen and J.J. Boon (poster). Characterisation of Dammar and Aged Dammar Varnishes by DTMS and HPLC/GCMS.

K.J. van den Berg, J.J. Boon, J. Pureveen, G. van der Doelen, K. Groen, J. van Och and A. van Grevenstein. (poster). A Versatile Tool for the Study of Painted Art: Direct Temperature-resolved Mass Spectrometry of "De Eendenfamilie" by W. Maris.

J.J. Boon en J. Pureveen (poster). Molecular aspects of painter's materials and painting technique in Rembrandt's Jewish Bride as revealed by DTMS and GCMS.

Conservation Science Group Meeting of the UKIC, Tate Gallery, London, United Kingdom, July 28, 1995

J.J. Boon (invited lecture). Mass spectrometry as a tool to study paintings.

3rd European workshop on ICR, Bremen, Germany, September 4-6, 1995

C.G. de Koster, M.C. Duursma, P.W. Arisz, R.M.A. Heeren and J.J. Boon (lecture). MALDI-FTICR-MS/MS of Cellulose O-Methyl ethers.

G.J. van Rooij, M.C. Duursma, R.M.A. Heeren, C.G. de Koster and J.J. Boon (lecture). Polymer characterisation by MALDI-FTICR-MS.

Resins Ancient and Modern, Symposium Aberdeen, Aberdeen, Scotland, September 13-14, 1995

G. van der Doelen and J.J. Boon (lecture). Mass spectrometry of resinous compounds from paintings: Characterisation of dammar and naturally aged dammar varnish by DTMS and HPLC/GCMS.

"Holland at CERN", CERN, Geneva, Switzerland, October 16, 1995

J.J. Boon (invited lecture). Mass spectrometric analysis of paintings.

Symposium Patroonherkenning ivm. 50-jaar Gerechtig Laboratory, Scheveningen, November 16, 1995

J.J. Boon, G. Eijkel and C. Visser (invited lecture). Microschaal onderzoek van schilderijen: deconvolutie van temperatuursopgeloste massaspectrometrische data met een neuraal netwerk.

European Commission Research Workshop: Effects of the Environment on Indoor Cultural Property, Würzburg, Germany, December 11-13, 1995

J.J. Boon, E.R.E. van der Hage, J. Pureveen and O.F. van den Brink (poster). Dosimetry of Paintings: Molecular changes in paintings as a tool to determine the impact of environment on works of art.

National Gallery of Art, Washington, February 21, 1996

J.J. Boon (lecture). Mass spectrometric studies of painting materials and paintings.

Art Historical Institute, University of Amsterdam, Amsterdam, February 26, 1996

M. van Eikema Hommes (lecture). Zeventiende eeuwse schildersrecepten.

Canadian Conservation Institute, Ottawa, Canada, February 28-29, 1996

J.J. Boon (lecture). Paintings and painting materials studied by organic mass spectrometry and the MOLART project

R. Hoppenbrouwers (lecture). Reconstruction of historical paintings at SRAL.

The 44th ASMS Conference on Mass Spectrometry and Allied Topics, Portland, Oregon, May 12-16, 1996

J.J. Boon, K.J. van den Berg, G. van der Doelen, E. van der Hage, J. Pureveen and G. Eijkel (lecture). Role of MS techniques in the characterisation of fresh and aged natural and synthetic varnishes used for paintings.

K.J. van den Berg, I. Pastorova, L. Spetter and J.J. Boon (poster). Oxidation of diterpenoid Pinaceae resins in old master paintings.

P.O'Connor, R.M.A. Heeren, M. Duursma, G. van Rooij, E.R.E. van der Hage and J.J. Boon (poster). Analysis of polymers using FTMS.

R.M.A. Heeren, P.B. O'Connor, M.C. Duursma, G.J. van Rooij, E.R.E. van der Hage, C.G. de Koster and J.J. Boon (lecture). FTICR-MS characterisation of (co)-polymers.

New Vermeer Studies, Den Haag, May 30-31, 1996

K. Groen (lecture). The background of Vermeer's 'Girl with the pearl earring'.

K. Levy-van Halm (lecture). What's in between theory and practice? Buying painting materials.

E. van de Wetering (lecture). Vermeer's pictorial means.

11th Triennial Meeting ICOM-CC, Edinburgh, Scotland, September 1-6, 1996

J.J. Boon and J. van Och (lecture). A mass spectrometric study of the effect of varnish removal from a 19th century solvent-sensitive wax oil painting.

O.F. van den Brink, J.J. Boon, E.R.E. van der Hage and S. Peulvé (poster). Dosimetry of paintings: molecular changes in paintings as a tool to determine the impact of environment on works of art - Part II.

K.J. van den Berg, I. Pastorova, L. Spetter and J.J. Boon (lecture). State of oxidation of diterpenoid Pinaceae resins in varnish, wax lining material, 18th century resin oil paint, and a recent copper resinate glaze.

G. van der Doelen, K.J. van den Berg and J.J. Boon (poster/short oral). Mass spectrometric studies on triterpenoid varnishes sampled from easel paintings in museum collections.

M.H. van Eikema Hommes (lecture). A proposal for the classification of painting recipes.

K. Groen, M. de Keyzer and E. Baadsgaard (lecture). Examination of the painting technique of nine Dutch pictures of the first half of the 18th century.

M.M. te Marvelde (lecture). Jan van Dijk, an eighteenth-century restorer of paintings.

I.D. van der Werf, S. Schmitt, K.J. van den Berg and J.J. Boon (poster/short oral). Copaiba balsam used in the Pettenkofer process and as an additive in 19th century painting techniques: studies of its effect on paint layers.

DESORPTION '96, Ronne, Bornholm, Denmark, September 18-21, 1996

G.J. van Rooij, R.M.A. Heeren and J.J. Boon (poster/short oral). A New Set-Up for Spatially Resolved Surface Analysis by Ion Trap Mass Spectrometry.

Early Italian Painting Techniques and Analysis, Maastricht, October 9-10, 1996

J.J. Boon, O. van den Brink, S. Peulvé and D. Rainford (invited lecture). Molecular aspects of mobile and bound fractions in egg tempera.

O.F. van den Brink, P.B. O'Connor, M.C. Duursma, S. Peulvé and J.J. Boon (poster). Characterization of the lipid fraction of egg binding medium by mass spectrometry.

O.F. van den Brink, J.J. Boon, E.R.E. van der Hage, and S. Peulvé (poster). Dosimetry of paintings: molecular changes in paintings as a tool to determine the impact of environment on works of art - Part II.

P.B. O'Connor, S. Peulvé, O.F. van den Brink, M.C. Duursma, R.M.A. Heeren and J.J.

Boon (poster). Mass spectrometric analysis of protein networks in tempera paints.

G. A. van der Doelen, L. Spetter and J.J. Boon (poster). Preliminary results: investigation on the effects of varnish removal on early Italian panel paintings (study of the mobile and chemically bound phase).

I.D. van der Werf, S. Schmitt, K.J. van den Berg and J.J. Boon (poster). Copaiba balsam used in the Pettenkofer process and as an additive in 19th century painting techniques: studies of its effect on paint layers.

Pyrolysis '96, Venice, Italy, October 14-18, 1996

J.D.J. van den Berg, J.J. Boon, O. van den Brink, L. Spetter, J. Pureveen, and I. Fiedler (poster). Identification of an unknown synthetic varnish from the early 20th century oil painting "The White Horse" (1929) by H. Menzel.

K.J. van den Berg, I. Pastorova, I.D. van der Werf, L. Spetter and J.J. Boon (lecture). Ageing phenomena in works of painted art as studied by thermally assisted methylation and pyrolysis techniques.

I. Pastorova, K.J. van den Berg and J.J. Boon (poster). Composition and state of preservation of diterpenoid acids in archaeological artefacts: a study of Mediterranean Bronze Age amber artefacts and sealing glues from Roman barrels.

Art Historical Institute, University of Amsterdam, Amsterdam, October 17, 1996

M. te Marvelde (lecture). Massaproductie van portretten door de schilder Martinus Meytens (1695-1770); bewijs vanuit technische aspecten.

Art Historical Institute, University of Amsterdam, Amsterdam, October 24, 1996

M. van Eikema Hommes (lecture). Schildertechniek van het kostuum in de portretkunst.

Rotterdams Natuurkundig Genootschap, Rotterdam, November 18, 1996

J. J. Boon (lecture). Massaspectrometrisch onderzoek aan geschilderde kunst-voorwerpen.

NVMS (Dutch Society for Mass Spectrometry), Eindhoven, November 22, 1996

J.D.J. van den Berg, J.J. Boon, O.F. van den Brink, L. Spetter, J. Pureveen and I. Fiedler (poster). Identification of an unknown synthetic varnish from the early 20th century oil painting "The White Horse" (1929) by H. Menzel.

K.J. van den Berg, I. Pastorova, L. Spetter and J.J. Boon (poster). State of oxidation of diterpenoid Pinaceae resins in old master paintings.

O.F. van den Brink, P.B. O'Connor, M.C. Duursma, S. Peulvé, and J.J. Boon (poster). MALDI-FT-MS of egg yolk lipids and their oxidation products.

G. van der Doelen, K.J. van den Berg and J.J. Boon (poster). Mass spectrometric studies on triterpenoid varnishes sampled from easel paintings in museum collections.

I.D. van der Werf, S. Schmitt, K.J. van den Berg and J.J. Boon (poster). Copaiba balsam used in the Pettenkofer process and as an additive in 19th century painting techniques: studies of its effect on paint layers.

Bio-Rad FTIR users meeting, Baarn, November 27, 1996

R.M.A. Heeren, N. Wyplosz and J.J. Boon (lecture). Infrarood imaging aan verfdwarsdoorsnedes; Schilderijen in een ander licht bekeken.

Seminar: Beelden uit het Atelier. Historisch onderzoek naar de praktijk en theorie van de kunstproductie, Vincent van Gogh Museum, Amsterdam, December 13, 1996

R. Hoppenbrouwers (lecture). Reconstructie van historische schildertechnieken.

Varnishes Colloquim, SRAL, Maastricht, March 17, 1997

A. Phenix (lecture). Artists' varnishes: an historical perspective.

A. Phenix (lecture). Synthetic resins as surface coatings for paintings.

4th European Workshop on FTMS, Pont à Mousson, France, April 27-30, 1997

O.F. van den Brink, P.B. O'Connor, M.C. Duursma, S. Peulvé, R.M.A. Heeren, and J.J. Boon (lecture). MALDI-FTMS of fresh and oxidised egg lipids in tempera paint samples: Determination of the degree of oxygenation.

O.F. van den Brink, P.B. O'Connor, M.C. Duursma, S. Peulvé, and J.J. Boon (poster). MALDI-FT-MS of egg yolk lipids and their oxidation products.

45th ASMS Conference on Mass Spectrometry and Allied Topics, June 1-5, 1997, Palm Springs, CA, USA

O.F. van den Brink, P.B. O'Connor, M.C. Duursma, S. Peulvé, and J.J. Boon (poster). Analysis of egg lipids and their oxidation products by MALDI-FTMS.

Staatliche Akademie der Bildenden Künste, Institut für Technologie der Malerei, Stuttgart, June 3, 1997

A. Phenix (lecture). The cleaning of painting with organic solvents re-considered.

1st International ICCROM course on the contribution of sciences to the conservation of cultural heritage, Paris, June 2 – July 4 1997

N. Wyplosz (lecture). The participation of the FOM-AMOLF in the NWO project on molecular aspects of aging in painted works of art.

5th European Meeting on Mass Spectrometry in Occupational and Environmental Health, Nijmegen, June 9-11, 1997

O.F. van den Brink, P.B. O'Connor, M.C. Duursma, S. Peulvé, R.M.A. Heeren, and J.J. Boon (poster). MALDI-FT-MS of egg lipids and their oxidation products.

14th International Mass Spectrometry Conference, Tampere, Finland, August 25-29, 1997

J.D.J. van den Berg, K.J. van den Berg and J.J. Boon (poster). GC/MS analysis of fractions of cured and aged drying oil paint.

K. J. van den Berg, J. van der Horst, J.J. Boon, N. Shibayama and E.R. de la Rie (lecture). Mass spectrometry as a tool to study ageing processes of diterpenoid resins in works of art.

J.J. Boon, S. Peulvé, P.B. O'Connor, M.C. Duursma, O.F. van den Brink, and R.M.A. Heeren (poster). Mass spectrometric studies of cross linked proteins in egg tempera paint.

O.F. van den Brink, P.B. O'Connor, M.C. Duursma, S. Peulvé, and J.J. Boon (poster). Determination of the degree of oxygenation of egg lipids in paint media by MALDI Trapped Ion MS.

R.M.A. Heeren, G.J. van Rooij, N. Wyplosz, P.B. O'Connor, M.C. Duursma and J.J. Boon (poster). A novel external ion source MALDI QITMS for analysis of biomaterials and synthetic polymers.

I.D. van der Werf, G.M. Languri, K.J. van den Berg and J.J. Boon (poster). Py-TMAH-GCMS and isomerisation of copalic acid and related diterpenoids.

Historical sources on Painting Techniques and Materials: Interpretation, Reconstruction and their Place in Art Historical Research, Netherlands Institute for Cultural Heritage, Amsterdam, August 28-29, 1997

M. H. van Eikema Hommes (lecture). The painters anticipation on colour changes as discussed in sixteenth to eighteenth century sources on oil painting techniques.

Netherlands Institute for Cultural Heritage (ICN), Amsterdam, September 12, 1997

M.M. te Marvelde (lecture). "Restoration history of the paintings in the Oranjezaal Huis ten Bosch". For: external advisory commission to the preliminary research report on the condition of the paintings in the Oranjezaal.

Scientific Workshop on FTIR Spectroscopic Imaging, Amsterdam, 21 October 1997

N.Wyplosz (lecture). Analysis of embedded paint cross-sections with FTIR and mass spectrometric imaging.

SRAL, Maastricht, October 27, 1997

M.M. te Marvelde (lecture). History of the wax/resin lining in Holland; Development of ideas, materials en techniques, research done in the past, research in MOLART today.

Varnishes Colloquium, Netherlands Institute for Cultural Heritage (ICN), Amsterdam, November 4, 1997

A. Phenix (lecture). Artists' varnishes: an historical perspective.

A. Phenix (lecture). Synthetic resins as surface coatings for paintings.

NVMS (Dutch Society for Mass Spectrometry), 's Hertogenbosch, November 14, 1997

J. J. Boon, S. Peulvé, P. B. O'Connor, M. C. Duursma, O.F. van den Brink, and R.M.A. Heeren (poster). Mass spectrometric studies of cross linked proteins in egg tempera paint.

O.F. van den Brink, J.J. Boon, E.R.E. van der Hage and S. Peulvé (poster). Dosimetry of paintings: molecular changes in paintings as a tool to determine the impact of environment on works of art - Part II.

O. F. van den Brink, P.B. O'Connor, M.C. Duursma, S. Peulvé, and J.J. Boon (poster). Determination of the degree of oxygenation of egg lipids in paint media by MALDI Trapped Ion MS.

N. Wyplosz, A. Wallert, R.M.A.Heeren, J.J. Boon. (Poster). Organic pigment analysis with mass spectrometry.

Symposium: In Restauratie. Het wel en wee van de beeldende kunst, Museum Het Catharijneconvent, Utrecht November 22, 1997

R. Hoppenbrouwers (lecture). Natuurwetenschappelijk onderzoek in dienst van conservering en restauratie. Het NWO MOLART-project: aspecten van veroudering en de invloed van restauratieprocedures op kunstvoorwerpen.

Conference "Fifth International Symposium on Hyphenated Techniques in Chroma-

tography and Hyphenated Chromatographic Analyzers", Brugge, Belgium, Feb 11-13, 1998

G.A. van der Doelen, K.J. van den Berg, and J.J. Boon (poster). Comparative chromatographic and mass spectrometric studies of triterpenoid varnishes.

Beobachtungen zur Gemäldeoberfläche und Möglichkeiten ihrer Behandlung, Kolloquium, Bern, Germany, March 13-14, 1998

J.D.J. van den Berg (invited lecture). Analytical chemistry of oil paint: a revised chemical model of aged paint relevant to the cleaning of paintings.

K.J. van den Berg, G.A. van der Doelen and J.J. Boon (invited lecture). Ageing of varnishes. Theoretical viewpoints, practical consequences.

A. Phenix (invited lecture). Organic solvents and the cleaning of paintings: an introduction.

A. Phenix (invited lecture). The effects of organic solvents on paint and varnish.

NVMS (Dutch Society for Mass Spectrometry) meeting, Amsterdam, March 31, 1998

J.D.J. van den Berg (lecture). Analytical techniques for identification of the non-cross-linked fraction of drying oil paints.

N. Wyplosz, R. Heeren, J.J. Boon (lecture). Q-ITMS/MS in the analysis of organic pigments.

SSCR Site Effects: The impact of location on conservation treatments, Dundee, United Kingdom, 5-6 May 1998

O.F. van den Brink, S. Peulvé and J.J. Boon (lecture). Dosimetry of paintings: chemical changes in test paintings as tools to assess the environmental stress in the museum environment.

89th American Oil Chemists' Society Annual Meeting and Expo, Chicago, USA, May 10-13, 1998

J.D.J. van den Berg (poster). Analyses of cured and aged traditional linseed oil paints (*honoured with gold medal*).

Canadian Conservation Institute, Ottawa, Canada, May 19, 1998

J.D.J. van den Berg (lecture). Analytical chemistry of oil paint: a chemical model of aged paint relevant to the cleaning of paintings.

Third infrared users group in conservation research, Wintherthur museum, Winterthur, Delaware, USA, May 28-30, 1998

R.M.A. Heeren (invited lecture). Rapid FTIR imaging of paint cross-sections in the MOLART project.

46th ASMS meeting, Orlando, Florida, USA, 31 May - 4 June 1998

N. Wyplosz, R.M.A. Heeren, J.J. Boon (poster). Analyses of natural organic pigments by ITMS, FTMS and TOF-MS.

Conference "Firniss", Braunschweig, Germany, June 15-17, 1998

J.J. Boon, G.A. van der Doelen, J. van der Horst, K.J. van den Berg and A. Phenix (lecture). Changes in the composition of natural varnishes as a result of ageing processes.

L. Carlyle, N. Binnie, A. Ruggles, B. McLean and G.A. van der Doelen (lecture). Traditional painting varnishes project – preliminary report on the natural and artificial ageing and the preparation of cross-sections.

G.A. van der Doelen, K.J. van den Berg, and J.J. Boon (poster). Cross-linking processes in dammar and mastic varnishes.

Painting Techniques: history, materials and studio practice. IIC conference, Dublin, Ireland, September 7-11, 1998

J.H. Townsend, L. Carlyle, A. Burnstock, M. Odlyha and J.J. Boon (lecture). Nineteenth-century paint media: the formulation and properties of megilps.

E. Hendriks, M.H. van Eikema Hommes, K. Levy- van Halm (lecture). Indigo used in the Haarlem Civic Guard group portraits by Frans Hals.

O.F. van den Brink, S. Peulvé and J. J. Boon (poster). Early changes in tempera paint.

M.M. te Marvelde and K.J. van den Berg (poster). An unusual pastiglia-like technique in the eighteenth century.

A. Wallert (lecture). Technical Aspects of Late Byzantine Psalter Illuminations; some preliminary results'.

M. Zeldenrust and A. Wallert (poster). Johannes Torrentius's Emblematic still life with flagon, glass, jug and bridle

'Art et Chimie' – la couleur, International congress on contribution of chemistry to the works of art, Palais du Louvre, Paris, September 16-18, 1998

K. J. van den Berg, M. H. van Eikema Hommes, K.M. Groen, J.J. Boon and B.H. Berrie (lecture). On copper greens in paintings.

N. Wyplosz, G. v. Rooij, J. van der Weerd, K. Groen, R. Heeren, J.J. Boon (lecture). Spatially-resolved studies of paint cross-sections by combined light microscopy, FTIR spectroscopic imaging and laser desorption mass spectrometry.

E. Hendriks and A. Wallert (lecture). Orpiment used in paintings by Maarten van Heemskerck (1498-1574); degradation of the pigment and related conservation problems

K.J. van den Berg, R. Huigen, J.J. Boon, C. van der Spek, W. Koot and G. Smith (poster). Investigation of paint samples with SIMS.

O.F. van den Brink, S. Peulvé and J.J. Boon (poster). Chemical changes in test paintings measure the environmental impact on the museum collection.

G.A. van der Doelen (poster). Molecular aspects of different artificial light ageing techniques as applied to dammar resin.

A. Wallert (poster). Auripigment, Kings-yellow and Rus-yellow; deterioration and polymorphism of arsenic sulfide pigments.

Informal ICOM-CC Interim Meeting of the working group Theory and History of conservation-restoration, Amsterdam, October 10, 1998

M.M. te Marvelde (lecture). Research on the effects of the wax/resin lining (observations on paintings related to the written sources and scientific investigation).

Fifth international conference and euroconference on optics within life sciences, Crete, 13-16 October, 1998

R.M.A. Heeren, N. Wyplosz, A. Wallert and J.J. Boon (lecture). The analytical use of laser desorption techniques in paintings research.

R.M.A. Heeren, J. van der Weerd and J.J. Boon (poster). FTIR imaging spectroscopy for organic surface analysis

Infrarood discussie groep, Philips Natlab, Eindhoven, 5 November, 1998

R.M.A. Heeren (invited lecture). FTIR imaging of embedded paint cross-sections

Chemie anders, Chemieconferentie, Woudschoten, November 6-7, 1998

J.J. Boon (invited lecture). Chemische veranderingsprocessen in schilderijen.

Recent developments in Secondary Ion Mass Spectrometry (SIMS). Symposium NEVAC & BELVAC, Eindhoven, 13 Nov. 1998

K.J. van den Berg, R. Huigen, R.M.A. Heeren, J.J. Boon, C. van der Spek and W. Koot (lecture). Investigation of paint samples of Old Masters with SIMS.

Bio-Rad FTIR imaging workshop, Krefeld, Germany, November 18, 1998

R.M.A. Heeren (invited lecture). The application of FTIR imaging in paintings research.

Dyes in History and Archeology 17, National Maritime Museum, Greenwich, United Kingdom, November 26-27, 1998

N. Wyplosz (lecture). Analysis of natural organic pigments by laser desorption mass spectrometry.

Beelden uit het Atelier. Historisch onderzoek naar praktijk en theorie van de kunstproductie. Onderzoekschool Kunstgeschiedenis, Utrecht, 11 December 1998

R. Hoppenbrouwers and R. Jongasma (lecture). Onderzoek en restauratie van de Oranjezaal Huis ten Bosch.

A. Wallert (lecture). Wat er was en wat er is. Kleur op 17e-eeuwse Nederlandse stillevens.

Tate Gallery and Courtauld Institute of Art, London, UK, February 15, 1999

K.J. van den Berg, G.A. van der Doelen, J. van den Berg, I van der Werf, J.J. Boon (lecture). Ageing phenomena in resin, oil media, and varnishes.

Research Laboratory for Archaeology & the History of Art, Oxford University, UK, February 18, 1999

K.J. van den Berg, G.A. van der Doelen, J.D.J. van den Berg, I van der Werf, J.J. Boon (lecture). Ageing phenomena in resin, oil media, and varnishes.

Dutch paintings. Pittcon '99, Orlando, Florida, USA, March 7-12, 1999

R.M.A. Heeren (lecture). FTIR-Imaging of paint cross-sections from 17-19th century.

NVMS (Dutch Society for Mass Spectrometry) meeting, Ede, March 17-18, 1999

J.J. Boon (lecture). Mass spectrometric examination of paintings.

N. Wyplosz, M.C. Duursma, R.M.A. Heeren, J.J. Boon (poster). MS/MS analysis of flavonoids by laser desorption and ionization – ion trap mass spectrometry.

J. van der Weerd, R.M.A. Heeren, N. Wyplosz, J.J. Boon, P. Noble, J. Wadum (poster). Chemical imaging of 'The anatomy lesson of dr. Nicolaes Tulp'.

O.F. van den Brink, G.B. Eijkel, J.J. Boon (poster). Measuring the quality of the museum environment.

European Commission workshop "Advantages and challenges of the modern technology for the protection of the cultural heritage", Venice, Italy, May 24-26, 1999

O.F. van den Brink, G.B. Eijkel, J.J. Boon (lecture). Mass spectrometric studies of the quality

of the museum environment using egg tempera test paintings as dosimeters.

47th ASMS conference, Dallas, Texas, USA, June 13-17, 1999

N. Wyplosz, M. Duursma, R. Heeren and J.J. Boon (poster). MS/MS analysis of flavonoids by laser desorption and ionization – ion trap mass spectrometry.

R.M.A. Heeren, J. van der Weerd, N. Wyplosz, J.J. Boon (poster). Chemical imaging of 'The anatomy lesson of dr. Nicolaes Tulp'.

J. van der Weerd, N. Wyplosz, P. Noble, J. Wadum, S. Bryan, A. Belu, J.J. Boon, R.M.A. Heeren (lecture). Mass spectrometric examination of the surface of an embedded paint cross-section.

J.J. Boon, G.M. Languri, J.H. Townsend, R. Jones (lecture). Direct temperature resolved mass spectrometric studies of paintings by Sir Joshua Reynolds (1723-1792).

Symposium 'The causes of cracks in paintings and the effect of craquelure upon the perception of the pictorial image', SRAL Bonnefantenmuseum, Maastricht, 14-15 June 1999

M.M. te Marvelde (lecture). 'The effects of wax-resin lining on paintings and the problem of dealing with the altered pictorial image'.

Joint Meeting of the Phytochemical Societies, Amsterdam, July 26-30, 1999

J.J. Boon (lecture). Paintings and natural colors. 2000 years of natural products research – past present, future.

12th Triennial Meeting ICOM-CC, Lyon 29 August-3 September, 1999.

J.D.J. van den Berg (lecture). Chemical changes in curing and ageing oil paints.

K.J. van den Berg, J. van der Horst, J.J. Boon (lecture). Mass spectrometric methodology for the recognition of copals in aged resin/oil paints and varnishes.

O.F. van den Brink, M. van Eikema Hommes, J.J. Boon (poster). The discoloration of indigo in a paint matrix.

R.M.A. Heeren, J.J. Boon, P. Noble, J. Wadum (lecture). Integrating imaging FTIR and secondary ion mass spectrometry for the analysis of embedded paint cross-sections.

R. Jones, J.H. Townsend, J.J. Boon (lecture). A technical assessment of eight portraits by Reynolds being considered for conservation treatment.

M. Kokkori, A. Phenix, J.J. Boon (lecture). Solvent extraction of organic compounds from oleo-resinous 'megilp' paint media.

G.M. Languri, J. van der Horst, J.J. Boon (poster). On the composition of 'Asphalt' and 'Mummy' in the early 19th century paint materials collection of Michiel Hafkenscheid.

M.M. te Marvelde (lecture). "Research into the history of conservation-restoration: Remarks on relevance and method".

Colloque XIII pour l'etude de la technologie et du dessin sous-jacent dans la peinture, Brugge, Belgium, September 15-17, 1999

J. van der Weerd, M. Geldof, R.M.A. Heeren and J.R.J. van Asperen de Boer (lecture). A European 640 x 486 PtSi camera for infrared reflectography.

A. Wallert (lecture). The holy kinship: problems of attribution.

A. Wallert (lecture). Two out of many: a pair of dyptich panels in the Rijksmuseum.

Genootschap Wessel Knoop, Arnhem, October 12, 1999

J.J.Boon (lecture). Schilderij-onderzoek.

Eastern Analytical Symposium. Session analytical investigations for art and archaeology: applications in mass spectrometry, Somerset, NJ, USA, November 14-19, 1999

J.J. Boon, K.J. van den Berg, R.M.A. Heeren (lecture). Mass spectrometric studies of paintings in MOLART.

Getty Conservation Institute, Los Angeles, CA, USA, December 3-4, 1999

J.J. Boon (lecture). Molecular aspects of ageing of natural resin varnish, egg tempera and oil paint.

J.J. Boon (lecture). Direct probe and imaging mass spectrometry as new tools for technical studies of paintings.

Bio-Rad FTIR imaging workshop, Krefeld, Germany, December 7, 1999

R.M.A. Heeren (lecture). FTIR imaging of cross-sections from 17th-19th century paintings.

Fourth Infrared Users' Group Meeting (IRUG4) in Conservation Science, Bonnefanten Museum, Maastricht, February 14-18, 2000

O.F. van den Brink (poster). Chemical changes in test paintings measure the environmental impact on the museum collection.

O.F. van den Brink (poster). Discoloration of indigo in the paint matrix.

M. Geldof (poster). Microscopic studies of paint cross sections.

W.J. Muizebelt (lecture). Crosslinking of linseed oil paints studied with FTIR.

J. van der Weerd (lecture). FTIR imaging and UVVIS analyses of intermediate layers in embedded paint cross-sections.

N. Wyplosz (poster). MS/MS analysis of flavonoids by laser desorption and ionisation- Ion trap mass spectrometry.

Eidgenössische Technische Hochschule, Zürich, February 17, 2000, Center of Excellence in Analytical Chemistry.

J.J. Boon (lecture). Chemical studies on paintings and painting materials.

Inspectie Cultuurbezit (National Agency for the Protection of Cultural Heritage), Den Haag, February 23, 2000

J.J. Boon (lecture). Nieuwe inzichten in de veroudering van schilderijen.

Bio-Rad FTIR Seminar, Kasteel Ophemert, March 2, 2000

J. van der Weerd (lecture). FTIR imaging and UVVIS analyses of intermediate layers in embedded paint cross-section.

Pyrolysis 2000, 14th International Symposium on Analytical and Applied Pyrolysis, April 2-6, 2000

J.J.Boon and O.F. van den Brink (lecture). Dosimetry of the display environment of fine art: monitoring of the early chemical changes in experimental egg tempera paintings by analytical pyrolysis MS and related MS techniques.

G.M. Languri (poster). Analytical Pyrolysis MS and GC/MS as analytical tools to investigate geo-materials in early nineteenth-century paint materials and their effect on paintings.

Oranjezaal Discussiedag (SRAL/Onderzoeksschool Kunstgeschiedenis), Rijswijk, April 7, 2000

G.A. van der Doelen and J.J. Boon (lecture). Chemical research within the Oranjezaal project.

The Croucher Advanced Study Institute Workshop on the Frontiers in Surface Science and their Novel Applications, The Chinese University of Hong Kong, Hong Kong, China, May 15-20, 2000

O.F. van den Brink, G.B. Eijkel and J.J. Boon (poster). Chemical changes in test paintings measure the environmental impact on the museum collection.

O.F. van den Brink, S. Peulvé and J.J. Boon (poster). Early changes in the chemical composition of tempera paints.

O.F. van den Brink, M.H. van Eikema Hommes and J.J. Boon (poster). Discoloration of indigo in the paint matrix.

- O.F. van den Brink, P. B. O'Connor, M.C. Duursma, R.M.A. Heeren and J.J. Boon (poster). Analysis of Egg Lipids and their Oxidation Products by MALDI-FT-ICR-MS(MS).
- G.A. van der Doelen, K.J. van den Berg and J.J. Boon (poster). Cross-linking processes in Dammar and Mastic varnishes.
- J.J. Boon (lecture). Traditional European painting methods.
- J.J. Boon (lecture). Key issues in maintenance of paintings.
- J.J. Boon (lecture). Molecular aspects of aging of painting media: oil paint and varnish.
- J.J. Boon (lecture). Direct probe and imaging mass spectrometry as new tools for technical studies of paintings.
- 48th ASMS Conference on Mass Spectrometry and Allied Topics, Long Beach, CA, USA, June 11-15, 2000**
- O.F. van den Brink (poster). An ESI-FTMSMS study of natural and photo-oxidized egg glycerolipids.
- 26th International Conference in Organic Coatings Science and Technology, Athens, Greece, July 3-7, 2000**
- W.J. Muizebelt (poster). Monitoring the effects of traditional nineteenth-century additives on the chemical drying of oil paint by mass spectrometry.
- Indoor Air Quality 2000, Oxford, United Kingdom, July 10-12, 2000,**
- O.F. van den Brink (lecture). Paint-based dosimetry of the museum environment.
- The painters palette in the 16th and 17th Century: pigment preparation and painting technology' seminar at the Amsterdam-Maastricht Summer University, July 17-18, 2000**
- A. Wallert (lecture). 'The technical examination of paintings: problems of chronology and authenticity'.
- 15th International Mass Spectrometry Conference, Barcelona, Spain, August 27-September 1, 2000**
- O.F. van den Brink, G.B. Eijkel and J.J. Boon (lecture). Mass spectrometry of mock paintings for environmental monitoring of museums
- O.F. van den Brink, M.C. Duursma, J.J. Boon and R.M.A. Heeren (poster). An ESI-FTMSMS study of the structure of photo-oxidised egg glycerolipids
- N. Wyplosz, M.C. Duursma, J.J. Boon and R.M.A. Heeren (poster). Spatially-resolved TOF-MS analysis of paint materials and easel paintings samples (poster).
- J.D.J. van den Berg, N. D. Vermist and J.J. Boon (poster). MALDI-TOF-MS and ESI-FTMS of oxidised triacylglycerols and oligomers in traditionally prepared linseed oils used for oil painting
- G.M. Languri, J. van der Horst, W.J. Muizebelt, R.M.A. Heeren and J.J. Boon (poster). Monitoring the effects of traditional nineteenth-century additives on the chemical drying of oil paint by mass spectrometry.
- NWO Conference 'Bessensap', Amsterdam, September 25, 2000**
- G.A. van der Doelen and J.J. Boon (lecture). Verval van oude kunst.
- Eastern Analytical Symposium, Atlantic City, NJ, October 30-November 3, 2000**
- R.M.A. Heeren (lecture). Controlled laser cleaning of easel paintings: fundamentals and application
- Fall meeting of the NVMS (Dutch Society for Mass Spectrometry), Groningen, December 8, 2000**
- O.F. van den Brink, G.B. Eijkel and J.J. Boon (lecture). Mass spectrometry of mock paintings for environmental monitoring of museums.
- O.F. van den Brink, M.C. Duursma, J.J. Boon and R.M.A. Heeren (poster). An ESI-FTMSMS study of the structure of photo-oxidised egg glycerolipids.
- G. Languri, J. van der Horst, W.J. Muizebelt, R.M.A. Heeren and J.J. Boon (poster). Monitoring the effects of traditional nineteenth-century additives on the chemical drying of oil paint by mass spectrometry
- J.D.J. van den Berg, N.D. Vermist and J.J. Boon (poster). MALDI-TOF-MS and ESI-FTMS of oxidised triacylglycerols and oligomers in traditionally prepared linseed oils used for oil painting.
- Sanibel Conference on informatics and mass spectrometry, Sanibel, Florida, USA, January 19-22, 2001**
- O.F. van den Brink, G.B. Eijkel, J.J. Boon (poster). Discriminant analysis and mass spectrometry of paint-based dosimeters for environmental monitoring of museums.
- Dwarsverbandcommissie, Rijksmuseum Amsterdam, January 26, 2001**
- J. van der Weerd (lecture). Ernstig zieke schilderijen.

**Queens University, Kingston, Canada,
14 February, 2001**

M.M. te Marvelde (lecture). Conservation history and what it means for conservation.

ICOMCC Interim meeting, modern materials workshop, Cologne, Germany, March, 12-14, 2001

J.J. Boon, T.S. Learner, N.Wyplosz, R.M.A. Heeren (lecture). Mass spectrometric identification of pigments and media in modern paints

49th ASMS conference on Mass spectrometry and allied topics, Illinois, USA, May 27-31, 2001

O.F. van den Brink, M. Duursma, S. Oonk, G.B. Eijkel, J.J. Boon, R.M.A. Heeren (poster). Probing changes in the lipid composition on the surface of laser treated artist paints by MALDI-MS

J.J. Boon, N. Wyplosz, B. Marino, M. Duursma, J. van der Horst, T. Learner (lecture). Mass spectrometric identification of pigments and media in modern paintings

Study day for ICN students, AMOLF, Amsterdam, May 29, 2001

J. van der Weerd, S. Luxembourg (lecture). Imaging techniques in the analysis of paint.

COST Action G8, Non-destructive Analysis and Testing of Museum Objects, Ghent, Belgium 25 January 2002.

K. Keune and J.J. Boon (lecture). Imaging mass spectrometry and microspectroscopy of paintings cross-sections.

Section Histoire de l'Art et d'Archéologie, Université Libre de Bruxelles, Brussels, Belgium, July 12, 2001.

R. Boitelle (lecture). Descending Cows and a Red Tree- Results of research and their relation to conservation and restoration methods. Examination of two XIXth Century Paintings by Rousseau and Redon.

1st International Conference on Advanced Vibrational Spectroscopy, Turku, Finland, August 19-24, 2001

R.M.A. Heeren, J. van der Weerd, J.J. Boon (lecture). FTIR imaging in biomedicine and culture.

J. van der Weerd, M. Geldof, R.M.A. Heeren, J.J. Boon (poster): Ongoing changes in old master's paint studied by imaging FTIR.

Seventeenth-century paints; pigments and stratigraphy lecture series, Amsterdam-Maastricht Summer University, Den Haag, August 20, 2001

A. Wallert (lecture). Developments in the Study of 17th Century Dutch Art.

First European Coatings Conference, Symposium on film formation, Zürich, Switzerland, September 6, 2001

W.J. Muizebelt (lecture). Crosslink mechanisms of alkyd resins studied using model compounds.

Deterioration of artists' paints: effects and analysis, British museum, London, United Kingdom, September 10-11, 2001

K.J. van den Berg, M. Geldof, S. de Groot and H. van Keulen (lecture). An analytical chemistry and microscopy study of seven darkened Dutch late 19th-and 20th-Century paintings

J.J. Boon, K. Keune, J. van der Weerd, P. Noble (lecture). Chemical changes in old master paintings: dissolution, metal soap formation, and remineralisation processes in lead pigmented paint layers of 17th C paintings.

R. Boitelle, K.J. van den Berg, M. Geldof, G. Languri (lecture). Descending into the details of Th.Rousseau's "La descente des vaches" (Museum Mesdag, The Hague) – Technical Research of a darkening painting.

G. Languri, J. van den Berg, J.J.Boon (lecture). Effects of additions of mastic, Copiava balsam, asphalt or earth pigments on the chemical drying of oil paint.

Conference 'Past Practice-Future Prospects', The British Museum, London, United Kingdom, September 12, 2001.

M.M. te Marvelde (lecture). How Dutch is 'The Dutch Method? A History of Wax-Resin Lining in its International Context.

FOM/Symposium, Amsterdam, September 21, 2001

B. Marino, J.J. Boon, J. van der Horst, T. Learner (poster). DTMS study of 20th century paints.

"La Sapienza", Università degli Studi di Roma, Roma, Italy, September 22, 2001

B. Marino (lecture). Imaging and Structural Analysis of Cross Sections of Paint Concorso a n°147 Borse di Studio per Corsi di Perfezionamento all'Estero.

XVI Congresso Nazionale di Chimica Analytica, Chimica Analytica e Scienze di Mare Portonovo, Ancona, Italy, September 24-28, 2001

J.J. Boon (lecture). Natural and destructive ageing processes in old master paintings.

**KNCV Macromoleculaire Sectie,
Wageningen, October 3, 2001**

W.J. Muizebelt, J. van der Weerd, M. Duursma, J.J.Boon (lecture). Oxidation and crosslinking of unsaturated fatty acids in alkyd resins and linseed oil.

**Instituut Collectie Nederland (ICN),
October 10, 2001**

J.J. Boon (lecture). Mass spectrometry and imaging studies of modern artist's materials and cross-sections.

**6th European workshop on Fourier
transform ion cyclotron resonance mass
spectrometry, Kerkrade, October 16-19, 2001**

O.F. van de Brink (lecture). ESI-FTMS(MS) of oxidized egg glycerolipids.

**Symposium on Technical art history,
RKD, The Hague, October 29-30, 2001**

L.B.J. Speleers (lecture). "Het valdt in t groodt ongelijk moeyelycker als gegist hadde...": making the large-scale paintings for the Oranjezaal.

**Allied Circle, Doelen Hotel Amsterdam,
October 31, 2001**

J.J. Boon (lecture). Scientific examination of paintings.

**20th Annual Meeting of Dyes in history
and archaeology, Instituut Collectie Nederland
(ICN), Amsterdam, November 1-2, 2001**

M. van Eikema Hommes (lecture). Fading of indigo in 17th century oil paintings.

B. Marino, J. van der Horst, M.C. Duursma, N. Wyplosz, J.J. Boon, T. Learner (poster). Mass spectrometric studies of 20th C organic pigments and their paints.

**Infrarood en Raman Discussiegroep,
RIVM, Bilthoven, November 15, 2001**

J. van der Weerd (lecture). Extreem verouderde verf.

**Rijksmuseum Twente, Enschede,
November 22, 2001**

R.M.A. Heeren (lecture). Controlled laser cleaning of paintings: fundamentals and applications, Symposium "Het gebruik van lasers bij schilderijrestauratie.

**International Symposium on Analytical
Pyrolysis of Polymers, Nagoya, Japan, January
24-25, 2002**

J.J. Boon and T. Learner (lecture). Analytical mass spectrometry of artist's acrylic emulsion paints by direct temperature resolved mass spectrometry and laser desorption ionisation mass spectrometry.

SRAL, Maastricht, March 14, 2002.

M.M. Marvelde (lecture). History of wax-resin lining in Holland; Development of ideas, materials and techniques, research done in the past, research in MOLART today.

**SIMS Conference, University of Siena,
Department of Chemical and Biosystem
Sciences and Technologies, Siena, Italy, March
21-22, 2002**

K. Keune, R. Heeren and J.J. Boon (lecture). Imaging mass spectrometry and microspectroscopy of paintings cross-sections.

**Colloquium Paint Research Group,
Department of Chemistry, Technical
University of Eindhoven, Eindhoven. April 9,
2002**

J.J. Boon (lecture) Natural and destructive ageing processes in oil paint.

**Center for Art Diagnosis and
Hagiography, Ormylia, Greece, May 16, 2002**

J.J. Boon (lecture). MOLART painting studies at AMOLF.

**Symposium on Smalt, Centraal
Museum, Utrecht. May 30, 2002**

J.J. Boon and K. Keune (lecture). Chemical changes in smalt and lead white containing layers in Old Master paintings.

**ART 2002, Antwerp, Belgium, June 2-6,
2002**

K. Keune, P. Noble and J. J. Boon (lecture). Chemical changes in lead-pigmented oil paints: on the early stage of formation of protrusions.

G. Languri and J.J. Boon (poster). Mass spectrometry as a microanalytical method to study Kassel Earth (Vandyke brown) pigments in oil paint.

K.J. van den Berg, J. Ossebaar and H. van Keulen (lecture). Analytical of copal resins in 19th Century oil paints and resin/oil varnishes.

**Onderzoeksschool Kunstgeschiedenis,
Utrecht, 5 July, 2002**

K.J. van den Berg (lecture). Natuurwetenschappelijk onderzoek in het 19de Eeuwproject (ICN-musea-MOLART). En verder?

**Getty Conservation Institute, Los
Angeles, USA, July 18, 2002**

K.J. van den Berg (lecture). Ageing phenomena in resin and oil media and varnishes.

**Secondary Ion Mass Spectrometry
Europe 2002, Münster, 15-17 September, 2002**

K. Keune, P. Noble and J.J. Boon. (poster).

Secondary Ion Mass Spectrometry on Paint Cross-Sections.

KNCV Najaarscongres 2002: Chemie en Schilderkunst, Den Haag, 19 September 2002

J.J. Boon. (lecture). Schilderijen onderzoek: een uitdaging voor chemici.

L. Carlyle (lecture). Historically accurate reconstructions: a tool in the history of painting.

13th Triennial Meeting of ICOMCC, Rio de Janeiro, 23-28 September 2002

J.J. Boon, K. Keune and T. Learner (lecture). Identification of pigments and media from a paint cross section by direct mass spectrometry and high-resolution imaging mass spectrometric and microspectroscopic techniques

L. Carlyle, N. Binnie and Elzbieta Kaminska (lecture). The Yellowing/bleaching of oil paintings and oil paint samples, including the effect of oil processing, driers and mediums on the colour of lead white paint. In: ICOM, Committee for Conservation 13th Triennial Meeting, Rio de Janeiro, 22-28 September 2002, p.328-337

M. Odlyha, O. F. van den Brink, J. J. Boon, M. Bacci, N. S. Cohen (lecture). Damage assessment of museum environments using paint based dosimetry.

J. J. Boon, J. van der Weerd, K. Keune, P. Noble, and J. Wadum (lecture). Mechanical and chemical changes in Old Master paintings: dissolution, metal soap formation and remineralization processes in lead pigmented ground/intermediate paint layers of 17th century paintings.

ICOM-CC Rio de Janeiro, Brazil, September 26, 2002

K.J. van den Berg, M. Geldof, S. de Groot and H. van Keulen(lecture). Darkening and surface degradation in 19th-and early 20th century paintings-an analytical study.

Eastern Analytical Symposium, Somerset NJ, USA, November 18, 2002

K.J. van den Berg, J.J. Boon, J.D.J. van den Berg and H. van Keulen (lecture). The chemistry of oil paint media and resin-oil varnishes: Analytical implications.

Art Institute of Chicago, Chicago, USA. November 26, 2002

J.J.Boon. (lecture). Phenomenology, chemistry and dynamics of metal soap aggregates and protrusions in 17th and 19th century paintings.

Presentation for the Courtauld Institute students, AMOLF, Amsterdam. 12 March, 2002

J.J.Boon (lecture). Methods of analysis and new ideas on the composition of oil paint.

PV lezing for Department of Colloid Chemistry, Universiteit Utrecht, Uithof, May 31, 2002

J.J.Boon (lecture). Schilderij onderzoek als uitdaging voor chemici.

Part 5: Contributions presented at MOLART meetings

MOLART dag, 10 January 1997, Van Gogh Museum, Amsterdam

J.J. Boon. Onderzoeklijnen binnen MOLART.

K. J. van den Berg & R.M.A. Heeren. Nieuwe mogelijkheden voor micro-analytisch onderzoek van schilderijen.

A. Phenix. MOLART and conservation in Holland seen from an international perspective.

J. Wadum. Restauratie en MOLART

J.P. Filedt Kok. Collectiebeheer en MOLART

R. Hoppenbrouwers. Interdisciplinaire samenwerking, theorie en praktijk

S. Schmitt, I. van der Werf & M.M. te Marvelde. Pettenkofers regeneratiemethode en het gebruik van copaivabalsem in Nederland

MOLART workshop, 15-16 January 1998, Amsterdam

G.A. van der Doelen. Ageing of triterpenoid varnishes.

K.J. van den Berg. Diterpenoid resins in paintings.

K.J van den Berg & J. van der Horst. Resin Macromolecules.

J.D.J. van den Berg. Analytical chemistry of oil paint.

A.Phenix. Oil paint models and cleaning processes.

K. Sutherland. Solvent-extractable components in oil paint films.

R. Heeren. Infrared imaging in paintings research.

G.J. van Rooij. Spatially- resolved laser desorption mass spectrometry.

N. Wyplosz. Laser desorption mass spectrometry of pigments.

E. Hendriks & M. van Eikema Hommes. Discoloration of indigo used in easel paintings.

R. Boitelle. Asphalt Part 1: Asphalt and drying problems in paintings.

G. Languri & J.J. Boon. Asphalt Part 2: Preliminary analytical results.

A. Wallert. Aspects of ageing in arsenic sulphides.

O.F. van den Brink. Dosimetry of the museum environment: environmental effects on the chemistry of paintings.

R. Hoppenbrouwers. Reconstruction of historical paints and accelerated ageing.

M. te Marvelde. The role of conservation-restoration historical research in the Molart project and its relevance for present-day maintenance of paintings: the Oranjezaal in Huis ten Bosch.

S. Schmitt. Effects of regeneration methods on paint layers in the Pettenkofer process: solvents and copaiva balsam

K. Groen, H. de Boer, M.H. van Eikema Hommes & R.M.A. Heeren. The MICRODAT database: exploring digital storage and retrieval of paint cross-sections and related microscopic images.

MOLART-day for Dutch Restorers, Rijksmuseum, 28 October 1998, Amsterdam

(Lectures)

J.J. Boon. De MOLART formule.

G.A. van der Doelen. Moleculair inzicht in de verouderingsprocessen in dammar en mastiek.

J.D.J. van den Berg. Verharde verf: hoe komt het dat olieverf drastisch van eigenschappen verandert bij veroudering?

R.M.A. Heeren. Nieuw ontwikkelde microscopische technieken om dwarsdoorsnedes te kunnen bestuderen.

A. van Grevenstein, Ruth Jongma & René Hoppenbrouwers. Restauratieproject Oranjezaal.

A. Wallert. Restauratieproject stilleven.

(Posters)

J.D.J. van den Berg, K.J. van den Berg & J.J. Boon. Analysis of cured and traditional linseed oil paints

G.A. van der Doelen, K.J. van den Berg & J.J. Boon. Cross-linking processes in dammar and mastic varnishes

G.A. van der Doelen, K.J. van den Berg & J.J. Boon. Molecular aspects of different artificial light ageing techniques as applied to dammar resin

I.D. van der Werf, S. Schmitt, K.J. van den Berg & J.J. Boon. Copaiba balsam used in the Pettenkofer process and as an additive in 19th century painting techniques: studies of its effect on paint layers

G. Languri, K.J. van den Berg & J.J. Boon. The chemistry of asphalt and its effect on oil paint

O.F. van den Brink, S. Peulvé, M. Duursma & J.J. Boon. Early changes in tempera paint

O.F. van den Brink, S. Peulvé & J.J. Boon. Chemical changes in test paintings measure the environmental impact on the museum collection

K.J. van den Berg, L. Spetter & J.J. Boon. Degree of Oxidation of Diterpenoid Pinaceae Resins used in paintings

R. Huigen, K.J. van den Berg, J.J. Boon, C. van der Spek, W. Koot, G. Smith. Investigation of paint samples with SIMS

N. Wyplosz, A. Wallert, R.M.A. Heeren, & J.J. Boon. Analysis of natural organic pigments by ITMS, FT-MS and TOF-MS

R.M.A. Heeren & J.R.J. van Asperen de Boer. IRR imaging of 'the Lamentation of Christ' by Jan van Scorel with a new 640x486 pixel PtSi array camera

N. Wyplosz, R.M.A. Heeren, & J. J. Boon. Spatially resolved Laser Desorption Mass Spectrometry

J. van der Weerd, R.M.A. Heeren, & J.J. Boon. FTIR imaging spectroscopy for organic surface analysis

M.M. te Marvelde & K.J. van den Berg. An unusual pastiglia technique in the 18th Century

M. Geldof, J. van der Weerd, N. Wyplosz, R. Koper, R.M.A. Heeren & J.J. Boon. A new polishing procedure for embedded paint samples

Picture credits

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Abbreviations

AMOLF	FOM Institute for Atomic and Molecular Physics
DTMS	Direct Temperature resolved Mass Spectrometry
ESI	Electro Spray Ionisation
FOM	Fundamenteel Onderzoek der Materie
FT-IR	Fourier Transform Infrared Spectroscopy
FTMS	Fourier Transform Mass Spectrometry
GC/MS	Gas Chromatography/Mass Spectrometry
HPLC	High Performance Liquid Chromatography
ICN	Instituut Collectie Nederland
IR	Infrared
ITMS	Ion Trap Mass Spectrometry
LD	Laser Desorption
(M)	Financially supported by MOLART
(M-Fellow)	MOLART Fellow
MALDI	Matrix Assisted Laser Desorption Ionisation
MOLART	Molecular aspects of ageing in painted Art
NWO	Nederlandse Organisatie voor Wetenschappelijk Onderzoek
RUG	Rijksuniversiteit Groningen
SEC	Size Exclusion Chromatography
SEM / EDX	Scanning Electron Microscopy / Energy Dispersive X-ray Analyser
SIMS	Secondary Ion Mass Spectrometry
SLIM	Spatially resolved Laser induced Ion Mass Spectrometry
SRAL	Stichting Restauratie Atelier Limburg
TOF-MS	Time-of -flight Mass Spectrometry
UV	Ultra-violet
UvA	University of Amsterdam
VIS	Visible (light)
XRD	X-ray Diffraction

Contact details and websites

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MOLART Home Page (Reports, bibliographies, descriptions of projects):
www.amolf.nl/research/biomacromolecular_mass_spectrometry/molart/molart.html

On-line theses:

www.amolf.nl/publications/theses/	(v/d Weerd, v/d Berg & v/d Brink)
www.amolf.nl/publications/theses/doelen/	(v/d Doelen)