#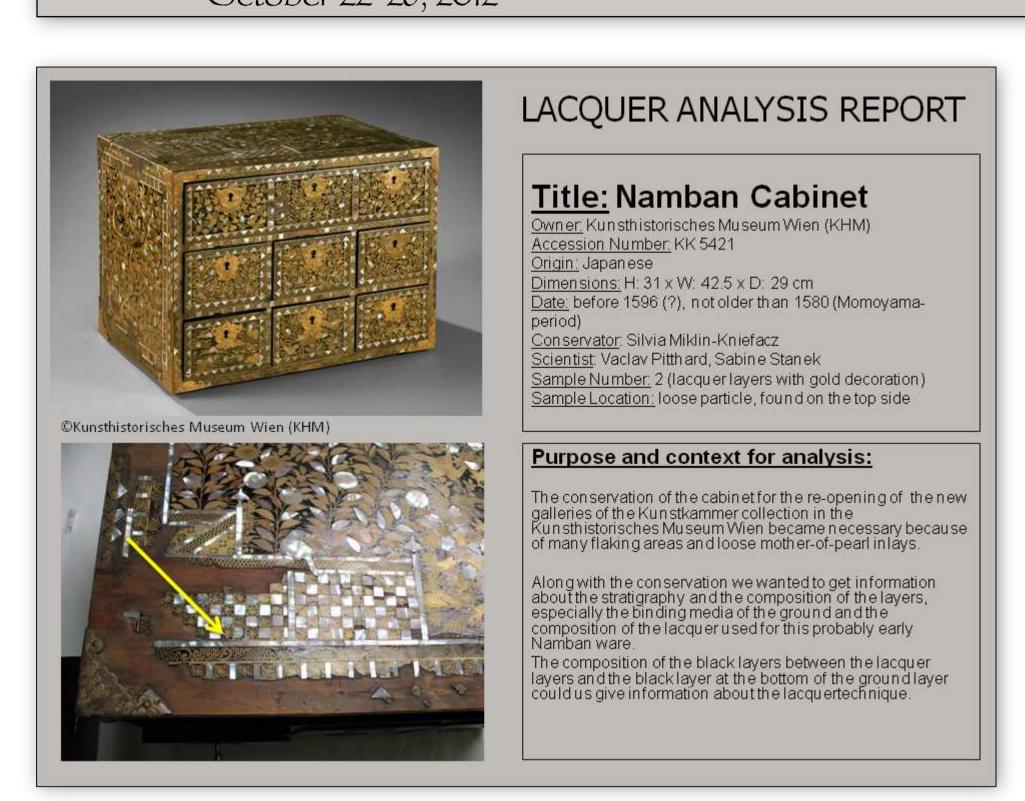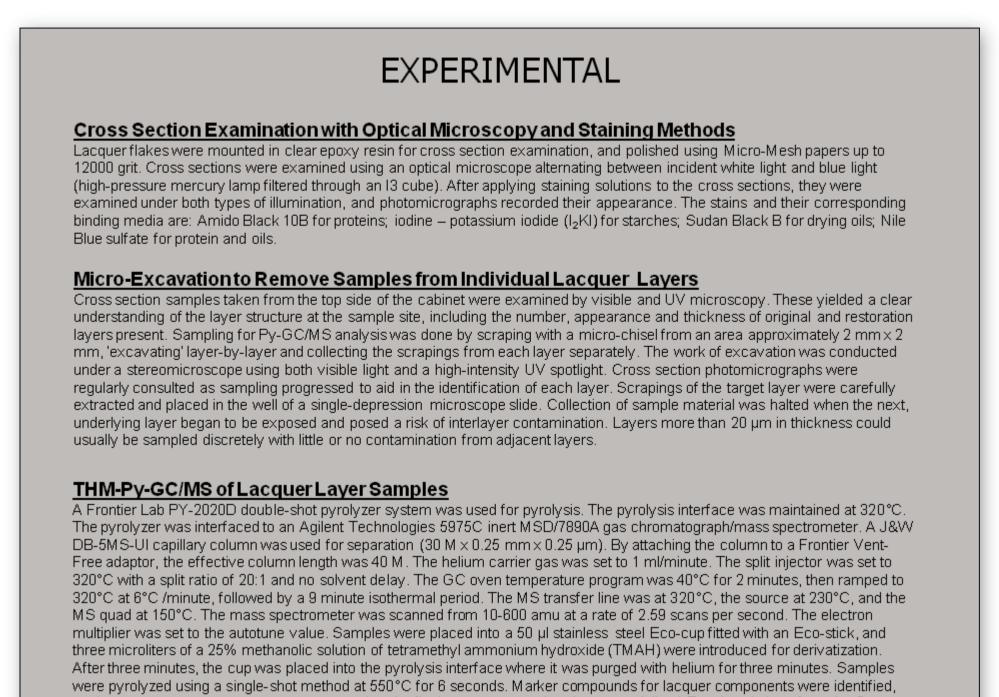 Organic Analysis of Asian lacquer layers from a Momoyama period Japanese Namban cabinet from the collection of the Kunsthistorisches Museum Vienna
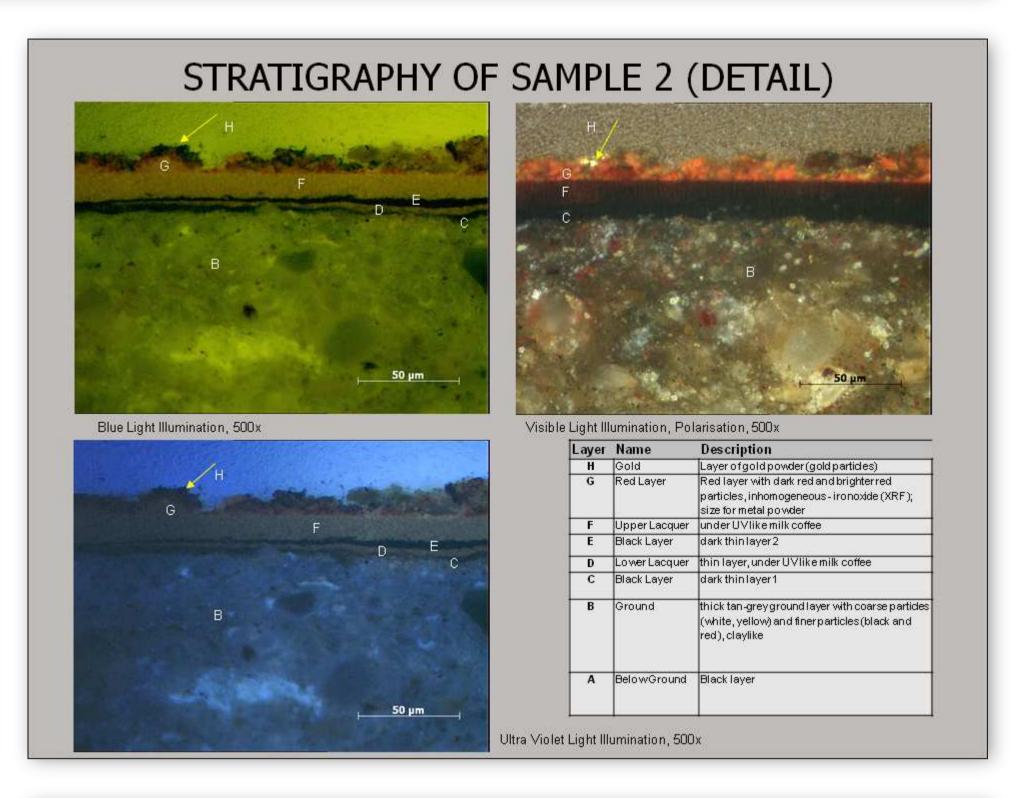
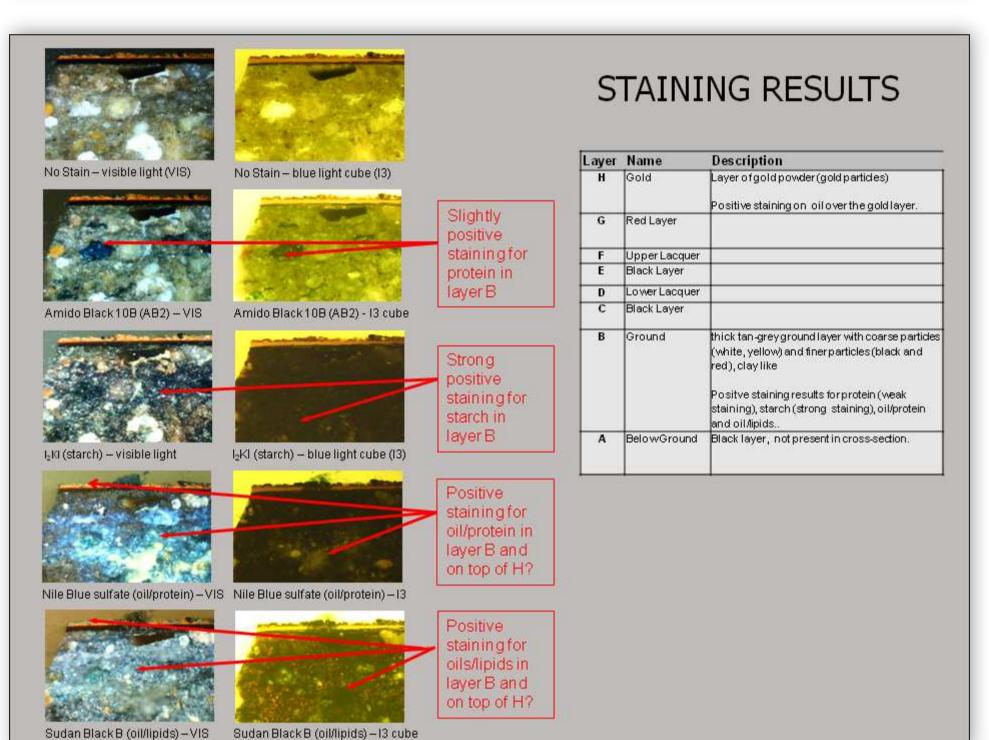Silvia Miklin-Kniefacz, Vienna
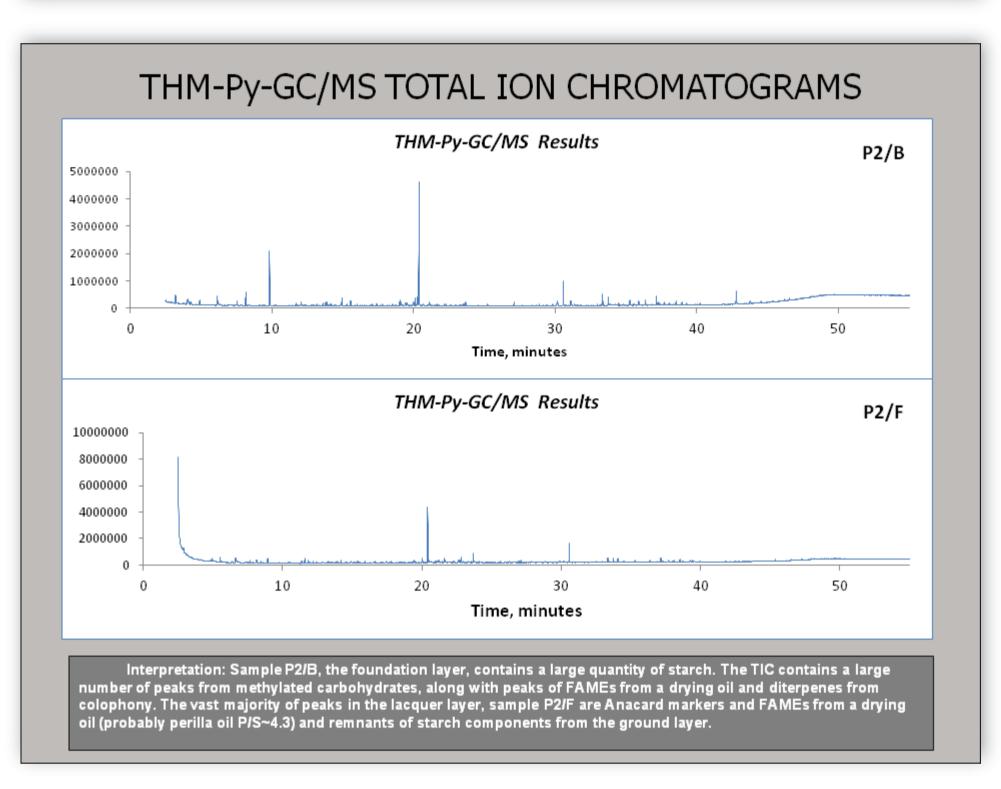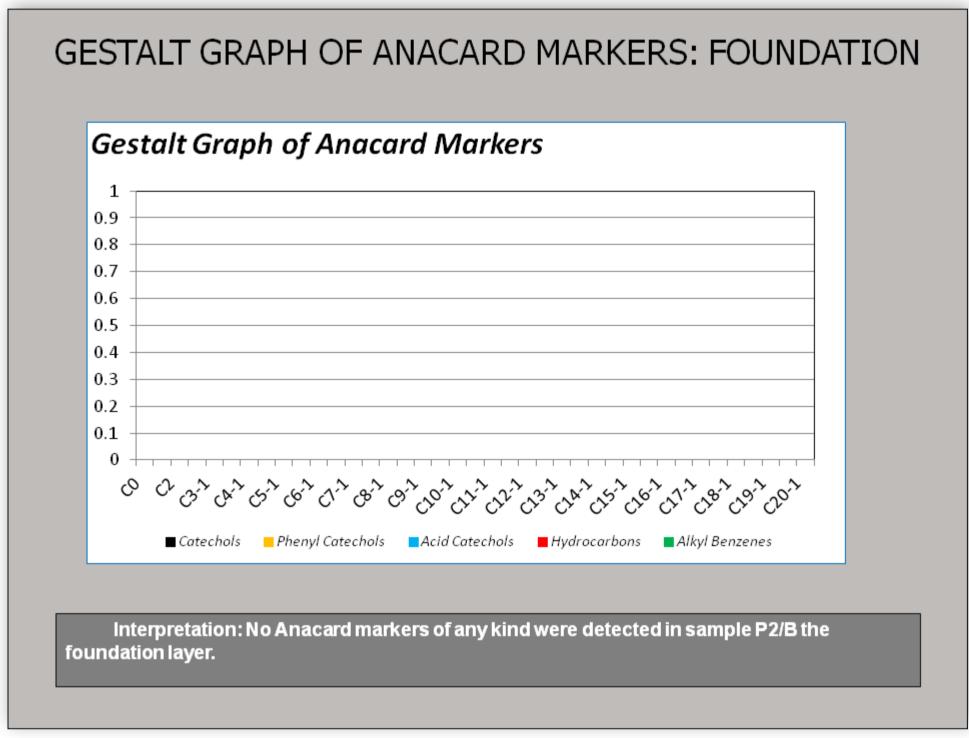Vaclav Pitthard, Kunsthistorisches Museum Vienna

RAdICAL Workshop, Session 1
Held at the Getty Center, Los Angeles
October 22–26, 2012

LACQUER ANALYSIS REPORT

©Kunsthistorisches Museum Wien (KHM)

Title: Namban Cabinet

Owner: Kunsthistorisches Museum Wien (KHM)
Accession Number: KK 5421
Origin: Japanese
Dimensions: H: 31 x W: 42.5 x D: 29 cm
Date: before 1596 (?), not older than 1580 (Momoyama-period)
Conservator: Silvia Miklin-Kniefacz
Scientist: Vaclav Pitthard, Sabine Stanek
Sample Number: 2 (lacquer layers with gold decoration)
Sample Location: loose particle, found on the top side

Purpose and context for analysis:

The conservation of the cabinet for the re-opening of the new galleries of the Kunstkammer collection in the Kunsthistorisches Museum Wien became necessary because of many flaking areas and loose mother-of-pearl inlays.

Along with the conservation we wanted to get information about the stratigraphy and the composition of the layers, especially the binding media of the ground and the composition of the lacquer used for this probably early Namban ware.

The composition of the black layers between the lacquer layers and the black layer at the bottom of the ground layer could us give information about the lacquertechnique.

EXPERIMENTAL

Cross Section Examination with Optical Microscopy and Staining Methods

Lacquer flakes were mounted in clear epoxy resin for cross section examination, and polished using Micro-Mesh papers up to 12000 grit. Cross sections were examined using an optical microscope alternating between incident white light and blue light (high-pressure mercury lamp filtered through an I3 cube). After applying staining solutions to the cross sections, they were examined under both types of illumination, and photomicrographs recorded their appearance. The stains and their corresponding binding media are: Amido Black 10B for proteins; iodine – potassium iodide (I₂KI) for starches; Sudan Black B for drying oils; Nile Blue sulfate for protein and oils.

Micro-Excavation to Remove Samples from Individual Lacquer Layers

Cross section samples taken from the top side of the cabinet were examined by visible and UV microscopy. These yielded a clear understanding of the layer structure at the sample site, including the number, appearance and thickness of original and restoration layers present. Sampling for Py-GC/MS analysis was done by scraping with a micro-chisel from an area approximately 2 mm x 2 mm, 'excavating' layer-by-layer and collecting the scrapings from each layer separately. The work of excavation was conducted under a stereomicroscope using both visible light and a high-intensity UV spotlight. Cross section photomicrographs were regularly consulted as sampling progressed to aid in the identification of each layer. Scrapings of the target layer were carefully extracted and placed in the well of a single-depression microscope slide. Collection of sample material was halted when the next, underlying layer began to be exposed and posed a risk of interlayer contamination. Layers more than 20 μm in thickness could usually be sampled discretely with little or no contamination from adjacent layers.

THM-Py-GC/MS of Lacquer Layer Samples

A Frontier Lab PY-2020D double-shot pyrolyzer system was used for pyrolysis. The pyrolysis interface was maintained at 320°C. The pyrolyzer was interfaced to an Agilent Technologies 5975C inert MSD/7890A gas chromatograph/mass spectrometer. A J&W DB-5MS-UI capillary column was used for separation (30 M x 0.25 mm x 0.25 μm). By attaching the column to a Frontier Vent-Free adaptor, the effective column length was 40 M. The helium carrier gas was set to 1 ml/minute. The split injector was set to 320°C with a split ratio of 20:1 and no solvent delay. The GC oven temperature program was 40°C for 2 minutes, then ramped to 320°C at 8°C /minute, followed by a 9 minute isothermal period. The MS transfer line was at 320°C, the source at 230°C, and the MS quad at 150°C. The mass spectrometer was scanned from 10-600 amu at a rate of 2.59 scans per second. The electron multiplier was set to the autotune value. Samples were placed into a 50 μl stainless steel Eco-cup fitted with an Eco-stick, and three microliters of a 25% methanolic solution of tetramethyl ammonium hydroxide (TMAH) were introduced for derivatization. After three minutes, the cup was placed into the pyrolysis interface where it was purged with helium for three minutes. Samples were pyrolyzed using a single-shot method at 550°C for 8 seconds. Marker compounds for lac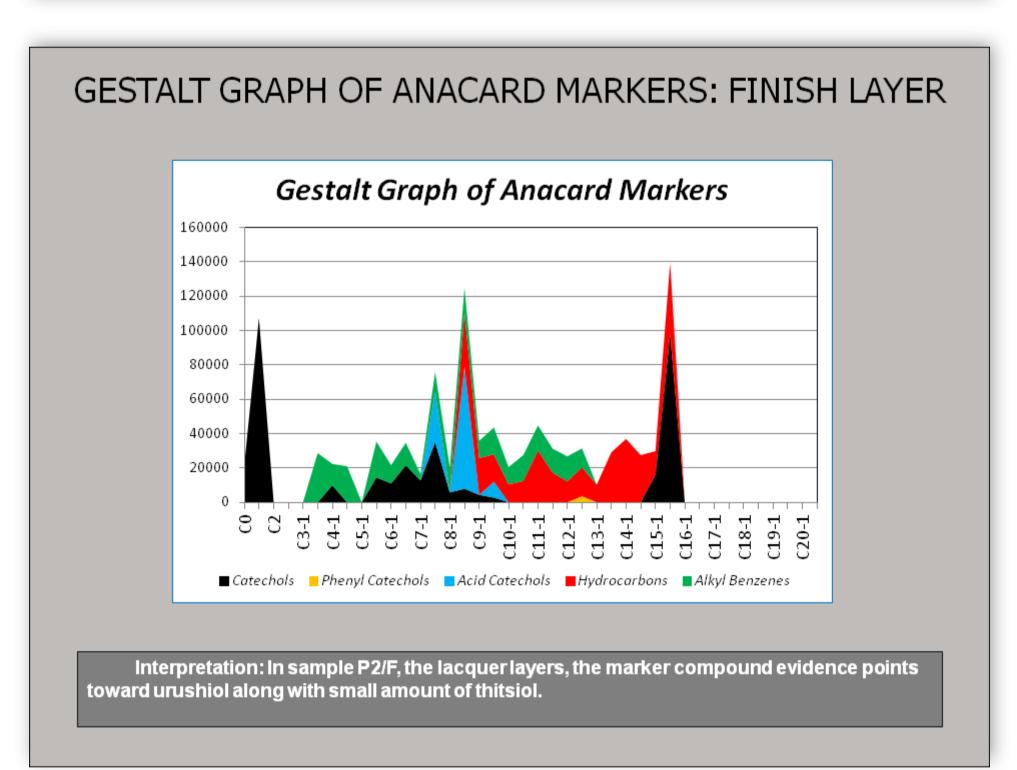quer components were identified, peak area percentage reports were generated and a final report was made using a specialized Excel worksheet from the Getty.

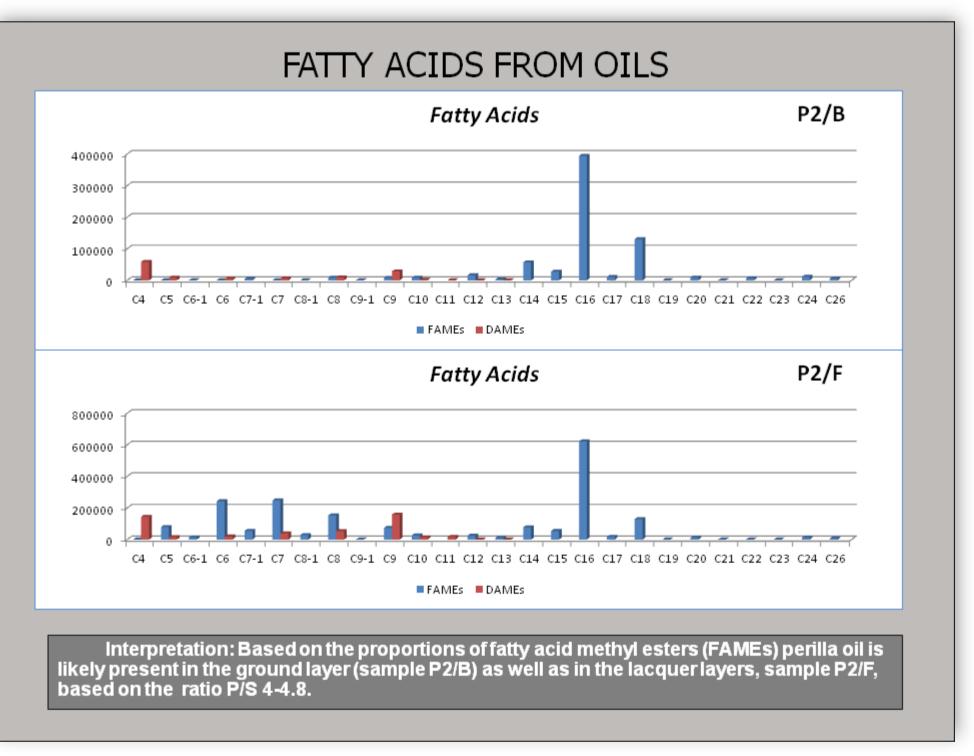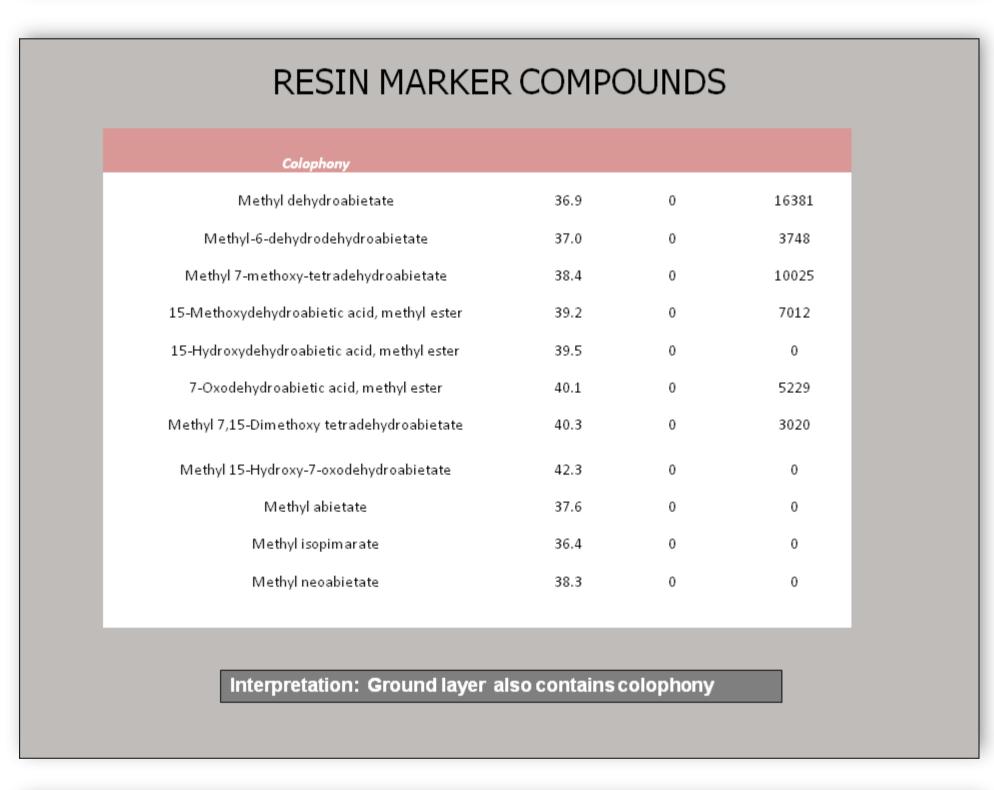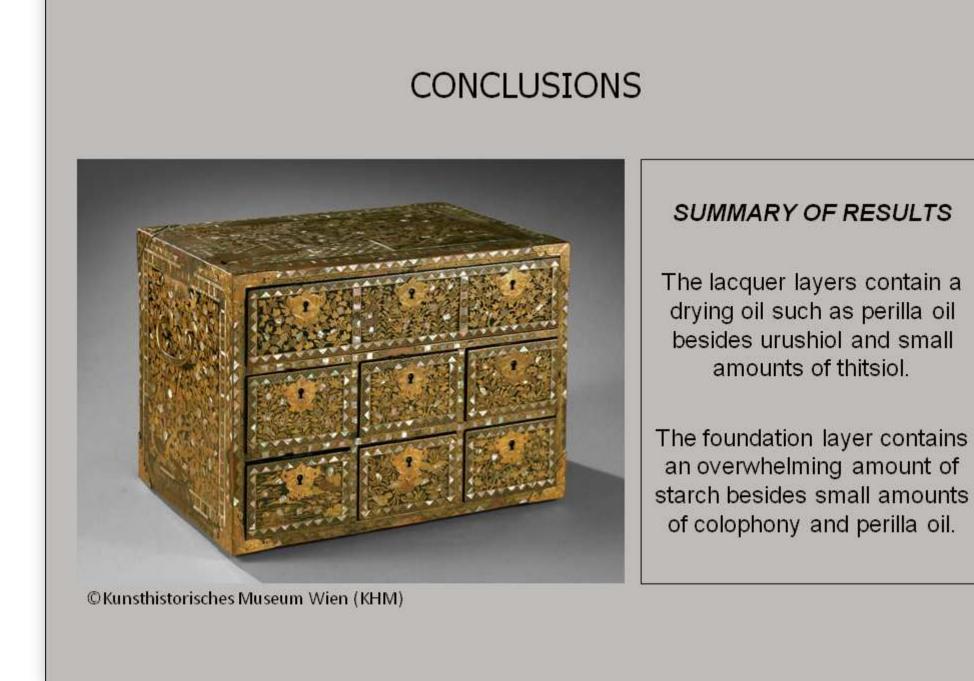## STRATIGRAPHY OF SAMPLE 2 (DETAIL)

Blue Light Illumination, 500x

Visible Light Illumination, Polarisation, 500x

Ultra Violet Light Illumination, 500x

Layer	Name	Description
H	Gold	Layer of gold powder (gold particles)
G	Red Layer	Red layer with dark red and brighter red particles, inhomogeneous - ironoxide (XRF?), size for metal powder
F	Upper Lacquer	under UV like milk coffee
E	Black Layer	dark thin layer 2
D	Lower Lacquer	thin layer, under UV like milk coffee
C	Black Layer	dark thin layer 1
B	Ground	thick tan-grey ground layer with coarse particles (white, yellow) and finer particles (black and red), clay like
A	Below Ground	Black layer

STAINING RESULTS

No Stain – visible light (VIS)
No Stain – blue light cube (I3)
Amido Black 10B (AB2) – VIS
Amido Black 10B (AB2) - I3 cube
I₂KI (starch) – visible light
I₂KI (starch) – blue light cube (I3)
Nile Blue sulfate (oil/protein) – VIS
Nile Blue sulfate (oil/protein) – I3
Sudan Black B (oil/lipids) – VIS
Sudan Black B (oil/lipids) – I3 cube

Slightly positive staining for protein in layer B

Strong positive staining for starch in layer B

Positive staining for oil/protein in layer B and on top of H?

Positive staining for oils/lipids in layer B and on top of H?

Layer	Name	Description
H	Gold	Layer of gold powder (gold particles)
G	Red Layer	Positive staining on oil over the gold layer.
F	Upper Lacquer	
E	Black Layer	
D	Lower Lacquer	
C	Black Layer	
B	Ground	thick tan-grey ground layer with coarse particles (white, yellow) and finer particles (black and red), clay like
		Positive staining results for protein (weak staining), starch (strong staining), oil/protein and oil/lipids.
A	Below Ground	Black layer, not present in cross-section.

THM-Py-GC/MS TOTAL ION CHROMATOGRAMS

THM-Py-GC/MS Results P2/B

THM-Py-GC/MS Results P2/F

Interpretation: Sample P2/B, the foundation layer, contains a large quantity of starch. The TIC contains a large number of peaks from methylated carbohydrates, along with peaks of FAMEs from a drying oil and diterpenes from colophony. The vast majority of peaks in the lacquer layer, sample P2/F are Anacard markers and FAMEs from a drying oil (probably perilla oil P/S~4.3) and remnants of starch components from the ground layer.

GESTALT GRAPH OF ANACARD MARKERS: FOUNDATION

Gestalt Graph of Anacard Markers

Catechols Phenyl Catechols Acid Catechols Hydrocarbons Alkyl Benzenes

Interpretation: No Anacard markers of any kind were detected in sample P2/B the foundation layer.

GESTALT GRAPH OF ANACARD MARKERS: FINISH LAYER

Gestalt Graph of Anacard Markers

Catechols Phenyl Catechols Acid Catechols Hydrocarbons Alkyl Benzenes

Interpretation: In sample P2/F, the lacquer layers, the marker compound evidence points toward urushiol along with small amount of thitsiol.

FATTY ACIDS FROM OILS

Fatty Acids P2/B

FAMEs DAMEs

Fatty Acids P2/F

FAMEs DAMEs

Interpretation: Based on the proportions of fatty acid methyl esters (FAMEs) perilla oil is likely present in the ground layer (sample P2/B) as well as in the lacquer layers, sample P2/F, based on the ratio P/S 4-4.8.

RESIN MARKER COMPOUNDS

Colophony			
Methyl dehydroabietate	36.9	0	16381
Methyl-6-dehydrodehydroabietate	37.0	0	3748
Methyl 7-methoxy-tetradehydroabietate	38.4	0	10025
15-Methoxydehydroabietic acid, methyl ester	39.2	0	7012
15-Hydroxydehydroabietic acid, methyl ester	39.5	0	0
7-Oxodehydroabietic acid, methyl ester	40.1	0	5229
Methyl 7,15-Dimethoxy tetradehydroabietate	40.3	0	3020
Methyl 15-Hydroxy-7-oxodehydroabietate	42.3	0	0
Methyl abietate	37.6	0	0
Methyl isopimarate	36.4	0	0
Methyl neoabietate	38.3	0	0

Interpretation: Ground layer also contains colophony

CONCLUSIONS

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SUMMARY OF RESULTS

The lacquer layers contain a drying oil such as perilla oil besides urushiol and small amounts of thitsiol.

The foundation layer contains an overwhelming amount of starch besides small amounts of colophony and perilla oil.

ACKNOWLEDGMENTS