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Marie Radepont, Céline Bonnot-Diconne, Laurianne Robinet, Sylvie Thao, Claire Pacheco, et al.. The impact of manufacturing and conservation on silver leaf tarnishing in gilt leather. ICOM-CC 18th Triennial Conference, Sep 2017, Copenhagen, Denmark. hal-02634934

**HAL Id: hal-02634934**

**<https://hal.science/hal-02634934>**

Submitted on 10 Jun 2020

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# The impact of manufacturing and conservation on silver leaf tarnishing in gilt leather

## MARIE RADEPONT\*

Centre de Recherche sur la Conservation  
Centre de Recherche et de Restauration des Musées  
de France  
Paris, France  
marie.radepont@gmail.com

## CÉLINE BONNOT-DICONNE

Centre de Conservation et de Restauration du Cuir  
Moirans, France  
cbonnotdiconne@aol.com

## LAURIANNE ROBINET

Centre de Recherche sur la Conservation  
Paris, France  
laurianne.robinet@mnhn.fr

## SYLVIE THAO

Centre de Recherche sur la Conservation  
Paris, France  
thao@mnhn.fr

## CLAIRE PACHECO

Centre de Recherche et de Restauration des Musées  
de France  
Paris, France  
claire.pacheco@culture.gouv.fr

## MURIEL BOUTTEMY

Institut Lavoisier de Versailles  
Université de Versailles St-Quentin-en-Yvelines  
Versailles, France  
muriel.bouttemy@uvsq.fr

## ELISE DELBOS

Université de Versailles St-Quentin-en-Yvelines  
Versailles, France  
KMG Ultra Pure Chemicals  
St. Fromond, France  
edelbos@kmgchemicals.com

\*Author for correspondence

**KEYWORDS:** gilt leather, silver leaf, tarnishing, burnishing, lubricants, cleaning, atomic absorption spectroscopy, py-GC/MS

## ABSTRACT

While the removal of corrosion products from silver surfaces without damage is not possible for gilt leather, improving our knowledge of the tarnishing of silver leaves and how to prevent it is essential. Silver tarnishing is common, generally localized, and, in extreme situations, total blackening of the silvered areas may occur, affecting the readability of the decors. In some cases, this degradation may be related to conservation interventions. This research investigates the role of the materials and environment

## INTRODUCTION

Silver tarnishing on gilt leather decorations is commonly observed, generally localized, and, in extreme situations, total blackening of the silvered area can occur (Figure 1). A grid pattern sometimes observed on these decors may also be associated with preferential corrosion on the leaf edges (Figure 2). The consequences are important, as it affects readability and many of these objects are then left in storage. In some cases, this degradation may be related to conservation interventions. Consequently, the impact of maintenance and conservation treatment on gilt leather and its effect on silver leaf is questioned.

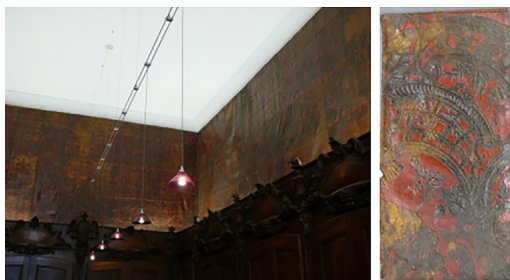
In the cultural heritage field, silver tarnishing has been studied on foils in paintings or massive objects, but never on gilt leather (Costa 2001, Ingo et al. 2006, Salvado et al. 2011). The principal alteration product identified, silver sulfide ( $\text{Ag}_2\text{S}$ ), is formed in the presence of humidity, oxygen and sulfur-containing compounds, mainly according to the following reaction mechanism:  $2\text{Ag} + \text{H}_2\text{S} + 0.5 \text{O}_2 \rightarrow \text{Ag}_2\text{S} + \text{H}_2\text{O}$  (Palomar et al. 2016).

Better understanding of the processes involved in silver alteration would help advise suitable conservation strategies for these decors. For that reason, this research investigates the role of materials and environmental interaction with the silver leaf by considering two steps in the life of these decors: manufacture and maintenance, including conservation.

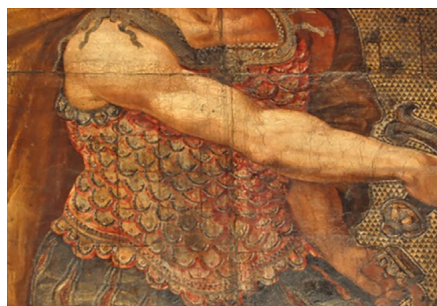
Gilt leather is a composite material made from a vegetable tanned leather, on which is glued a silver leaf, which is then burnished and covered with an oil-resin varnish to give a gold-like appearance (Fougeroux de Bondaroy 1762). For esthetic effects, these decors can combine “gilt” and “silvered” areas, generally prepared by applying a very thin varnish or protein-based layer over the leaf. On some decors, paint layers were also added on top (Fournet 2004). The conservation of gilt leather may involve different steps, depending on the diagnosis of the surface condition of the decor, such as cleaning, gluing, reshaping, infilling or lubricating, implying the use of different materials and chemicals (Fournet et al. 2006).

This paper first examines the tarnishing role of the materials present in gilt leather, such as the properties of metal leaf, the influence of the burnishing step, and the presence of “gold” varnish or a potential protective layer.

interacting with the silver leaf, considering two stages in the life of these decors: manufacture and maintenance, including conservation.



**Figure 1.** Photograph and detail of a gilt leather wall covering with blackening of the “gilt” area (City Hall, Aix-en-Provence, Toro Room, France, 1738)



**Figure 2.** Detail of a gilt leather wall covering presenting a grid pattern (*Les Héros*, Musée national de la Renaissance, Ecouen, France)

Then, it tackles the impact of some of the chemicals and techniques used during conservation treatments.

## **MATERIALS AND METHODS**

### **Manufacturing stage**

Different kinds of modern samples were prepared.

Gilt leather models were produced following the recipe transposed by Fougereux de Bondaroy (1762) using three modern types of silver leaf: pneumatically beaten Manetti and Dauvet leaves, and hand-beaten Berta leaves. Silver leaves were attached to humidified vegetable tanned leather (Azais) with skin glue, burnished and then covered with two layers of a “gold” varnish, made essentially of linseed oil and pine resin. “Silvered” areas were made by removing the varnish while still liquid on the surface as described in the recipe. Samples of gilt leather models were exposed to different environments at ambient temperature (uncontrolled) for over one year: 50% relative humidity (RH) in daylight behind a window, 50% RH in the dark, and 80% RH in the dark.

Other model samples were prepared by applying silver leaves on a glass slide or gluing with skin glue onto leather and were partly burnished with an agate stone. The aim was to evaluate the influence of the leaves’ properties and the mechanical action on their reactivity. Some of these samples were covered with egg white, skin glue, or “gold” varnish to examine the influence of these materials on corrosion.

The composition and thickness of silver leaves were determined respectively by particle induced x-ray emission (PIXE) and Rutherford backscattering spectroscopy (RBS) performed at the AGLAE facility at atmospheric pressure with a 30- $\mu\text{m}$  external beam composed, respectively, of proton and alpha particles at 3 MeV (Pichon et al. 2014). Porosities in silver leaves were observed using a scanning electron microscope with a field emission gun (SEM-FEG) at 4 kV and a working distance of 6 mm. A small piece of silver leaf was glued onto carbon tape and a bridge between the leaf and tape was made with carbon lake. Tarnish products were identified using a Renishaw inVia Raman microscope with a 532 nm laser, a 20 $\times$  objective and a power of 4.5  $\mu\text{W}$  at the sample surface.

### **Conservation stage**

The corrosiveness of materials interacting with gilt leather was studied using a modified version of the accelerated corrosion test (Oddy test) (Robinet et al. 2003). Silver leaves were deposited on a glass slide and placed in the tube with saturated humidity in two different positions: either installed at the bottom of the test tube in contact with the tested materials, or inserted in the silicone stopper and exposed to the volatile compounds emitted by the materials. The tubes were kept at 40°C for one month or 28°C for three months. Conservation materials tested included:

- detergents: Avel regenerated, Synperonic N, Brecknell soap
- adhesives: Mowilith DMC2, Beva 371, Plexisol P550, Acryl 33, skin glue, Klucel G

- lubricants: Renaissance wax, Vaseline oil, castor oil, neatsfoot oil, Chamonix cleaning lotion, Avel nutritive cream, wax 213, British Museum leather dressing with and without beeswax, Nivea cream
- modern leathers: tanned with vegetable tannins and lubricated with different oils
- support materials for cleaning: melamine foam, laponite gel.

In this list, some conservation materials are not necessarily used by conservators, but they were tested as leather maintenance products.

The impact of basic solvents and mechanical cleaning during conservation was assessed through the analysis of cotton swabs and sponges (melamine foam) collected during the cleaning of three ancient gilt leather decors from Villeneuve-Lembron (wall hanging, France, 17th century), Aubazat (altar frontal, France, 17th century), and a private collection (wall hanging, the Netherlands, 17th century). Saliva, demineralized water, and white spirit are the first solvents usually used first hand to test the cleaning options. To examine if materials originating from the gilt leather were removed during the cleaning process, analyses were performed by pyrolysis-gas chromatography/mass spectrometry (py-GC/MS) to detect the presence of oil-resin varnish and by atomic absorption spectroscopy (AAS) and portable x-ray fluorescence (p-XRF) spectroscopy to detect the presence of silver. For the py-GC/MS analyses, a small fragment (around 50 µg) from a dirty part of the cotton or the foam was directly introduced into a Shimadzu GCMS-QP2010Plus/Frontier Lab PY-2020iD system using thermally assisted methylation with tetramethylammonium hydroxide (TMAH), according to the procedure developed for the analysis of ancient varnishes (Echard et al. 2007). Quantitative AAS analyses were performed using a Thermofisher SOLAAR M6QZ GF-AAS spectrometer on dosages of silver particles released from the cotton matrix in acidic solution (2% HNO<sub>3</sub>). The furnace method (air-acetylene flux of 0.9 L·min<sup>-1</sup>) was favored to minimize matrix effects, and the main 328.1 nm wavelength was selected for detection. Each result presented was an average of 5 measurements. Comparative measurements with an Elio m-XRF instrument were carried out directly on cottons and foams with an Rh tube at 40 kV, 60 µA, during 180 seconds per point.

## **RESULTS AND DISCUSSION**

### **Influence of gilt leather manufacturing**

The chemical composition and thickness of the modern silver leaves were measured by PIXE and RBS analyses. The common elements identified in the three modern leaves were silver and chlorine as well as zinc, strontium, nickel, and copper as trace elements (relative concentration below 0.1%) (Table 1). Differences were seen in the quantity of gold in the three leaves, with only traces in the Manetti and Berta leaves and a minor amount (relative concentration above 0.1%) in the Dauvet leaves. Finally, some trace elements were specific to a supplier, with germanium and selenium in the Dauvet leaves, and brome in the Berta leaves. Thicknesses of around 220 nm for the Manetti and Dauvet leaves and around 240 nm for the Berta leaves were calculated (Table 1). Differences in leaf porosity were

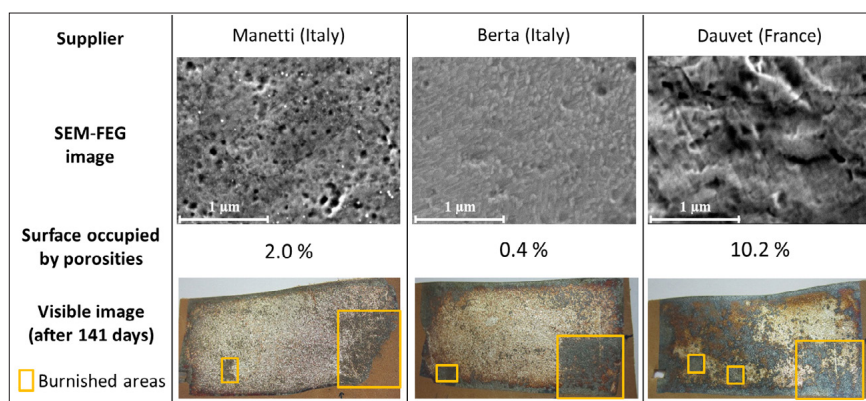
LEATHER AND RELATED MATERIALS

THE IMPACT OF MANUFACTURING  
AND CONSERVATION ON SILVER LEAF  
TARNISHING IN GILT LEATHER

**Table 1.** Composition and thickness of modern silver leaves glued onto leather obtained, respectively, by PIXE (3-MeV proton) and RBS (3-MeV alpha) analyses, where (x) stands for a major or minor element and (Tr) for a trace element

Supplier	Composition										Thickness
	Ag	Cl	Zn	Sr	Au	Ni	Cu	Ge	Se	Br	
Manetti	x	x	x	x	Tr	Tr	Tr	-	-	-	220 ± 40 nm
Berta	x	x	x	x	Tr	Tr	Tr	-	-	Tr	240 ± 60 nm
Dauvet	x	x	x	x	x	Tr	Tr	Tr	Tr	-	220 ± 40 nm

observed using SEM-FEG image analysis (Figure 3). Interestingly, the Dauvet leaves, which had a higher porosity, were the first to show corrosion in non-burnished and uncovered leaves attached to the leather (Figure 3). The tarnishing product was identified by Raman microspectroscopy as silver sulfide (Ag<sub>2</sub>S) from the presence of two main peaks at 150 and 240 cm<sup>-1</sup>. In areas where the leaf was burnished, an accelerated corrosion phenomenon was observed for all samples (Figure 3). The hypothesis is that the mechanical action induced by burnishing damages the thin natural and protective silver oxide layer, increasing the reactivity of the leaf. A specific study is ongoing to understand the impact of burnishing on the structure of metallic leaves.

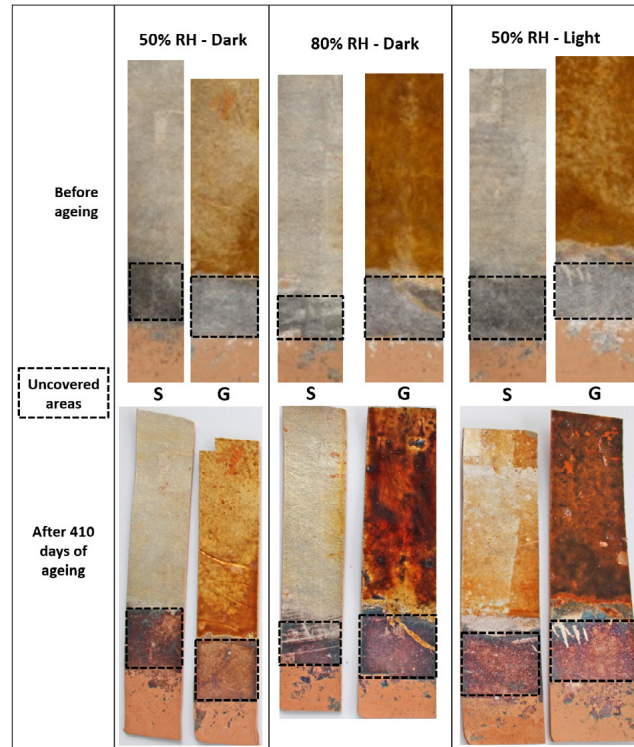


**Figure 3.** SEM-FEG images and photographs of silver leaves from three suppliers; burnished areas are highlighted with yellow squares

When egg white, skin glue or oil-resin varnish was applied on the silver leaves, even as a very thin layer of a few microns, it acted as an efficient barrier against atmospheric induced corrosion. In gilt leather model samples exposed to controlled environments, different surfaces exist: uncovered silver leaf, and “silvered” and “gilt” areas with, respectively, a thin and a thick layer of oil-resin varnish (Figure 4). When silver is totally uncovered, greater tarnishing is observed for the samples exposed to light or high RH (80%), which is consistent with previous studies showing that moisture and light accelerates silver corrosion (Inaba 1996). In the “silvered” area, no tarnishing is observed in any of the environments regardless of the silver leaf supplier (Figure 4). This confirms the protective effect of the thin “gold” varnish layer that acts as a barrier against humidity. For “gilt” areas, however, a darkening is observed for the samples exposed to light or high RH (Figure 4). Silver tarnishing may be responsible for this change of visual aspect, but, in the case of the sample exposed to light, part of the darkening may be associated with the yellowing of the oil-resin varnish as observed in the silvered area (Figure 4). Indeed, in the presence of humidity,



hydrolysis of the fatty acids present in the oily part of the varnish takes place and forms alcohol and other acids. This observation suggests that a thick oil-resin varnish layer may become corrosive to silver in the presence of light or high humidity. This particular point should be investigated in more detail. The corrosive effect of materials from the paint layers was not examined here; however, the effects of some sulfur-containing compounds such as the vermilion ( $\alpha$ -HgS) pigment could be studied.



**Figure 4.** Photographs of gilt leather model samples prepared with Manetti silver leaves before and after being exposed to different atmospheres at ambient temperature for over one year; “silvered” and “gilt” areas are respectively indicated by (S) and (G)

### Influence of conservation

Different products used in the past and today in the conservation or maintenance of leather were tested. All detergents, adhesives and most lubricants tested did not induce any corrosion of the silver. Only two lubricants showed a detrimental effect on silver leaf when applied in direct contact at both tested temperatures: neatsfoot oil and British Museum leather dressing with or without beeswax. Neatsfoot oil is made by boiling the shin bones and feet of cattle, but other compounds may be added to the formulation nowadays. British Museum leather dressing contains a mixture of lanolin, cedar oil, and beeswax with hexane solvent. Although lubricants are often applied on the back of leather, experience has shown that the lubricant generally migrates through all the different layers to accumulate on the decor surface, meaning that it can come into contact with the silver leaf and react with it (Posthuma de Boer 2012). Although no corrosion was observed on the silver, some changes in the appearance of the silver or varnish were observed with the following products: Renaissance wax, Vaseline oil and Avel regenerated detergent. All modern vegetable tanned leathers tested have shown silver tarnishing to various degrees both with and without contact (from emitted vapors). Nowadays, sulfur-based compounds are largely used in the manufacturing of leather, either during the liming or lubricating stage, and unfortunately enhance corrosion. This was clearly demonstrated with leather lubricated with sulfited oil, which presents the greatest degree of corrosion (Table 2).

LEATHER AND RELATED MATERIALS

THE IMPACT OF MANUFACTURING  
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**Table 2.** Results of accelerated corrosion experiments with silver leaves glued onto modern leathers and on glass slides in a closed environment for one month at 40°C; (+) to (+++) indicate the gradual level of corrosion observed

Supplier	Lubricant	Degree of corrosion	
		Direct contact	Volatile emission
Lieutard	Sulfited oil	(+++)	(+++)
Azais	Fish oil	(++)	(+)
STEP (mimosa)	Neatsfoot oil	(+)	(+)
STEP (sumac)	Neatsfoot oil	(+)	(+)
Leather Conservation Centre	Neatsfoot oil	(+++)	(++)
Leather Conservation Centre	Sulfited fish oil	(+++)	(++)

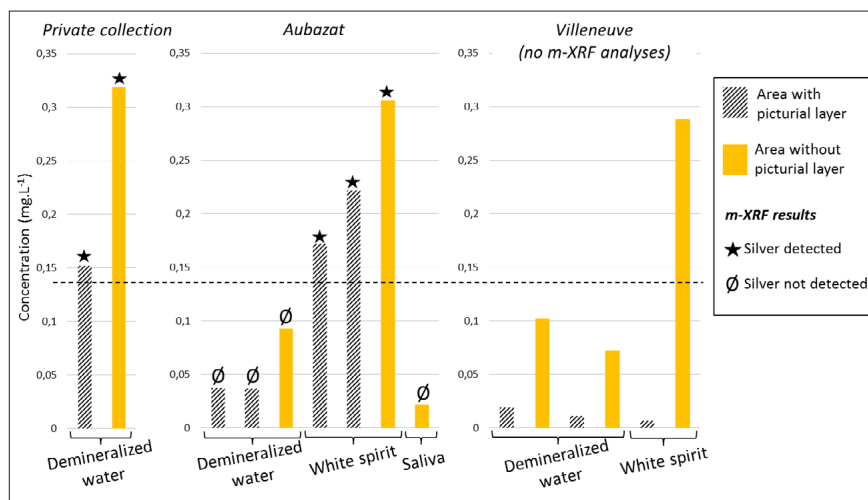
One of the first treatments performed by conservators on ancient gilt leathers is to clean the dust and grime accumulated on the surface. The main concern during this treatment is the risk of removing elements from the decors such as the “gold” varnish protective layer or even silver.

In the cotton swabs and foams used to clean the three ancient gilt leather decors, py-GC/MS detected the presence of oil-resin varnish based on the presence of markers of siccative oil (azelaic acid) and pine resin (dehydroabietic acid, DHA) (Table 3). Semi-quantitative information could be extracted from these data. When no or only traces of resin were detected, it suggests that no or very little “gold” varnish was removed, as in the “gilt” area cleaned with distilled water in the Villeneuve-Lembron decor. Py-GC/MS also detected the presence of conservation products such as beeswax and lanolin, particularly in the decor from Aubazat. Results indicate that all solvents can remove “gold” varnish to some degree, but these are influenced by the presence of other layers such as paint or previous conservation treatments. Melamine foam was only tested on one decor, but the analyses suggest that the foam removes a greater quantity of matter than cotton swabs (lubricants and varnish), which supports the conservators’ view during cleaning. Melamine foam is a new material in conservation, and research should be conducted to assess its action during cleaning.

Concerning the detection of silver, AAS proved to be the most sensitive technique for detecting traces of silver (a detection limit of around 0.001 mg·L<sup>-1</sup>) and its quantification was possible from cotton swabs. Whatever the origin of the cotton swabs, cleaning with white spirit led to the highest amount of silver detected, whereas those with saliva showed the lowest amount of silver (Figure 5). In the case of the Aubazat samples, the difference in the amount of silver might come from the presence of beeswax (identified by GC/MS), which formed a barrier to the saliva or distilled water during cleaning. Lower amounts of silver were also detected by AAS in painted areas, which acts as a protective layer (Figure 5). The mechanical effect of the cotton and foam is probably responsible for removing silver from cracks and lightly abrading the “gold” varnish layer. Although elements of the decor are removed in all cases, the total amount of silver and “gold” varnish detected remains very low and invisible to the eye.

Finally, p-XRF was also performed on cotton swabs collected on two ancient decors (Figure 5) and compared to the AAS results to evaluate the possibility of conservators using this more accessible technique to

detect silver removal during cleaning. Measurements showed that this instrument was able to measure silver content above  $0.1 \text{ mg}\cdot\text{L}^{-1}$  with respect to AAS dosage (Figure 5). Therefore, the use of p-XRF could be proposed as a simple and rapid method to assess the impact of cleaning methods on silver in gilt leather decors.



**Figure 5.** Results of AAS and p-XRF analyses of cotton swabs used to clean three ancient gilt leathers, in areas with paint layers (striped bars) or with only “gold” varnish applied on silver leaves (filled yellow bars). Measurement accuracy of 10%

**Table 3.** Py-GC/MS analyses of the cotton swabs and foams used to clean gilt leather decors from: private collections, Aubazat and Villeneuve-Lembron

Gilt leather decor	Cleaning method	Oil-resin varnish			Conservation products	
		Oil marker	Resin marker	→ “Gold” varnish	Beeswax	Lanolin
Private collection “gilt” area	Cotton + distilled water	Yes	Traces	?	Traces	Traces
Private collection paint area	Cotton + distilled water	Yes	Yes	Yes	Traces	Traces
Aubazat “gilt” area	Foam/cotton + distilled water	Yes	Yes	Yes	Yes	Yes
Aubazat “gilt” area	Cotton + saliva	Yes	Traces	?	-	Traces
Aubazat “gilt” area	Foam + white spirit	Yes	Yes	Yes	Yes	Yes
Villeneuve “gilt” area	Cotton + distilled water	Traces	-	-	-	-
Villeneuve paint area	Cotton + white spirit	Yes	Traces	?	Other wax	-
Villeneuve “gilt” area	Cotton + white spirit	Yes	Yes	Yes	-	Traces

## CONCLUSION

While the removal of corrosion products from silver surfaces without damage is not possible for gilt leather, improving our knowledge of the tarnishing of silver leaves and how to prevent it is essential.

During manufacturing, the characteristics of the leaf have an influence on the development of tarnishing, especially on account of the presence of porosities and the action of burnishing. The role of egg white, skin glue or “gold” varnish as a protective barrier against silver corrosion has



been confirmed. However, in the presence of a thick “gold” varnish layer combined with high humidity and/or light, darkening may occur.

During conservation, the use of lubricants (in particular neatsfoot oil and British Museum leather dressing) as well as modern leathers for infilling or backing gilt leathers can induce silver leaf corrosion, so these materials should be avoided. Moreover, the analyses showed that the action of basic cleaning with cotton swabs or sponges removes oil-resin varnish and silver particles from gilt leather surfaces, which may expose the silver and enhance its tarnishing. Therefore, repeated cleaning processes should be avoided and great care should be taken during cleaning, especially for silvered areas.

## **ACKNOWLEDGEMENTS**

This study was performed under the CORD’ARGENT Project, funded by the Fondation des Sciences du Patrimoine (LabEx PATRIMA), and as part of the New AGLAE project (grant ANR-10-EQPX-22). The authors would like to thank Arnaud Etcheberry and Hanane Elbelghiti (ILV-KMG) for their commitment to the project; Laurent Pichon, Quentin Lemasson and Brice Moignard for their support with the PIXE and RBS analyses at the AGLAE facility, Eric Laval for the m-XRF analyses, and Roland Février from the C2RMF Conservation Department for the mock-up samples. We would also like to thank Marie Heran for collecting the cleaning swabs and foams. The authors are grateful to Jean-Pierre Fournet for sharing his expertise on ancient gilt leather and to all the owners of the collections.

## **MATERIALS LIST**

### *Conservation and maintenance materials*

From [www.laverdure.fr/](http://www.laverdure.fr/)

- Avel regenerated detergents
- Avel nutritive cream
- Chamonix cleaning lotion
- Vaseline oil
- Castor oil
- Neatsfoot oil
- Rabbit-skin glue

From [www.cxd-france.com/](http://www.cxd-france.com/)

- British Museum leather dressing with/without beeswax
- Wax 213
- Brecknell soap
- Renaissance wax
- Laponite gel

From [www.ctseurope.com/](http://www.ctseurope.com/)

- Mowilith DMC2 adhesives
- Beva 371
- Plexisol P550
- Acryl 33
- Klucel G

From [www.conservationresources.com/](http://www.conservationresources.com/)

- Synperonic N

From [www.flexolan.com/](http://www.flexolan.com/)

- Melamine resin foam (Basotect® **V3012**)

From classic supermarket

- Nivea cream

### *Vegetable tanned leathers*

- Sheep skin, quebracho tannin, sulfited oil lubricant: [www.lieutard.com/](http://www.lieutard.com/)

– Sheep skin, quebracho tannin, fish oil lubricant: [www.azais-megisserie.com/](http://www.azais-megisserie.com/)

*Silver leaves*

Berta: “thickness 30” reference, dimension 85 × 85 mm: [www.bertha-battiloro.com/](http://www.bertha-battiloro.com/)

Dauvet: dimension 80 × 80 mm: [www.dauvet.com/en/](http://www.dauvet.com/en/)

Manetti: dimension 95 × 95 mm: [www.manetti.it](http://www.manetti.it)

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### How to cite this article:

Radepon, M., C. Bonnot-Diconne, L. Robinet, S. Thao, C. Pacheco, M. Bouttemy, and E. Delbos. 2017. Impacts of manufacturing and conservation on the silver leaf tarnishing in gilt leather. In *ICOM-CC 18th Triennial Conference Preprints, Copenhagen, 4–8 September 2017*, ed. J. Bridgland, art. 0603. Paris: International Council of Museums.