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## Abstract

The display of silver collections will often require careful consideration of aesthetic preferences with regard to surface finish, balanced against the resources required for cleaning and the potential for loss of surface material, detail or decoration. Hence, methods to reduce the rate of tarnish formation are a priority, particularly for collections containing a large number of highly polished decorative silver objects. This paper presents a holistic examination of the management of silver tarnish comprising five strands of investigation, assessing: subjective perceptions of tarnish from different curatorial perspectives and the subsequent requirement to clean objects; methods of passive and active control of internal showcase environments to mitigate tarnish formation; the impact of cellulose nitrate lacquers and the potential for silver nitrate cyanide formation; and the efficacy of sealed bags for the reduction or removal of tarnish during storage.

## INTRODUCTION

Tarnishing of silver presents issues in many heritage institutions. If an object is thick, has no plating or fine engraving, it is a resource issue as to whether it is cleaned sufficiently frequently. Many major museums essentially employ the equivalent of a full-time conservator to clean silver objects. If plating or fine engraving is present, an object can only withstand a certain number of cleans before value is lost. This paper presents a series of five discrete investigations aimed at furthering our understanding of the most important practical methods for managing and mitigating silver tarnish.

### Perception of tarnish and when to clean

The visual appearance of silver tarnish drives cleaning frequencies in many institutions. This has been investigated through the views of stakeholders (curators, conservators and scientists) combined with data mining of cleaning records. A variety of approaches have been assessed to slow the tarnish rate, if required.

### Passive control

Showcases, if well designed, can slow tarnish dramatically. The silver tarnish rate in one set of showcases has already been shown to have a roughly linear inverse relationship with the air exchange rate (Thickett et al. 2006). A further series of showcases was investigated in this study.

### Active control

Previous work has shown that incorporating sorbents in pumps is much more effective than passive deployment. Only desktop type cases with the absorbent near the seals have significant potential for making passive employment of sorbents work. Hydrogen sulfide has a very strong affinity for silver (Pope et al. 1968) and if air passes over the silver surface before reaching the sorbent, the surface very efficiently removes any hydrogen sulfide. With most showcase designs this will be the situation, with air leaking in from door seals. For deployment in pumps, a ventilation rate of approximately five times the air exchange rate of the showcase is most effective (Thickett and Short-Traxler 2010). The early stages of tarnish have been observed to be associated with dust particles (Thickett and Hockey 2002). Trials assessed the performance of pumps with both HEPA and activated charcoal filters.

## **Impact of lacquer**

Silver on open display, or in showcases with unsuitable environments, is often lacquered. Cellulose nitrate-based lacquers are most frequently used. The lacquers become less soluble on ageing, requiring harsher methods of removal. This limits the lifetime and always occurs before a loss of protection (provided a complete film has been applied). Lacquers should never be applied unless resources are in place to replace them at suitable intervals. The choice of lacquer is guided by aesthetic opinion and other potential lacquers have been rejected because of their unsatisfactory appearance. Recent research has indicated a potential for formation of silver nitrate cyanide when coated with, or displayed with, lacquer-coated objects (Ziegler et al. 2014, Eggert et al. 2019). Cyanide formation with cellulose nitrite has been investigated.

## **Storage**

Many historic houses open predominantly over the summer months. Valuable items, such as silver, are often stored securely over the winter months when there is limited public access. Heat-sealed Marvelseal 360 bags are presently used in such storage. The efficacy of this method has been assessed. Storage of tarnished silver in Corrosion Intercept bags has recently been reported to remove tarnish due to the greater thermodynamic stability of copper sulfide compared to silver sulfide. The potential of this system for winter storage was investigated, particularly if the reaction was fast enough to remove tarnish acquired during the display months over the cold winter months.

## **METHODS**

### **Perception of tarnish**

A series of tarnished silver coupons were produced by exposing cleaned silver coupons (Robinet and Thickett 2004) to different museum atmospheres for two months to five years. This produced a wide visual range of tarnish levels. The coupons were kept in heat-sealed Marvelseal bags until use. The coupon surfaces were measured with a colorimeter (Minolta 2600D). A variety of stakeholders involved in cleaning decisions – curators, conservators and participants in an advanced European preventive conservation workshop (45 in total) – were asked to rank the coupons by how tarnished they appeared. The ranking was done in daylight with a sheet of white paper at 45° over the coupons to reduce reflections. Each participant was asked to indicate the point at which cleaning should occur. The ordering was compared to colorimetric values recorded on the coupons' surfaces.

The conservation records for silver objects since 1972 were assessed to determine the average cleaning times in different curatorial departments in The British Museum.

### **Passive control**

Tarnish rates (increase of  $b^*$  on silver coupons) were measured for three other sets of showcases inside showcases and at ambient room levels for

12 months to incorporate seasonal variations. The showcase air exchange rates were measured using carbon dioxide decay (Thickett and Stanley 2008). In some cases, silver objects were displayed and those with suitable flat areas were also measured with colorimetry. Melinex masks aligned with the design on the objects were used to accurately reposition the colorimeter head for each measurement.

### **Active control**

A series of showcases with different air exchange rates (AERs), geometries and silver loading were fitted with proprietary pumps (Emcel fan-assisted carbon/HEPA) incorporating both HEPA and activated charcoal filters. Some of the showcases were historically important, dating from the 1840s. Narrow bore tubing was fed through existing holes. Clean air from the pumps, on top of the cases, was introduced through the tubing. Silver tarnish rates in the cases and rooms were measured with colorimetry on silver coupons and AirCorr loggers.

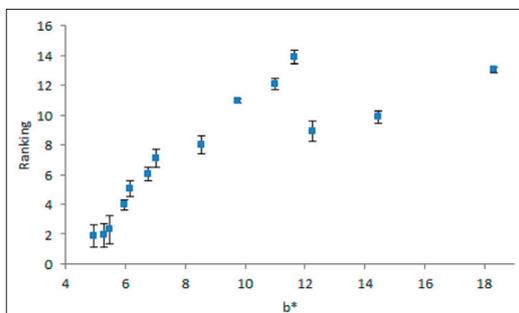
### **Impact of lacquer**

The surfaces of several hundred silver objects on display had previously been examined with Fourier transform infrared (FTIR) microscopy (Nicolet Inspect IR) (Thickett and Hockey 2002). Silver cyanide nitrate has a characteristic spectrum and these previous analyses were re-examined to ensure its presence was not missed. In the previous work, the formation of silver cyanide nitrate was looked for using spectral searching. At that time, only the spectrum, and not the identity and importance of the corrosion product, was known and all the spectra were thoroughly re-examined.

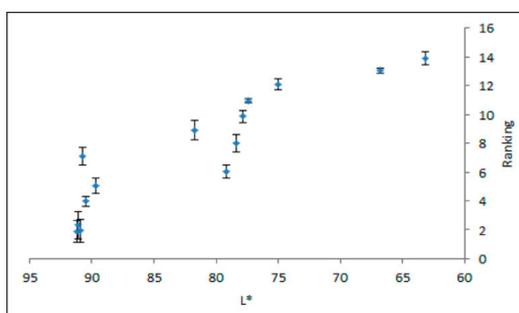
The atmosphere in 15 showcases containing high surface areas of Frigilene-coated silver objects were assessed. Silver-coated glass slides were exposed for 12 months. The coupons were clipped to the underside of glass shelves, with the silver-coated surface pointing downwards to minimise dust deposition. After exposure, any dust was removed by attaching the slide to a vibratory mill. The silver surface was analysed using an Amplif-IR accessory with a PerkinElmer 2000 FTIR with the distance between gold plate and sample set at 40  $\mu\text{m}$  (Thickett and Pretzel 2020). This produces an extremely surface-sensitive spectrum.

Linear stripping voltammetry was used to analyse over 200 silver coupons exposed in showcases at English Heritage previously. A sample of silver nitrate cyanide synthesised by Gerhardt Eggert was analysed with this method in a glassy carbon electrode. The voltammetry was run in a three-electrode cell, with a 0.1 M sodium nitrate electrolyte, a saturated silver/silver chloride reference electrode and a Palmsens potentiostat.

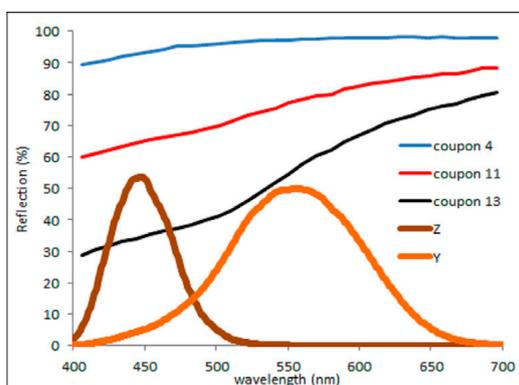
Over 40 silver coupons, many from showcases with high concentrations of Frigilene-lacquered silver, were examined with an Atomica secondary ion mass spectrometer (SIMS). The full conditions have been previously published (Thickett 2001). Eleven coins were being cleaned and re-lacquered for display. The Frigilene lacquer was applied between 8 and 14 years ago and was removed with acetone swabs. The cleaned coin surfaces were



**Figure 1.** Average coupon ranking against  $b^*$



**Figure 2.** Average coupon ranking against  $L^*$



**Figure 3.** Reflectance spectra of the representative coupons and CIE 1931 colour-matching functions Y and Z

then examined with SIMS in static and dynamic modes. The dynamic SIMS area was dropped to 0.05 mm square. Additionally, the depth of the microscopic ablation pits was determined with a Zygo Newview 200 microscope.

### Storage

The tarnish rate of silver, in heat-sealed Moistop and translucent oxygen impermeable (Escal) bags, was measured with Air-Corr loggers. The oxygen concentration was also measured in the Escal bags with a PreSens Fibox 4 meter. Samples of black felt, known to rapidly tarnish silver (Green and Thickett 1993), were also placed in two Escal bags.

Silver coupons exposed in several English Heritage locations for 12 months were placed in Corrosion Intercept bags. Details of the environments have been previously published (Thickett and Costa 2014). The change in  $b^*$  was measured over different periods, from 4 to 12 months. The bags were placed in three locations to provide different temperature conditions: a fridge at 7°C, a basement store running at 10°C–17°C and a storeroom with temperatures ranging from 17°C to 24°C.

## RESULTS

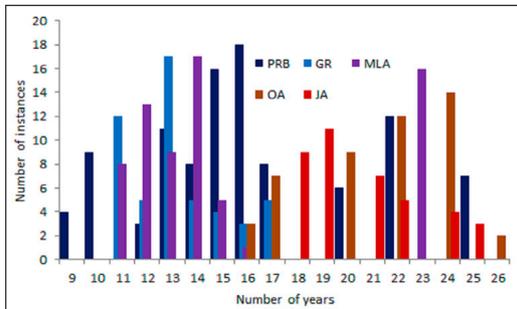
### Perception of tarnish

The results of the perception tests are shown in Figures 1 and 2.

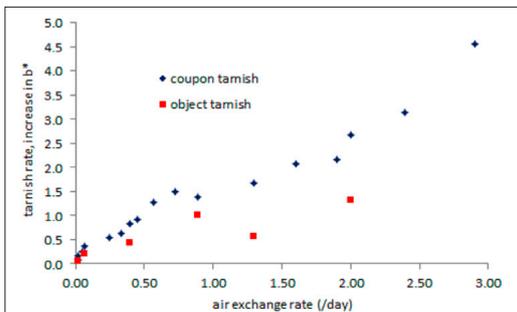
In the following text, coupons are numbered by the average ranking assigned. At very low tarnish levels (coupons 1 to 3), the standard deviations were very large, indicating a random distribution of results, as the tarnish was not perceivable to the human eye. Coupon 3 had a higher average value, perhaps indicating the onset of perception. For the early stages of tarnish,  $b^*$  better correlated with the visual ranking (coupons 4–8). For more tarnished coupons, this broke down, a coupon 9 had a higher standard deviation, indicating more uncertainty. Figure 2, which shows the rankings versus  $L^*$ , has a better correlation for the higher ranked coupons (10–14). These results are consistent with the colour space used. Very little variation in  $a^*$  was observed, allowing  $b^*$  to be treated as an independent value. Several studies have shown that a  $\Delta E$  colour change of around 1.5–2 is the limit of human perception (Witzel et al. 1973, Billmeyer and Saltzmann 1981, Boyce 1987, Pretzel 2008), which translates to  $b^* < 5.5$  in these instances. Figure 3 shows the visible reflectance spectra for coupons 1, 6 and 9 with the CIE 1931 colour matching functions Y and Z plotted under D65 illumination.

The early stages of tarnish produce a reduction in the low nanometre range of the reflectance spectrum of silver (coupon 6, Figure 3). This early stage of tarnish is best described by Z, which has a strong impact on  $b^*$  calculated using the CIE system. As the tarnish progresses, an absorption peak around 520 nm begins to appear. This correlates well with Y, which solely defines  $L^*$ .

The points at which the participants decided that the level of tarnish would trigger cleaning are shown in Table 1.



**Figure 4.** Cleaning frequency distribution by department



**Figure 5.** Tarnish as b\* versus AER

**Table 1.** Coupon with tarnish at a point at which participants thought the silver should be cleaned

Coupon ranking	Number who selected this coupon		
	Whole cohort	Western Greek and Roman / Medieval and Later / Prehistoric and Romano British	Eastern Oriental Antiquities / Japanese Antiquities
6	8	8	
7	16	16	
8	6	6	
9	4	4	
10	3	2	1
11	3		3
12	3		3
13	2		2
14			

The cleaning frequencies from records are shown in Figure 4.

The two eastern antiquity curatorial departments, OA and JA, do not show any cleaning (records) before 16 years on display and the recorded cleaning activity mainly clusters between 17 and 24 years. The western departments mainly cluster at below 17 years, with 25% for PRB and 23% for MLA cleaned after greater time periods. There are several extra factors that determine cleaning frequency, such as resources and the opening of new galleries. However, the frequencies do show some correlation with the cleaning requirement assessments of the curators and conservators in those departments.

### Passive control

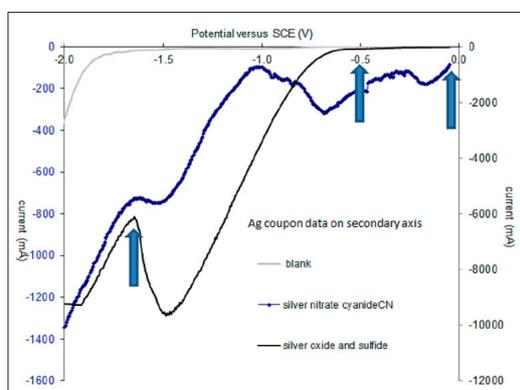
A series of approximately straight lines with different slopes were observed in plots of the silver coupon tarnish rate against AER (Figure 5).

The tarnish rates of objects were found to be lower in all measured instances, but again showed the same trend. Variations were found to correlate well with the differences between the silver surface area and the showcase volume. In cases with large surface areas of silver, the ingressing gases were spread over a larger area, hence the average tarnish thickness was reduced.

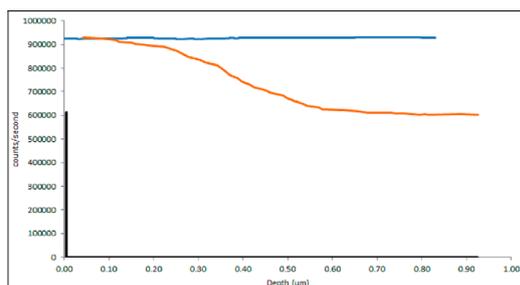
### Active control

Silver tarnish rates, measured with colorimetry on silver coupons and AirCorr loggers, are shown in Table 2.

The tarnish rates were decreased by at least 95% in all instances and more for some cases. The pumps require 24-hour power and small holes for the narrow bore tubing. It is better to spread the clean airflow over the showcase joins (door seals, etc.) and this can be achieved with splitter fittings. With 3 mm tubing there is no reduction in airflow over 3 m and less than 30% reduction over 6 m. With pumps, perhaps the main risk is the pump failing without this being noticed. The Emcel pumps have warning lights to change the sorbent when the pressure drop across them exceeds a certain value. This is due to dust blocking the HEPA filter. This appears to occur earlier than the chemical filter being exhausted. No instances of increased tarnish rate were measured with the Aircorr loggers over the



**Figure 6.** Potentiodynamic stripping curves for silver cyanide nitrate, sulfide, oxide and chloride



**Figure 7.** Dynamic SIMS profile for cyanide and sulfide in silver coins

**Table 2.** Performance of chemical and HEPA pumps

	Dimensions (m)	AER (/day)	Silver loading (m <sup>2</sup> /m <sup>3</sup> )	b* <sub>case</sub> /b* <sub>room</sub> (%)	Tarnish rate case/room (%)
Apsley 1	1.5 × 1.0 × 1.5	0.4	0.5	95.92	97.71
2	1.5 × 1.0 × 1.5	0.7	0.2	95.26	99.19
3	1.5 × 1.0 × 1.5	0.8	0.7	96.62	98.81
4	1.5 × 1.0 × 1.5	1.01	0.3	96.01	98.67
5	1.0 × 0.3 × 1.5	1.21	0.02	98.21	96.70
6	1.0 × 0.3 × 1.5	1.45	0.12	97.18	97.69
7	1.0 × 1.0 × 1.5	1.72	0.4	96.24	97.33
Wellington 1	0.8 × 0.8 × 2.0	1.1	0.3	96.09	98.41
2	0.8 × 0.8 × 2.0	0.8	0.1	96.16	97.80
3	0.8 × 0.8 × 2.0	1.2	0.6	96.47	99.49
4	1.6 × 0.8 × 2.0	2.5	1.2	97.42	95.93
Osbourne 1	0.37 × 0.8 × 2.2	0.4	4.1	95.72	95.99
2	0.45 × 0.35 × 0.45	1.6	2.5	95.74	99.29
3	1.0 × 0.6 × 0.33	0.2	1.2	95.47	95.22

periods prior to or during filter changes. Both filters are supplied in a single unit, so both are changed together.

### Impact of lacquer

No peaks due to silver cyanide nitrate were observed in the FTIR spectra from more than 500 silver object surfaces. Neither were they observed in the much more sensitive reflection absorption FTIR spectra from silver-coated glass slides.

Figure 6 shows the peaks due to silver nitrate cyanide along with characteristic peaks for silver oxide, chloride and sulfide.

The first reduction peak (onset  $-0.045V$  vs SCE) was unique for the silver cyanide nitrate. Re-examination of the previous linear stripping voltammetry results did not identify this characteristic peak in any of the analyses.

The static SIMS analyses of silver coupons identified several species present on the surfaces, a mixed tarnish layer, containing sulfide (S- negative ions at  $m/z$  32 and 33), sulfate (negative ions at  $m/z$  85 {SO<sub>4</sub>-} and 80 {SO<sub>3</sub>-}), chloride (Cl- at  $m/z$  35 and 37), organic carbonyl species (M/Z 58 {C<sub>3</sub>H<sub>6</sub>O-}, 59 {C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>-} and 73 {C<sub>3</sub>H<sub>5</sub>O<sub>2</sub>-}) and a range of positive organic mass fragments between C<sub>2</sub> and C<sub>6</sub> ( $m/z$  15, 28, 29, 41, 43, 53, 55, 57, 63, 67, 71, 73, 77, 79, 81, 83 and 85). No cyanide was detected.

Static SIMS for the coins identified similar species in five instances. Three coins also had mass fragments at  $m/z$  133 and 135 characteristic of cyanide ions.

Results for the dynamic SIMS analyses are shown in Figure 7.

Peaks, characteristic of cyanide, were observed in static SIMS spectra on three coins. These disappeared rapidly when dynamic SIMS was used. No peaks due to nitrate ( $m/z$  47 {NO<sub>2</sub>-} and 63 {NO<sub>3</sub>-}) were observed. Assuming the ablation is linear, the cyanide was present in the four uppermost surface atomic layers only. The other nine coins had no cyanide present, despite in four instances having had Frigilene on their surfaces for a longer period. All the coins had been displayed in the same location.

No evidence was found of silver cyanide nitrate being produced on clean silver surfaces in environments with Frigilene-lacquered silver present. For the small number of object surfaces examined after Frigilene lacquer removal, cyanide was found on three. However, no nitrate was observed, which would be expected if silver cyanide nitrate were present. Cyanide was not found on the other nine objects despite four having had Frigilene present on their surface for a longer period and in the same environment. The conservation records provided dates for the last cleaning and lacquering but were uninformative as to the cleaning method used. Cyanide-based dips had been disposed of from that departmental workshop in the 1990s. It is more likely the cyanide detected is the residue of an old cyanide stripping treatment and not related to the Frigilene lacquer. Other authors have reported cyanide present on cleaned Frigilene-lacquered silver surfaces (Pouliot et al. 2013). Here no nitrate was detected, as would be expected for the presence of silver cyanide nitrate. It is difficult to envision other likely sources of silver cyanide on objects. Cyanide is thankfully rare in the environment, and there are no obvious other sources in museum operations, beyond facsimile production.

### **Storage**

In all the impermeable heat-sealed bags tested, silver tarnish reactions dropped to below detection limits (0.2 nm/30 days) within two days of sealing the bag. The oxygen concentration dropped slightly from 20.8% to 19.7% but stayed constant after that.

Even with known sulfide sources present, the tarnish process halted rapidly. Tarnish levels measured after 18 months had not risen above the Aircorr's detection limit of 0.1 nm in any of the Escal or Marvelseal bags. The exact mechanism needs further investigation and is not due to oxygen depletion. However, the empirical result is significant and indicates this is a very effective storage strategy for mixed media objects that would be problematic in closed storerooms.

Results for the tarnished silver in the Corrosion Intercept bags are shown in Table 3.

The results were quite variable. Some of the tarnished samples lost tarnish rapidly, shown by a decrease in  $\Delta b^*$ . Others were not affected within the 12 months, even at the highest temperature location. Where the coupons were affected, temperature had a strong effect. This difference in behaviour may be due to differences in the composition of tarnish produced in the different locations. High chloride deposition rates and, to some extent, high ozone concentrations seem to generate more resistant tarnishes. However, that produced at Audley End was very resistant and both values were relatively low, so other factors must be involved.

### **CONCLUSION**

For human perception, it appears that  $b^*$  corresponds well with the early stages of tarnish, once a certain amount has been produced, and  $L^*$  with the later stages. There is some overlap – rankings 11 and 12 – where either colorimetric co-ordinate correlates with the visually observed order.

**Table 3.** Measured  $b^*$  values after 12 months storage in Corrosion Intercept

Tarnished silver from	Temperature stored at (°C)	$\Delta b^*$ before	$\Delta b^*$ after	Max Cl deposition rate (mg/m <sup>2</sup> /day)	Max ozone (ppb)
errors		5%	5%	20%	15%
Brodsworth	7	3.55	0.52	1.6	12.1
	10–17		0.24		
	17–24		0.12		
Audley End	7	3.10	3.08	1.3	5.0
	10–17		3.11		
	17–24		3.12		
Apsley Dining	7	4.04	0.02	9.1	0.7
	10–17		0.02		
	17–24		0.01		
Apsley Waterloo	7	1.05	0.12	1.0	5.7
	10–17		0.08		
	17–24		0.02		
Apsley Plate	7	6.43	3.21	15.6	8.9
	10–17		2.87		
	17–24		2.24		
Rangers	7	3.10	0.14	3.8	1.9
	10–17		0.05		
	17–24		0.01		
Walmer	7	3.43	3.44	94.4	7.1
	10–17		3.45		
	17–24		3.42		

Curators and conservators working in ‘western’ antiquity departments seemed to decide silver needed cleaning at an earlier stage than those working in ‘eastern’ antiquity departments.

Large differences in cleaning rates were found between the western and eastern curatorial departments, and these roughly correlated with the cleaning requirement assessments of the curators and conservators in those departments. These differences probably reflect a different aesthetic approach, with western departments preferring bright, reflective silver.

A series of linear drops in the tarnish rate in tighter showcases was observed in passively controlled showcases. This will help guide showcase design, with the costs of producing a tighter showcase being balanced against increased conservation resources to keep silver clean.

Pumps, removing both fine particles and tarnishing gases, have caused an impressive reduction in silver tarnish rates and their practical limitations have been elucidated.

The formation of silver nitrate cyanide in several real situations has been studied. Formation on objects displayed with cellulose nitrate-lacquered objects seems extremely infrequent, with no instances detected in over 740 measurements. A number of extremely sensitive analyses of objects after lacquer removal were undertaken. Caution should be exercised due to the small sample set. Some, but a minority of, instances of ultra-thin layers of silver cyanide were detected on the surfaces of objects with lacquer cleaned off. Over 75% of the objects did not show any cyanide on their surfaces (with the detection limit conservatively estimated at less than 1/20th of a molecular layer), with many having been lacquer

coated for longer periods than those with cyanide present. The absence of nitrate in the SIMS was unusual and may indicate a different source, possibly cyanide-based dips.

It was shown that storage in impermeable bags such as Marvelseal stopped silver tarnish. It appeared to stop tarnish even when sulfide generating materials are present, such as in mixed media objects.

Storage in Corrosion Intercept can remove tarnish from some, but not all, objects. Trials are continuing in order to understand the long-term behaviour and whether the resistant tarnishes will eventually reduce. Further work to understand the differences in the tarnish that render it more resistant to this extremely promising method would be beneficial. Both methods have obvious utility in the longer-term storage of silver.

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