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Examination and experimentation: conservation of an archaeological glass unguentarium for display

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SUMMARY A Roman unguentarium (No.1851,0813.493) excavated in Apt, France was assessed during a conservation survey of the glass reserve collection of the Department of Greece and Rome at the British Museum. On examination the unguentarium was highlighted as a very unusual example, because of its size, shape, colour and decoration. It was therefore decided to place it on permanent display and a complex programme of conservation and investigation was instigated.

Before conservation, the lower section of the object was covered in a dense gap fill that was fracturing and obscuring the fragile glass. X-radiography showed a complete profile under the fill, which enabled the object to be dismantled and reassembled; the missing areas were then filled for stability and display. Examination and scientific analysis using optical microscopy, scanning electron microscopy with energy dispersive X-ray spectrometry and Raman spectroscopy helped to identify materials and inform the conservation project. When the unusual, historic fill was analysed to aid its removal it was found to contain large quantities of mica and lead; the poisonous nature of the lead meant that the unguentarium had to be dismantled under carefully controlled conditions.

In addition to reassembling the glass elements of the vessel, large gap fills were needed to integrate the remaining fragments and to support the relatively intact upper part of the vessel. After reviewing the gap-filling methods available and conducting experiments with materials and techniques, a procedure was developed that involved: moulding and modelling the missing areas using polyester paste; creating a two-part mould of the polyester fragment; casting a translucent, tinted epoxy gap fill from the mould; and reassembly of the vessel fragments and gap fills so that the unguentarium was ready for display.

Introduction

The Department of Greece and Rome (G&R) at the British Museum holds a large collection of archaeological and historical glass [1]. The collection reflects the majority of types of glass production and manufacture in the classical world and includes masterpieces such as the Portland vase [2]. Much of the collection is in storage but remains available for study and handling.

In 2001 a conservation survey of the G&R archaeological glass reserve collection was undertaken to assess its overall condition [3]. Those objects identified as requiring conservation were gradually added to the conservation work programme over a number of years. In 2009, the remaining objects, including the unguentarium, were brought to the ceramics and glass studio, where preliminary examination raised interesting questions about the nature of the object and its restoration. After consultation between the conservator and curator, it was decided to undertake a programme of investigation, analysis and re-restoration.

The unguentarium

The unguentarium forms part of the Comarmond Collection and was purchased by the British Museum in 1851 from Ambroise Comarmond (1786–1857), the director of the Musée des Beaux-Arts de Lyon, France. The vessel was excavated in Apt, France and is made of amber blown glass with concave, marvered sides and a bulbous end decorated with chips of white glass. However, there are no records to show exactly how the vessel appeared when it was purchased by

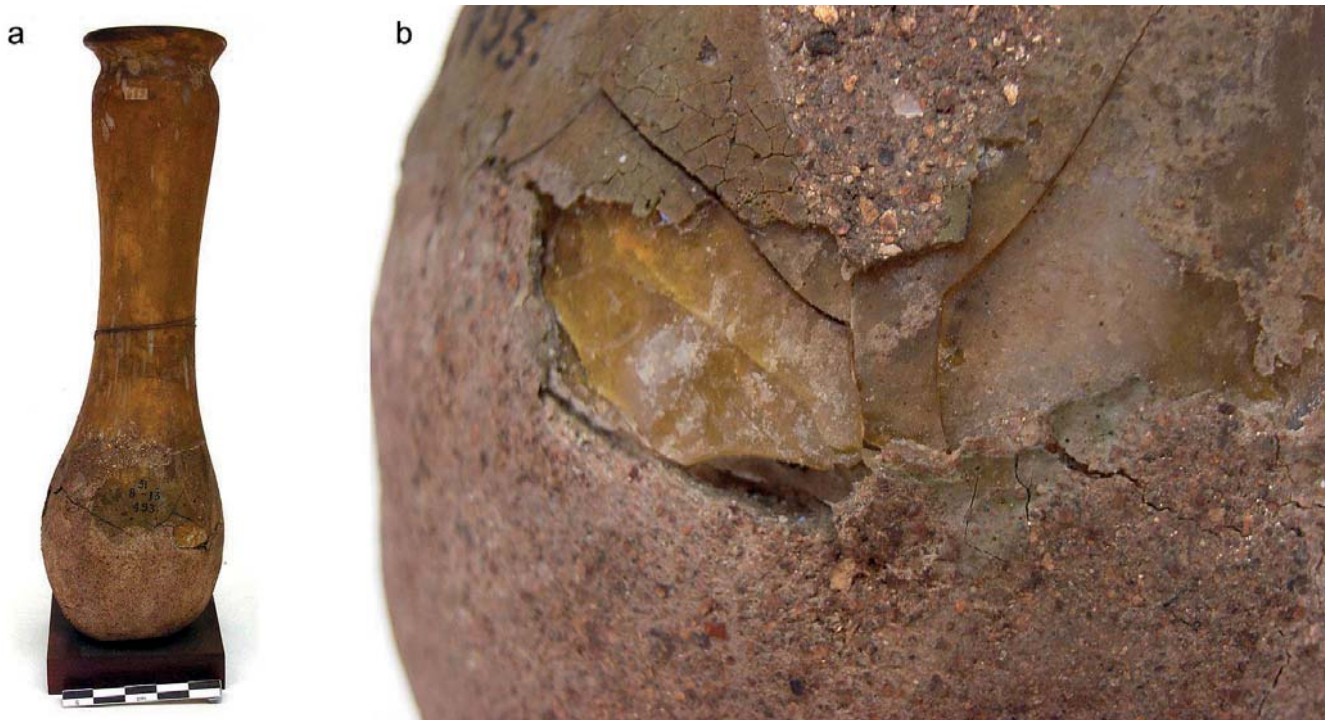

Figure 1. Unguentarium (1851,0813.493) showing: (a) the whole object before treatment (20 cm tall, 4.6 cm wide at the rim); and (b) detail of fractures and lamination caused to the glass by the old gap fill

the British Museum, and it is not known if the object has been displayed or restored since 1851 or whether it has sat untouched in the Museum's collection.

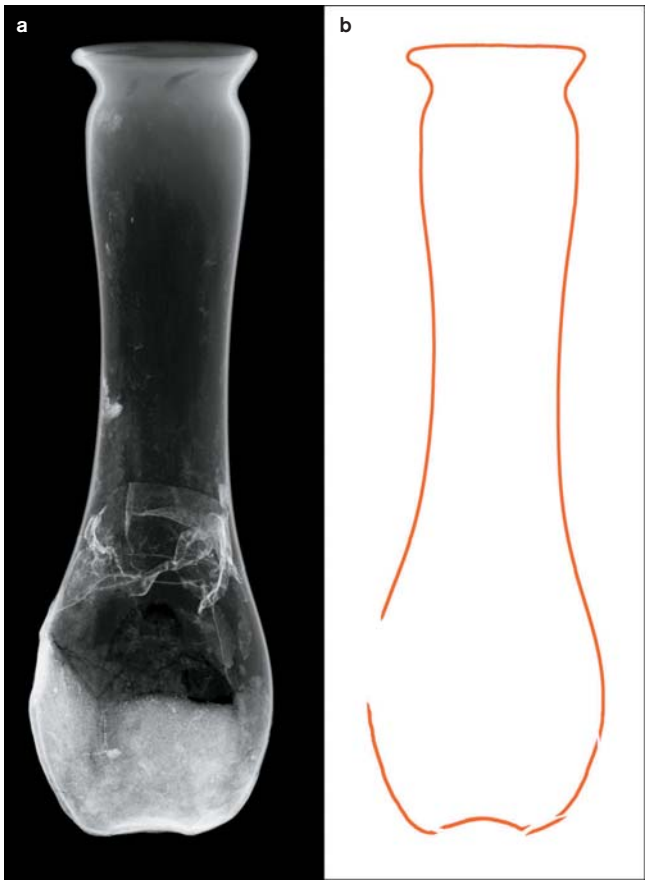

Figure 2. (a) X-radiograph of the unguentarium; and (b) diagram showing the profile of the vessel beneath the previous fill

The vessel was formed using a simple glassblowing technique. Soft molten glass would have been gathered on the end of a blow pipe, rolled on a marver (a slab of polished stone or iron) with chips of opaque white glass, then reheated and blown to the desired shape. During the blowing and shaping process the white opaque glass would extend and lengthen, producing the design that is visible on the unguentarium [1; pp. 223–226].

Unguentaria were commonly used to hold perfumes. Roman perfumes, which were made by steeping flowers in oil, were expensive and so the unguentaria made to hold them were usually small; glass was ideal for this purpose as it did not absorb the perfumed oil [4; p. 134]. This raises questions about the exact use of the unguentarium under discussion, since its large size makes it unusual; unfortunately no residues were found within the vessel that could be analysed to determine what it might have stored.

Condition prior to treatment

A very dense material coated the lower section of the object, but it was unclear how much of the original glass was covered, Figure 1a. There was evidence that this fill (referred to hereafter as the 'previous fill') was stronger than the glass as in places it was possible to observe that it was causing the glass to laminate and fracture, Figure 1b. Dirt and soil from burial coated the inside of the object, which was packed with thick paper and tissue paper. It was unclear whether the previous fill had been applied as an aid to excavation of the unguentarium or was a later restoration.

Scientific examination and analysis

Examination and analysis first focused on establishing whether there was a complete profile of the vessel preserved under the previous fill, and on identifying the constituent materials of

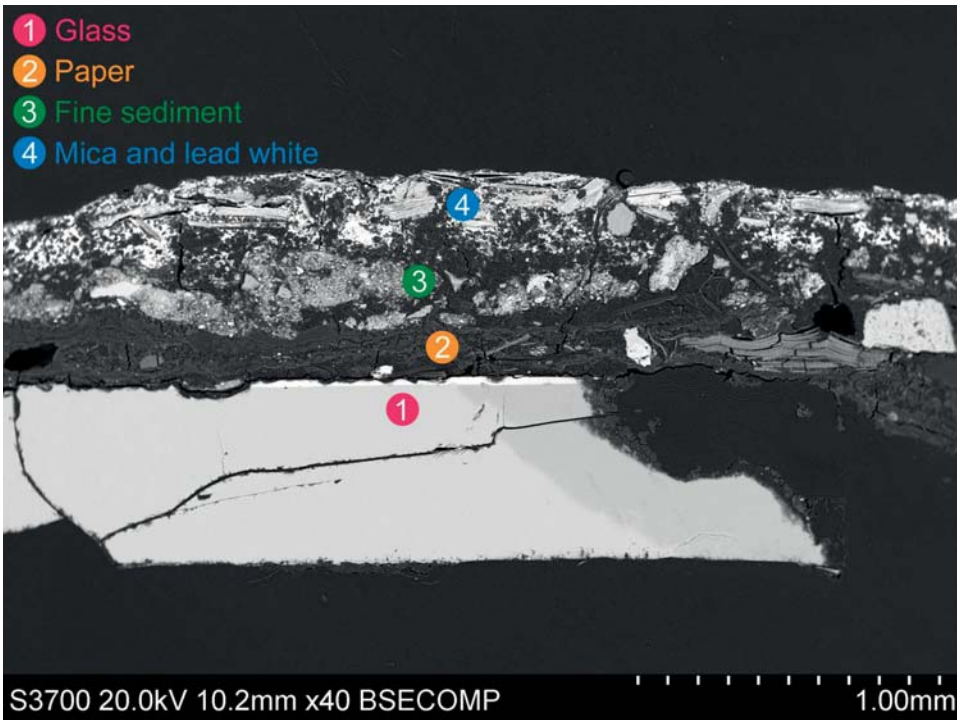

Figure 3. Image made in BSE mode in the SEM of the prepared cross-section of a sample of the previous fill and the attached glass fragment

this fill to inform and aid the conservation treatment. Had the vessel lacked a complete profile, this would have markedly affected the proposed treatment as it was considered to be unethical to attempt to recreate the missing areas without a complete profile [5; p. 242].

Vessel profile

X-radiography was carried out to investigate whether a complete profile of the object was present, Figure 2. Different exposures and orientations were used in order to capture the state of repair and level of restoration of the unguentarium and to try to obtain further information on any residual original contents; see the experimental appendix for details.

In the X-radiographs (Figure 2a), it was possible to observe that a continuous profile could be made from the fragments, Figure 2b. Once this was established, the removal and investigation of the previous fill began.

The X-radiographs also revealed considerable information about this fill; it was obviously very opaque to X-rays, suggesting that it was made from a dense material or a material containing elements with a high atomic number, Figure 2a. Finally the X-radiographs showed the extent of the paper packed inside the unguentarium.

Previous fill

A portion of the previous fill that was loose and laminating from the other areas was removed for sampling; it had a small, displaced flake of glass attached. The materials used in earlier treatments or restorations were investigated using a combination of optical microscopy, scanning electron microscopy with energy dispersive X-ray spectrometry (SEM-EDX) and Raman spectroscopy, see the experimental appendix.

Small fragments of the previous fill were first investigated using optical microscopy and then analysed by Raman spectroscopy; these samples required no preparation prior to analysis. Optical microscopy suggested that the lowest layer in the fill, closest to the glass, was composed of a fibrous, paper-like material and a white adhesive. The external surface of the previous fill consisted of fragments of a golden-coloured tabular material embedded in a light grey coating that lay on top of the lower fibrous layer, Figure 1b.

As it was impossible to locate the original position of the glass flake, it was used for SEM-EDX analysis. The fragment of glass, to which layers of the previous fill still adhered, was mounted in MetPrep EPO-SET epoxy resin, polished with diamond paste to a 1 μm finish, coated with carbon and analysed. The sample was imaged in backscattered electron (BSE) mode, which provides a monochrome image that reveals differences in composition as differences in lightness; the higher the atomic number of an element the lighter it will appear in the image.

An image of the prepared cross-section of the fill made using the SEM in BSE mode suggested that it consisted of three layers (Figure 3), rather than the two layers identified using optical microscopy. The dark appearance of the lower layer (Figure 3, layer ②) suggests that the materials contained in this layer have a low atomic number. While there are other possibilities, this result is consistent with the presence of paper and an organic adhesive. Due to the difficulties involved in separating this layer from the middle layer without contamination it was decided that further analysis of its constituents would prove complex and potentially inconclusive.

Qualitative EDX analysis of the middle layer (Figure 3, layer ③) suggested the presence of various minerals within a fine sediment, possibly a clay-like substance, surrounded with an organic adhesive. However, more precise analysis was not possible due to problems in preparing such a friable sample for quantitative SEM analysis and imaging.

Table 1. Quantitative SEM-EDX analysis of the vessel glass

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	FeO
Unguentarium	18.5	0.5	2.6	68.5	0.1	0.2	1.4	0.7	7.2	nd	nd	0.3

Note. The data are expressed as weight percentages (wt%) of the oxides of the elements normalized to 100%; 'nd' indicates the element was not detected.

SEM-BSE imaging of the uppermost layer suggested that its composition was similar to that of much of the middle layer. However, it also contained large fragments of a laminar material and another finer material, which appeared very light in the BSE images (Figure 3, layer ④) indicating that an element with a high atomic number is present.

Areas of the uppermost layer were analysed qualitatively using EDX spectrometry. The laminar material, which was seen to have a tabular crystal morphology under the optical microscope, was found to contain magnesium, aluminium, silicon, potassium and iron. These, combined with its laminar structure and appearance, suggest that it is probably a mica. The matrix in which these mica particles are embedded was also analysed using SEM-EDX and found to be high in lead. Raman spectroscopy confirmed the presence of the pigment lead white and traces of hematite within this layer. Hematite was present in such small quantities that it is not believed to be an intentional addition.

These results suggest that the previous fill was constructed in a three-stage process. First, paper was attached to the fractured surface of the vessel using an organic adhesive. This was then covered with a clay-like substance bound in with adhesive. Finally, a layer of adhesive containing lead white and mica particles was applied, the purpose of which may have been to imitate the colours and optical properties of the glass vessel.

This is not a standard method of restoration and neither the authors nor their colleagues have previously observed its use on a glass vessel. It seems likely to have been carried out around the time of the vessel's discovery or breakage in order to keep the fragments in place.

Glass composition

To investigate its composition and provenance, the fragment of vessel glass within the prepared cross-section (see Figure 3, layer ①) was analysed quantitatively using SEM-EDX; the results are presented in Table 1. The unguentarium was produced from soda-lime-silica glass. The low magnesium and potassium levels (less than one weight percent) indicate that the glass was produced using a mineral form of soda known as natron. This compositional type is common to the majority of Roman glass objects analysed previously [6]. Based on the compositional information it is not possible, therefore, to suggest a particular date range within the Roman period or place of production for the object.

The amber colouration is due to the presence of iron and develops when glass is melted in an oxidizing environment. It was not possible to analyse the white opaque glass present on the object as it is not represented in the fragment described above and there were no suitable sites from which to take a further sample.

Conservation treatment

As X-radiography had revealed a complete profile, it was decided that the previous fill could be removed and the unguentarium made suitable for display by dismantling and reconstructing the fragments of the glass vessel, then inserting gap fills that rendered the object stable. These new gap fills would not only need to support the object but would also be required to be easily reversible for the safety of an object that, as is often the case for archaeological glass, is particularly fragile [7].

Dismantling

The principal reason for removing the previous fill from the object was that it was causing the surrounding glass to fracture. Because of the toxicity of some of the materials in the fill, it was dismantled and removed in a controlled environment using appropriate personal protective equipment (PPE) and adhering to current health and safety guidelines for working with lead [8]. The removal of the fill and dismantling of the glass were carried out simultaneously. To soften the organic adhesive used to adhere the paper to the glass in the previous fill, warm water was applied to the layers of paper with a soft sable brush. The water was allowed to penetrate and slightly soften the layers before they were gently lifted off with a spatula and cut away with scissors. Although the paper and tissue lodged inside the object were examined carefully after they had been removed, they yielded no further information.

A small part of the old fill was saved for future reference as an interesting example of a historical repair material [9; pp. 74–75]. This fragment was sealed inside two plastic bags and labelled accordingly with appropriate hazard labels; information about its origin and lead content were entered into the Museum's database to inform future conservators or other researchers.

After the unguentarium had been dismantled it was clear that the object was top heavy and that the glass at the rim was thicker than the base where, at its thinnest, it measured a mere 0.5 mm. The larger fragments were laid out into a shard map during the dismantling process as their correct location was apparent, while the location of the smaller fragments was not immediately obvious, Figure 4.

Initial reconstruction

To establish the size and complexity of the missing areas prior to gap filling, sections of the vessel were reassembled by dry bonding using strips of 3M Scotch Magic Tape (a cellulose acetate carrier tape with an acrylic adhesive). Due to their very small size (less than 5 mm) the smaller fragments and flakes were handled using a combination of suction tweezers, entomology tweezers and a wooden cocktail stick tipped with Groomstick (modified natural rubber). These small flakes were bonded together temporarily with Loctite Super Glue 3 (cyanoacrylate adhesive) to create groups of fragments that could then be dry bonded to the larger sections [10; p. 194].

Reconstruction was made more difficult by the thinness of the glass (less than 1 mm in some areas), the limited contact area between fragments and the complicated and irregular joins. It was clear during the dry bonding stage that sections

Figure 4. The fragments of the unguentarium laid out in a shard map after the vessel had been fully dismantled

of the object would next need to be bonded temporarily with an adhesive so that the joins did not move during the process of creating accurate gap fills. Two acrylic adhesives – HMG heatproof and waterproof adhesive (cellulose nitrate) and Paraloid B72 (ethyl methacrylate copolymer) – were tested, but both were found to be too weak to hold the fragments in place. It proved to be difficult to align the fragments correctly when joining them edge-to-edge and neither adhesive produced a strong enough join. Although not normally considered, a specialized epoxy resin (Fynebond) was used temporarily to bond the fragments into sections that could then be taped to the relatively intact upper half of the unguentarium [5; p. 263]. However, even with this epoxy resin, which was introduced into the joins by capillary action, it proved difficult to join some of the fragments because of the very small contact surfaces.

Choice of gap filling

Gap filling is used in conservation to recreate lost areas of an object to support the structure of the object or for aesthetic reasons. There are various methods for creating gap fills, depending on how the object will be stored, handled and interpreted in the future [7; p. 289]. For the unguentarium the range of different established methods of gap filling considered included:

- Japanese tissue or nylon gossamer impregnated with Paraloid B72 [11; pp. 205–206];
- epoxy resin sheet, cut and shaped while semi-cured [12; p. 29];
- slumping a partly cured epoxy sheet onto a former for final cure of the resin [12; p. 30];
- an *in situ* epoxy gap fill [13; pp. 76–78];
- making a removable polyester fragment that is then moulded and cast in epoxy resin [7; pp. 290–291].

The last of these methods was considered most appropriate for the unguentarium, but needed to be adapted to account for the thinness of the glass and the complexity of the missing areas; in particular, a lip was created around the edge of the fill to support the glass fragments and to enable them to be joined into position for extra strength and support.

Making the polyester fragment

There were two areas that required gap fills for support. It proved necessary to make the smaller of these removable fills first and to tape it into place temporarily to allow work to begin on the larger, more complex fill.

A barrier layer was used between the glass and the polyester to prevent the polyester resin from bonding to the glass [9; p. 125]. Tests were made on a number of barrier layers to find the most suitable; Parafilm M, cling film and aluminium foil were all too thick or did not give good contact with the glass [7; p. 290]. Following the tests a silicone, Elite Double 22, was chosen as the barrier material. It is a two-part silicone rubber, which is mixed in equal parts, has low viscosity, sets quickly and attaches well to glass. Elite Double 8 silicone was also tested but was found to give a barrier that was too thin and tore easily. The break edges of the missing areas and surfaces adjacent to them were coated with a thin layer of Elite Double 22 applied with a wooden toothpick, Figure 5a. A very thin layer acted as a good barrier that was easily removed from the surface after moulding. The silicone barrier layer did not appear to leave any residues, but as a precaution the surface was swabbed with acetone (propanone) after it had been removed.

A thin sheet of pink dental wax was heated with a hot air blower and backed with a sheet of cling film to prevent the filling material from sticking to the wax. This laminate was then softened by reheating and placed onto an intact area of the original object with the correct profile to mould the outer shape, Figure 5b [13; pp. 80–81]. The shaped wax was then placed over the missing area, cut to size slightly larger than the void and held in place with Elite Double 22 silicone and strips of Magic Tape, Figure 5c.

Figure 5. The process of modelling the polyester paste fragment, illustrated using a missing area of a Pyrex beaker: (a) Elite Double 22 silicone rubber was applied as a barrier to the break edges and inside and outside surrounding surfaces; (b) a pink dental wax support slightly larger than the missing area was positioned over the gap; (c) the barrier and wax support were removed and inverted to allow the polyester paste to be applied from the inside; (d) preparing the polyester paste; (e) applying the polyester paste to the missing area on the wax support and across the protected surrounding surface area to create a lip; (f) removing the silicone barrier layer; and (g) the cured polyester fragment removed from the Pyrex beaker, showing the lip by which the surrounding glass fragments will eventually be supported

Polyester paste was chosen to make the intermediate gap fills because of its working properties; it sets within four to five minutes, can be polished to a high finish and can be used to create very thin fills. One disadvantage of very thin polyester fills is that they are prone to tearing during polishing, although the tears can be repaired with Loctite Super Glue and refilled with polyester. The polyester paste currently in use at the British Museum is David's Isopon P38 [14; p. 69]. All filling, abrading and polishing of the polyester resin was carried out in a fume cupboard adhering to appropriate health and safety guidelines. The paste was mixed on a glazed ceramic tile in a fume cupboard and applied into the missing area using a spatula, Figure 5d. The fill was extended over the edges onto the protected glass surface to create the lip to support the thin glass, Figure 5e.

The gap fill was left on the object to cure, which prevented warping of the very thin polyester fragment, and the whole object was kept in the fume cupboard during this off-gassing stage. To remove the fill, the different sections of the unguentarium that had been joined temporarily were next dismantled. The barrier layer was easily removed by gently lifting from the glass with fingers and tweezers, and any residues were removed with acetone applied on a soft brush, Figures 5f and 5g. The fragment was abraded with a Dremmel drill, then polished with increasingly fine grades of Micro-Mesh (cushioned abrasive cloths with silicon carbide and aluminium oxide) until a smooth, polished surface was achieved.

Moulding the fragment

A two-part mould was made of the intermediate polyester fragment using Silcoset 105 (a silicone rubber). The half of the mould for the outside surface of the fragment was made first, using a glass tile as a base. The inside of the polyester fragment was supported on a bed of modelling wax (Scopas white modelling wax) that was smoothed using a silicone brush. The wax was applied along the bottom of the fragment edge so that all of the break edges could be moulded. Keying holes were pushed into the wax surface using the rounded end of a paintbrush. The thin tips of plastic pipettes were embedded into the modelling wax at the highest parts of the fragment to act as entry and exit holes for the resin during casting. Barrier walls to contain the silicone rubber during the moulding process

Figure 6. The process of moulding the polyester fragment, illustrated using a missing area of a Pyrex beaker: (a) the polyester fragment was supported on modelling wax; (b) a thin layer of silicone rubber was applied to the barrier walls, wax and fragment; (c) microballoons were added to the silicone rubber used for the second layer; (d) applying plaster as a 'mother' mould on top of the silicone rubber; (e) showing the distinct layers of the first half of the mould after the mould walls had been removed; (f) heating the wax to soften and remove the entire mould without disturbing the polyester fragment; (g) applying Vaseline as a barrier layer to the silicone mould; (h) applying silicone rubber to mould the inner surface of the fragment; and (i) the polyester fragment seen in the opened mould

were built up using glass microscope slides and the flat lids from clear polystyrene boxes, which were sealed together using the modelling wax, Figure 6a.

A thin layer of silicone rubber was poured into the mould to cover the whole surface of the polyester fragment, wax and barrier walls; any air bubbles that came to the surface were immediately pricked with a pin, Figure 6b. This first layer was left to partly cure for three hours, taking care not to disturb the silicone rubber, as it would form the surface of the final cast. To add strength to the mould, the second layer comprised silicone rubber bulked with microballoons (phenolic resin or glass) to produce a thicker consistency, Figure 6c. The mould was half filled with this mixture with the air bubbles again removed as described above. After the silicone had been left to cure overnight, a layer of superfine casting plaster was applied on top of the rubber to make a 'mother' mould, Figures 6d and 6e.

To prepare the second half of the mould (for the inside of the polyester fragment), it was necessary to invert the first half. A hot air gun was applied to the underside of the mould to soften the modelling wax so that the mould could be removed in one piece, Figure 6f. The mould was inverted and attached to the glass plate. The modelling wax was softened again and gently removed to reveal the inner surface of the polyester fragment. It was essential not to disturb the position of the polyester fragment while making the second half of the mould as this would create an inaccurate mould that would in turn affect the final epoxy cast.

A separating barrier layer of Vaseline (petroleum jelly) was applied to the silicone rubber using a silicone brush to achieve a consistently thin layer and taking care to avoid the polyester fragment, Figure 6g. Barrier walls were rebuilt around the silicone rubber mould and held in place with modelling wax. The second part of the mould was made using the same process described above, Figure 6h. Once all the components had dried the polyester fragment was removed from the silicone mould (Figure 6i), which could then be reassembled without the fragment [9; pp. 132–134].

Casting the gap fill

After a discussion between the curator and the conservator, the decision was made to colour the epoxy gap fill so that it blended with the original amber glass to create a visual coherence for display that was not distracting. Araldite 2020 epoxy resin was chosen to make the casts, as it is relatively inexpensive and large quantities can be prepared easily. Samples of coloured epoxy for testing were made by experimenting with the addition of different amounts of artists' dry ground pigments, Maimeri restoration colours (bound in mastic and turpentine) and Golden fluid acrylics, Figure 7a [14; pp. 103–106]. After a suitable colour had been selected, a batch for casting was made by adding the colourants to approximately

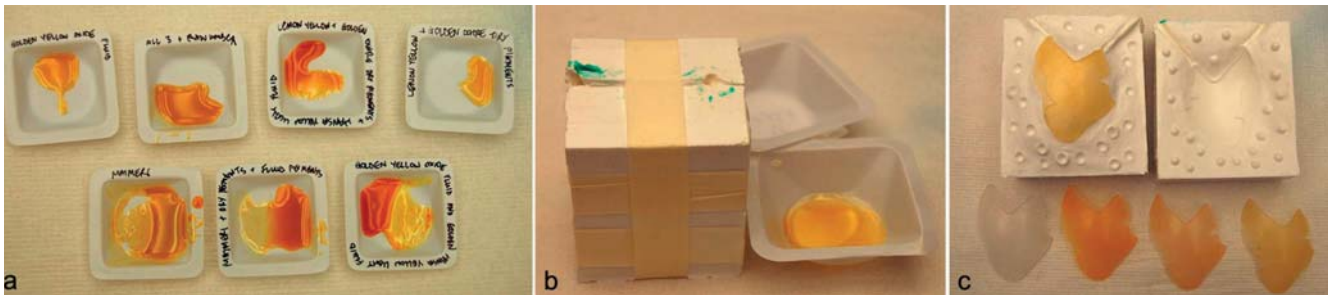

Figure 7. Casting the gap fill for the unguentarium: (a) testing pigments and paints to colour the epoxy resin; (b) showing the taped-up mould after it had been filled, with a sample of the tinted resin left to cure alongside to give an indication of the curing time; and (c) the small gap fill for the unguentarium cast in different coloured resins.

Figure 8. The unguentarium after conservation: (a) seen in full profile with the gap fills bonded into position; (b) a detail of the gap fills on the base showing their complexity; and (c) a detail of the lips on the gap fills (the slightly darker areas) into which the glass fragments register and which provide additional support

Figure 9. The unguentarium on display at the British Museum alongside other Roman glass objects

200 g of Araldite 2020 epoxy part A (epoxy). The required quantity of hardener (part B) to give a ratio of 3:1 by weight was added [13; p. 88].

Although the moulds could have been sealed with silicone rubber to prevent any leaks, because they were to be used several times it was decided to close them with strips of masking tape pulled tightly around the mould, Figure 7b. The mixed Araldite 2020 epoxy resin was introduced into the silicone mould one drop at a time using a wooden cocktail stick. The mould was gently tapped and agitated to dislodge any trapped air, which rose to the surface as bubbles. The mould was filled until resin was present in the pour and vent holes, Figure 7b. The resin was left in the mould for longer than the recommended curing time of 48 hours to allow for the presence of pigments and for the fact that the resin was completely enclosed, since each of these factors might be expected to extend the curing time [15].

Final reconstruction and fitting the gap fill

After curing, the epoxy cast was removed from the mould and any excess cut away with a scalpel, Figure 7c. The epoxy cast had a slightly cloudy appearance when released from the mould, so a small amount of clear epoxy resin was rubbed over the surface with a tissue and allowed to cure [7; p. 290]. This was then polished with Micro-Mesh fine abrasive cloth. The small gap fill was made first and held in place on the object with Magic Tape while the larger support fill was made using a three-part silicone rubber mould.

The sections of the unguentarium that had been bonded temporarily with Fynebond and Super Glue 3 were dismantled, cleaned and the edges degreased with acetone. Hxtal NYL-1 epoxy resin was chosen as the final resin for bonding the unguentarium due to its favourable long-term ageing properties. The fragments were fastened together using strips of Magic Tape and the resin was applied by capillary action [16; p. 220]. The epoxy gap fills were bonded into position with Paraloid B72 (Figure 8a) and, to complete the treatment and make the unguentarium ready for display, the remaining very small fragments were bonded directly to the epoxy fills, Figures 8b and 8c.

Conclusions

Because the unusual previous fill that covered the lower section of the object was causing it to fracture, its removal was required for the future physical safety of the object. Once scientific analysis had determined that the old gap fill contained quantities of lead, additional precautions were taken to ensure the safety of the conservator during its removal. X-radiography showed that there was a complete profile under the old fill, which allowed the object to be dismantled, reconstructed and gap filled for display.

More conventional methods of gap filling used at the British Museum were tested and found to be inappropriate for this object, which led to the use of a three-stage process of modelling, moulding and casting. A barrier layer of Elite Double 22 was found to be most effective for creating a thin barrier layer to protect the glass while an intermediate polyester fragment was made. The polyester was applied not only to the missing area but also to the surrounding inside surface to create a lip to which the glass fragments could be joined for strength and support. The polyester fill was moulded using a standard two-part silicone rubber mould and the epoxy resin gap fills that were cast from this mould gap were tinted amber to blend in with the original colour of the glass.

The object was placed on permanent display at the British Museum, alongside objects showing similar decorative techniques, in December 2012, Figure 9.

Experimental appendix

SEM-EDX

Analysis was carried out using a Hitachi S3700 VP-SEM under the following operating conditions: high vacuum mode: spectral range 20 kV, 0–10 keV; probe current 2.3 nA; counting time 180 s. The analyses were calibrated against pure element, mineral and oxide standards; a Corning A glass standard was analysed to verify that this calibration was producing accurate results. The detection limits were calculated using a spectrum synthesis programme within the Oxford Instruments INCA Analyser software. This analytical method resulted in detection limits for most metal oxides of around 0.1–0.2%.

Raman spectroscopy

A Horiba Infinity Raman spectrometer with a green (532 nm) laser was used. Measurements were made on fragments of the old restoration fill with no sample preparation. The spectra that were produced were compared with reference spectra from a British Museum in-house database and published data.

X-radiography

The X-radiographs were made using a TORREX TRX 5200 radiation-shielded X-radiographic/fluoroscope inspection system with a maximum voltage of 150 kV and operating at a standard 3 mA. Agfa Structurix D4 film was loaded into a film cassette that was fitted with a lead backing sheet and exposed for either two or three minutes at 80 kV, or two minutes at 65 kV.

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Materials and suppliers

- HMG cellulose nitrate, Paraloid B72, Araldite 2020 epoxy resin and Hxtal NYL-1 epoxy resin: Conservation Resources UK Ltd, Unit 2, Ashville Way, Watlington Road, Cowley, Oxford OX4 6TU, UK, www.conservationresources.com
- David's Isopon P38 polyester paste: widely available for car repair.
- Elite Double 22 silicone: Zahn Laboratories, Medcare House, Centurion Close, Gillingham, Kent ME8 0SB, UK, www.henryschein.co.uk
- Dental pink modelling wax: Wright Health Group, Canal Court, Brentford, London TW8 8JA, UK, www.wright-cottrell.com
- Scopas white modelling wax: Tiranti Ltd, 27 Warren Street, London W1T 5NB, UK, www.tiranti.co.uk
- Parafilm M: Whatman International Ltd, Springfield Mill, James Whatman Way, Maidstone ME14 2LE, UK, www.whatman.co.uk
- Silcoset 105 silicone rubber: ACC Silicones Ltd, Amber House, Showground Road, Bridgwater, Somerset TA6 6AJ, UK, www.acc-silicones.com
- MetPrep EPO-SET epoxy resin and hardener, MetPrep Ltd, Curriers Close, Charter Avenue, Coventry CV4 8AW, UK, www.metprep.co.uk

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