Supplementary issue paper
The technical investigation of an eighteenth-century Chinese imperial carved lacquer screen and its role in developing an appropriate conservation treatment

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This paper gives an overview of the results of a comprehensive conservation research project on an eighteenth-century Chinese carved lacquer screen. Special emphasis was put on scientific analyses of the original materials and techniques. The object’s history, previous restoration treatments, and the current condition were documented before the conservation treatment commenced. The extensive analytical investigations on a set of 24 samples, performed by means of optical and scanning electron microscopy as well as pyrolysis–gas chromatography–mass spectrometry, helped to determine the stratigraphy of the lacquer screen and to reveal the compositions of the materials used. The results and information presented here are the products of an interdisciplinary collaboration of conservators and scientists, which contributed significantly to the preparation of a conservation strategy for the screen and helped in the selection of conservation techniques and the choice of appropriate conservation materials.

Keywords: Asian lacquer, Oil-resinous varnish, Foundation, Proteinaceous binder, Pyrolysis–gas chromatography–mass spectrometry, Optical and electron microscopy, Conservation

Introduction
The subject of this study is a three-panel wooden screen from the Qianlong period (1736–1795) of the Qing dynasty (accession number 71.233). With its impressive dimensions (3.30 x 2.60 m), carved lacquer with yellow, green, and red layers on the front side and gold-painting on black lacquer on the back side (Figs 1 and 2), it was originally housed at the Imperial Hunting Palace at Nan-Haidze Park situated about 10 km outside of Beijing, China. It has been in the possession of the Weltmuseum Wien (formerly the Museum of Ethnology, Vienna) since 1902 and is ranked among the most exquisite furniture pieces in the collection. A matching throne (No. W.399:1), which originally belonged to the screen, is a central piece in the Chinese collection of the Victoria and Albert Museum, London (Strange, 1925).

The carved lacquer on the front of the screen depicts the Pan Tao Festival, to which the Queen Mother of the West (Xiwangmu) invites all immortals at the time when the peaches — the fruits of immortality — reach ripeness, 3000 years after blossoming. The peach banquet takes place in the garden and on the terraces of the palace of Xiwangmu, who can be seen in the centre of the screen with her female attendants (Fig. 3). There are different groups of immortals and deities of the Taoist pantheon, such as the famous Eight Immortals on the upper terrace, the monk Shide with his broom, riding on the three-legged toad, and the abbot Fenggan, sitting on the back of a tiger. The side panels also depict landscapes with a few figures, including three sages playing weiqi on one side and Magu, the goddess of longevity, on the other. This elaborately carved scene resonates with the painted images on the back of the screen, which feature peaches, bats, cranes, waves, rocks, and clouds, all of which convey the hope of good fortune and longevity (Bartholomew, 2006).

Condition and technical analysis
The surface of the screen had collected considerable dirt and dust over the time, particularly on the
horizontal areas of the base and in the carved parts. On the front side, a very glossy varnish had been applied, probably during later maintenance or restoration. It had dripped over the edges towards the back of the screen, which is unvarnished. Under the varnish at the front, small patches of a white and waxy substance are present, particularly in the cavities.

The black-lacquered backs of the panels showed a matt, light-damaged, and inhomogeneous surface and the gilded decorations are abraded to different degrees. There were several cracks in the lacquer caused by shrinkage of the lacquer as well as dimensional changes in the wooden structure. The cracks in the inner panels were relatively minor while those along the joins were larger, revealing the dowels and the construction in general. One long crack in the centre panel, already recorded in 1901 (Baustädter, 2013), had been repaired with animal glue and the glue had spread across the surrounding delicate carving. On the both sides, many lacquer losses were observed and a number of the fire-gilded brass or bronze fittings on the base were missing and had been replaced by painted cardboard.

The main questions posed to the scientists by the conservators concerned the identification of the binding medium of the ground layer, the composition and ingredients of the lacquer and varnish, and the identification of the white waxy substance. In addition, it was hoped to explain the cause of the grey areas on the inhomogeneous black lacquer on the back of the screen. Finally, all the pigments and metal powders, as well as the textile fibres in the ground layer, were analysed.

After a preliminary on-site examination, a set of samples was taken for the subsequent investigation (Table 1). The samples were initially examined by optical microscopy (OM) with a Zeiss Stemi 2000-C stereomicroscope. Subsequently, samples were prepared as cross-sections by embedding small particles in epoxy resin and polished after curing. These sections were used for microscopic investigation and histochemical staining (Jütte, 1991; Schramm & Hering, 1995) to identify and map the presence of binders in the multi-layered structure.

The microscopic studies were carried out under visible light using either polarized light or in the
dark-field mode, and then using ultraviolet fluorescence. Where necessary the microscopic investigations were complemented by studies using scanning electron microscopy with energy-dispersive X-ray (SEM–EDX) analysis to identify the inorganic components of the ground layers and decorative elements. Another set of samples was used directly for gas chromatography–mass spectrometry (GC–MS) to detect lipids, resins, and proteinaceous binding media. Thermally assisted hydrolysis and methylation coupled with pyrolysis–gas chromatography–mass spectrometry (THM–Py–GC–MS) was applied to identify the different Asian lacquers. The presence of oils, resins, proteins, or polysaccharides was determined through wet microchemical tests on those samples that were not analysed by OM. To detect the presence of starch and blood as a binder in the ground layers, spot tests were used for material characterization.

The latter analysis (for the presence of blood) is based on an enzymatic reaction of peroxidase: addition of H$_2$O$_2$ causes haemoglobin to decompose into water and oxygen. The latter oxidizes benzidine (1,1’-biphenyl-4,4’-diamine), which becomes visible through a change of colour to a bluish-green shade (Odegaard et al., 2005). The GC–MS analytical procedure for lipids is based on the transesterification of fatty acids and the determination of their relative ratios to identify particular lipids, while the analytical method for resinous binding media is based on the esterification of resinous acids followed by the identification of particular resin acid methyl esters. The analytical procedure for proteinaceous materials is based on an acidic hydrolysis of proteins to liberate amino acids, followed by the derivatization and quantification of the silyl derivatives of the amino acids (Pitthard et al., 2010). THM–Py–GC–MS using tetramethylammonium hydroxide (TMAH) as the methylating agent was performed on two different systems using either a Curie-point pyromat in Vienna or — through a collaboration with colleagues at the Getty Conservation Institute — a double-shot pyrolyzer in Los Angeles. The analyses of western lacquers based on resinous or oleo-resinous varnishes were performed by means of GC–MS while pyrolysis (Py–GC–MS) enabled the various chemical compositions of aged Asian lacquers to be differentiated in order to trace the trade and production of these lacquers. Full
details of the experimental procedures and the conditions for the analyses are given in the experimental appendix.

**Results and discussion**

*Method of manufacture and stratigraphy of lacquers*

The individual parts of the screen are connected by a system of mortise and tenon joints. In addition to the large central panel and two side panels, there is a base that consists of three parts, three cloud-like upper parts that rest on the main panels, and two small decorative elements at the sides.

The base is made from softwood and the panels and upper parts are constructed with an outer frame that holds an inner panel with the help of iron nails and dowels. A rather coarse and thick grey ground layer into which textile fibres have been mixed was applied to the panels. In certain areas (e.g. on the exposed corners and at the joints), fabric was also applied under the lacquer.

On the front of the screen numerous light brown (originally yellow) lacquer layers were applied, followed by a series of dark brown (originally green) and finally red lacquer layers. The total thickness of all the lacquer layers (of which there are around 50) is c.4 mm, as could be seen from investigations of the stratigraphy using optical microscopy and SEM.

Stratigraphic investigations by optical microscopy and SEM unveiled a multi-layered structure that began with grey ground layers that contained ochres (earth pigments), followed by the main lacquer layers, which were pigmented with carbon blacks for the black lacquer parts, with cinnabar in the case of red lacquers, orpiment in the yellow layers, and with orpiment and indigo to produce the green areas.

The assumption that indigo was used as the blue pigment in the green layer is based on the absence of significant quantities of any elements in the EDX analysis that might indicate any other type of pigment, for example azurite, smalt, or ultramarine. This assumption is supported by several studies that report the use of a mixture of orpiment and indigo to produce a green colour in Asian lacquerware (Sheasby, 1991; Kopplin, 2002; Ward, 2008). The use of carbon black as a colourant in the black lacquers is also known (Sheasby, 1991), although the SEM–EDX analysis of these layers did not give any further evidence of the type of carbon black used.

On the painted back of the screen, silver, gold, and silver–gold alloys were detected, but some of the design elements were also found to be pigmented with orpiment and ochre. All the pigments and metal foils and powders were identified by SEM–EDX.

During the manufacturing process, the layers were carved with the relief design (see Figs 4 and 5). The
The back side of the screen is mainly lacquered in black and decorated with a design in different shades of gold made from very fine sprinkled gold powders and gold leaf. The frames of the back faces are lacquered red over black and decorated with a painted design in black and yellow (Figs 6 and 7).

In addition, all textile fibres found scattered within the ground layers, as well as those in the fabrics used to strengthen the corners of the screen, were identified by OM as ramie (Chinese nettle) fibres.

**Identification of binding media**

Binding media analyses of the uppermost transparent coatings as well as remnants of waxy areas directly beneath were conducted using GC–MS. Pine resin (*Pinus* species) and traces of shellac were detected in

| Sample number | Sample description | Analytical methods | Results/composition |
|---------------|--------------------|--------------------|---------------------|
| P01           | Proper right panel; milky coating | GC–MS | Coating: ibota wax |
| P02           | Proper left panel; milky coating | GC–MS | Coating: ibota wax |
| P03           | Proper left panel; milky coating | GC–MS | Coating: ibota wax |
| P04           | Proper right panel; multi-layered sample | OM on cross-section, SEM | Pigments: ochres, organic layer, orpiment and cinnabar |
|               |                    | Microchemical test for blood | Negative |
| P05           | Central panel; multi-layered sample | OM on cross-section, SEM | Pigments: earth pigments, indigo, orpiment and cinnabar |
|               |                    | Microchemical test for blood | Positive |
| P06           | Proper left panel; multi-layered sample | Cross-section | Similar layer structure as in top layers of P04 |
| P07           | Proper right panel; ground layer | GC–MS | Ground: blood (?) |
| P08           | Central panel, base; coating | GC–MS | Upper coating based on drying oil, shellac, and pine resin |
| P09           | Proper right panel; ground layer, fibre | OM | Ramie |
| P10           | Proper left panel, reverse side; ground under lacquer | OM on cross-section, SEM | Pigments: earth pigments, organic layer, silver-gold foil, orpiment, and gold foil |
| P11           | Proper left panel, reverse side; retouching | OM on cross-section, SEM | Pigments: some earth pigments and cinnabar |
| P12           | Proper left panel; reverse | OM on cross-section, SEM | Pigments: earth pigments (organic layer), ochres, orpiment, lead white, and cinnabar |
| P14           | Proper left panel, front side; multi-layered sample | OM on cross-section | Stratigraphy |
| P15           | Proper left panel, front side; multi-layered sample | OM on cross-section | Stratigraphy |
| P16           | Upper part; multi-layered sample | OM on cross-section | Stratigraphy |
| P17           | Retouching | GC–MS | Beeswax |
| P18           | Proper left panel, reverse side; black lacquer | OM on cross-section | Stratigraphy |
| P19           | Proper left panel, reverse side; upper coating over black lacquer | GC–MS | Drying oil (?) |
| P20           | Proper left panel, reverse side; upper coating over black lacquer | GC–MS | Drying oil (?) |
| P21           | Central panel; grey hazy film | Py–GC–MS | Urushi not detected |
| P22           | Proper right panel; grey hazy film | Py–GC–MS | Urushi not detected |
| P23           | Proper right panel; red lacquer | Py–GC–MS | Urushi, cedar oil, and drying oil |
| P24           | Proper right panel; black lacquer | Py–GC–MS | Urushi and drying oil |

Figure 4  Structure at the front of the screen, showing multiple layers of yellow, green, and red lacquer applied over a grey ground layer.
the upper layer, while ibota wax (an Asian insect wax) was identified as the waxy substance. Pine resin was detected by virtue of the presence of diterpenoids as dehydroabietic acid (DHA) and its oxidation products — namely 7-oxo-DHA (Mills & White, 1994; Pitthard et al., 2011), while the presence of various shellac acids helped to characterize shellac resin (Sutherland, 2010; Sutherland and del Rio, 2014).

As seen in Fig. 8, ibota wax has a distinctive chromatographic profile containing a series of long-chain fatty acids with even numbers of carbon atoms, the most abundant being hexacosanoic acid (C\textsubscript{26}O\textsubscript{0}). Ibota-ro (the Japanese name for ibota wax) is a natural insect wax that is scraped from the ibota-no-ki tree (Ligustrum ibota), onto which it has been secreted by young larvae of the wax-scale insect (Ericeurus pela), known in Japanese as ibota mushi. The composition of ibota wax is quite similar to China wax or pela wax, which also contain fatty acids in range C\textsubscript{24} to C\textsubscript{28} and long-chain alcohols with C\textsubscript{24} to C\textsubscript{32} (Li, 1985). In addition to ibota wax, which presumed to originate from traditional maintenance treatments still undertaken in China, remnants of beeswax were also detected by GC–MS in one of the samples taken from a later European retouching (see Table 1).

In the ground layer, the presence of blood was revealed by microchemical tests using an enzymatic reaction with hydrogen peroxide and benzidine (Odegaard et al., 2005). The benzidine reaction is useful to screen for blood but does not give 100% confirmation; it gives negative results for other proteins, such as fish glue, casein, and egg, but a positive reaction for rabbit skin glue. To identify blood unambiguously, further parallel analytical techniques are required. Accordingly, simultaneous proteinaceous analyses by GC–MS were performed to look for the presence of the characteristic ratios of blood amino acid.

**Figure 5** Hirox image at ×50 (left) and a backscattered electron SEM image at ×80 (right) showing multiple lacquer layers in a sample taken from the right base.

**Figure 6** Location on the frame at the back of the proper left panel from which the sample for Fig. 5 was taken.

**Figure 7** Cross-section from the frame at the back of the proper left panel, magnification ×200 (right). The multi-layered structure comprises: (1) a ground layer; (2) black lacquer; (3) a second (levelling?) ground layer; (4 and 5) two red lacquer layers; and (6) a yellow decorative orpiment layer.
Figure 8  Total ion chromatograms of: (A) the transparent coating; (B) an ibota wax reference standard; and (C) the uppermost
top coating. The labels refer to the components: W, wax components (long-chain fatty acids as methyl esters,
cyclohydrocarbons, and alcohols); P, diterpenes from pine resin; S, shellac components; and fatty acids from the drying oil (Su,
suberic acid; Az, azelaic acid; Pa, palmitic acid; and St, stearic acid). W1, docosanoic acid methyl ester; W2, tetracosanoic acid
methyl ester; W3, heptacosanol; W4, cyclotetracosane; W5, hexacosanoic acid methyl ester; W6, octacosanol; W7,
cyclohexacosane; W8, octacosanoic acid methyl ester; W9, cyclooctacosane; W10, triacontanoic acid methyl ester; W11,
unknown alcohol; W12, dotriacontanoic acid methyl ester; W13, unknown alcohol; P1, methyl pimarate; P2, methyl
dehydroabietate (DHA); P3, 6-methyl-7-methoxy-DHA; P4, methoxy-15-hydroxy-DHA; P5, 7-oxo-DHA; P6, 6-methyl-7,15-
dimethoxy-DHA; S1, jalaric acid, trimethyl, TFMP ether; S2, shellolic acid, dimethyl ester, methyl ether, TFMP ether; S3, jalaric
acid, dimethyl, di-TFMP ether; S4, jalaric acid, dimethyl, di-TFTP ether (isomer); S5, shellolic acid, dimethyl ester, di-TFMP ether;
and S6, aleuritic acid, methyl ester, 9-methyl ether, 10,16-di-TFMP ether.
acids in the binding media of the grounds. The GC–MS analyses showed a clear indication of the use of blood, but did not identify the species from which it was derived. Subsequent nano-LC-MS/MS analyses of the amino acid sequences in the peptides confirmed pigs’ blood; these analyses have been described in detail elsewhere (Miklin-Kniefacz et al., 2014).

Pyrolysis (Py–GC–MS) studies enabled various aged Asian lacquers to be differentiated by means of their various chemical compositions, which in turn allowed the provenance of the lacquer to be traced. Urushiol tapped from Toxicodendron vernicifluum trees growing in China and Japan was identified by its characteristic markers and oxidation products, as shown in Fig. 9 (Lu et al., 2006; Pitthard et al., 2010; Le Hô et al., 2012, Schilling, 2012). Two particular methylated catechols that were detected — those of C_{15:1} catechol (m/z 346) and C_{15:0} catechol (m/z 348) — are major pyrolysis markers of urushiol, while the presence of the methyl ester of mazzeic acid (m/z 294) indicated that urushiol was already undergoing oxidation. Cedrene and cedrol were detected, showing the presence of cedar oil in the red lacquer. Moreover, tung oil was detected in the red lacquer and perilla oil in black lacquer, respectively. The identification of tung oil was based on the presence of alkyl phenyl alkanoates (APAs) with base peaks at m/z 105 and on the fatty acid ratio, which gave P/S ~ 1 and A/P > 1. This contrasts to the ratios for perilla oil at P/S ~ 4 and A/P > 1, with the latter also including some longer-chain fatty acids (Schoenemann et al., 2008; Heginbotham and Schilling, 2011; Wei et al., 2011; Schilling, 2012).

Conservation

The results of the analyses gave an overview over the manufacturing materials and techniques and the state of preservation of this exquisite piece of furniture and provided valuable information when developing an approach to the conservation of the screen. The aims of this conservation treatment were the stabilization of some unstable elements of the structure of the screen, the consolidation of lifting lacquer areas and the cleaning of the red carved and the light-damaged black lacquer surfaces.

The carved fronts of the screen were cleaned using a 1:8 (v/v) mixture of isopropanol and water, aiming to retain the recent varnish that contained colophony and shellac as well as the residues of ibota wax, which served as traces of a possible historical or traditional maintenance treatment undertaken in China.

Cleaning of the black-lacquered backs with gilded decoration — which showed no varnish and were extremely water-sensitive — was carried out using petroleum benzene (boiling point range 100–140 °C). The results of the analyses, which showed that the black lacquer contained mainly urushiol, supported the decision to apply urushi-gatame. Chinese lacquer (da qi) was applied, diluted in a slow-evaporating hydrocarbon solvent, and allowed to penetrate for some time, then wiped away with a fast-drying hydrocarbon solvent so that the lacquer remains in the micro-cracks, but is removed from the surface completely;

Figure 9 The ion extracted pyrograms at m/z 294, 346, and 348 for the sample from the black lacquer showing the pyrolytic products of urushiol: (A) m/z 294, mazzeic acid methyl ester (MA); (B) m/z 346, methylated C_{15:1}-catechol; and (C) m/z 348, methylated C_{15:0}-catechol.
a detailed description of this method is given by Rivers et al. (2011). For the screen, a 1:1 mixture of raw lacquer and transparent lacquer diluted in Shellsol® A was used and excess lacquer removed from the surface with petroleum benzene (boiling point range 80–110 °C). This process was repeated once allowing both the severely light-damaged surface and the endangered gold powder decoration to be consolidated. The grey areas could not be identified as a different material and were reduced mechanically.

On the backs of the screen, there were many areas of lifting lacquer and lacquer losses, especially along the wooden joints, some of which revealed old wax repairs (Fig. 10). On the fronts there were several large areas of loose and missing lacquer in the light brown background design (Fig. 11). There were also some fairly large missing parts of lacquer on some corners of the base (Fig. 12).

For the consolidation of the areas of lifting lacquer on the black lacquer sides mugi-urushi (a mixture of raw lacquer with wheat flour that has been kneaded with water into a dough), dissolved in petroleum benzene (boiling point range 80–110 °C), gave excellent results with good adhesion of the lacquer and a minimal effect on the surrounding surface (Fig. 13). In contrast, a more flexible adhesive was needed in the light brown background parts of the carvings. Here a 3:2 mixture of two acrylic emulsions Plextol® D360 and Plextol® D498 was used and the same mixture bulked with natural cork powder and phenolic resin beads proved a suitable filler for several large cracks in the wooden structure.

If missing parts of the carved red and light brown lacquer disturbing the overall appearance of the screen they were filled with a 1:1 mixture of TeCero® 30222 and TeCero® 30201 microcrystalline waxes to which pigments were added to give a good colour match (Figs 13–15). For reversibility reasons, a separation layer of the Plextol® mixture described above was applied between ground or lacquer layer and the wax filling. Missing fittings were replaced by newly cast replacements, which electroplated with gold and patinated using acrylic colours to match the original elements.
Conclusions

The results of the scientific investigations helped to answer a number of questions concerning the composition of materials and the coating techniques used on the screen. As the uppermost transparent varnish was found to contain pine resin (colophony) and shellac, it most likely originates from a previous European restoration treatment, while remnants of ibota wax stem from maintenance treatment carried out in China. The lacquer layers contain urushiol and different oils — cedar and tung oils in the red areas and perilla oil in the black lacquer. The ground layers contain blood (most likely pigs’ blood) while the fibres in the ground come from ramie (China nettle). The use of conservation treatments such as the application of urushi-gatame and the use of mugi-urushi were supported by the findings of the examination, specifically that the lacquer layers consist of da qi (Chinese) or urushi (Japanese), the sap of Toxicodendron vernicifluum. The remnants of the insect wax were kept untouched as a historical record of previous treatment.

Finally, this carved lacquer screen will be a major highlight at the reopening of the Weltmuseum Wien, scheduled for 2017.

Experimental appendix

Microscopy

OM was carried out using a Zeiss Axioplan 2 microscope fitted with a Sony Power HAD video system to capture the images. The examination was conducted using both the visible and ultraviolet fluorescence modes using either a 100 W halogen lamp for visible or a 100 W high-pressure mercury lamp for ultraviolet excitation (using a 365 nm cut-off filter) or in blue light (using a blue filter that transmits in the range 450–490 nm).

SEM investigations were carried out using an FEI Quanta 200 F instrument in high vacuum mode with an acceleration voltage of 20 kV; the SEM was equipped with an EDX detection system for elemental analysis.

Digital 3D-microscopy using a HIROX KH-7700 3D digital microscope (HIROX) was used at magnifications from ×20 to ×800 with an integrated 60 W light source. A flexible fibre-optic lighting system allowed different lighting configurations, allowing the objects to be viewed in, for example, diffuse and raking light. The images were recorded with a resolution of 1200 × 1600 pixels.

GC–MS

The procedure for the analysis of lipids by GC–MS was based on the transesterification of fatty acids and the determination of their relative ratios to identify particular lipids, while the analytical procedure for resinous components was based on the esterification of resin acids followed by the identification of particular resins on the basis of the methyl esters of the various resin acids. Finally, the analytical procedure for proteinaceous materials was based on acid hydrolysis of the proteins to liberate free amino acids, followed by derivatization and quantitative determination of these amino acids as their silyl derivatives.

For the analyses of lipids and resins, the samples were treated with 30 μl of a 0.2 M methanolic solution of Meth-Prep II in a 1:2 (v/v) mixture of methanol and toluene (methylbenzene). The sealed vials were heated to 60 °C for one hour, then cooled to room temperature before 1 μl aliquots were injected into the GC inlet at a temperature of 300 °C. The separations were performed on a DB-5 MS poly(5% phenyl-95% methylsiloxane) capillary column with 0.25-mm internal diameter, 0.25-μm film thickness, and 30-m length (Agilent Technologies J&W, Santa Clara, CA, USA). After an initial hold at 50 °C for one minute, the oven temperature was programmed...
to increase to 320 °C at 10 °C/min and then held at this temperature for 12 minutes.

For protein analysis the samples were treated with 100 μl of 6 M hydrochloric acid (HCl) and the sealed vials heated to 105 °C for 24 hours. After cooling to room temperature the contents were evaporated to dryness under a gentle stream of nitrogen while warming the vials to 60 °C. High purity water (40 μl) was added, stirred, and the contents were again evaporated to dryness. Ethanol (40 μl) was added twice, stirred, and the content evaporated to dryness. The vials were then placed into a sealed desiccator for 24 hours. The dried samples were then treated with a pyridine–pyridine hydrochloride mixture (15 μl) and a silylation reagent (MTBSTFA, 30 μl). They were maintained at 60 °C for one hour then cooled before injecting 1 μl aliquots into the GC inlet at a temperature of 300 °C. The same capillary column was used as above but a different temperature programme was applied. After an initial hold at 80 °C for one minute, the oven temperature was programmed to increase to 280 °C at 6 °C/min and then held at this temperature for one minute. All the GC–MS analyses were performed on a 6890 N gas chromatograph connected to a model 5973N quadrupole mass spectrometer (both Agilent Technologies, Santa Clara, CA, USA).

**THM–Py–GC–MS**

The ability to analyse a solid sample directly, without any preparatory steps, is a major advantage of the pyrolysis method, with the pyrolysed materials introduced directly into the GC–MS system. The procedure for thermally assisted hydrolysis and methylation pyrolysis in Vienna was as follows: the lacquer samples and 1 μl of a 25% aqueous solution of the TMAH methylation reagent were placed on a Curie-Point wire and pyrolysed for 20 seconds at 764 °C in the Curie-point pyromat (GSG, Germany) that was attached to the GC–MS system (Agilent Technologies, Santa Clara, CA, USA). The same DB-5 MS capillary column and the same temperature programme were applied as used in the analysis of lipids and resins described above.

At the Getty Conservation Institute, the procedure allowed not only the lacquer components to be detected but also any drying oils that were present. A PY-2020D double-shot pyrolyzer (Frontier Lab, Koriyama, Japan) attached to a GC–MS system (Agilent Technologies, USA) was used. Samples were placed into a 50 μl stainless steel cup and 3 μl of a 25% methanolic solution of TMAH were introduced for derivatization. After three minutes the cup was placed into the pyrolysis interface where it was purged with helium. Pyrolysis was performed at 550 °C for six seconds and the sample was introduced onto a DB-5MS-UI (Agilent Technologies J&W, Santa Clara, CA, USA) capillary column for separation (30 m × 0.25 mm × 0.25 μm). After an initial hold at 40 °C for two minutes, the oven temperature was programmed to increase to 320 °C at 20 °C/min and then held at this temperature for nine minutes (Heginbotham & Schilling, 2011).

**Suppliers**

Benzdine, tetramethylammonium hydroxide (TMAH), pyridine: Fluka Chemie AG, Switzerland.

Hydrogen peroxide: Riedel-deHaën, Germany.

Methanol, N-tert-butyltrimethylsilyl-N-methyltri-fluoroacetamide (MTBSTFA), pyridine, toluene: Sigma-Aldrich, Austria.

Meth-Prep II® (m-trifluoromethylphenyl) trimethylammonium hydroxide (TFTMAH): Alltech Associates, Belgium.

Petroleum benzene 80/110 and 140/200: Neuber’s Enkel, Austria.

TeCero® waxes: Tromm, Germany.

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