Cupel and crucible: the refining of debased silver in the Colonia Ulpia Traiana, Xanten

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Excavations in the Colonia Ulpia Traiana in the Archaeological Park Xanten (NW Germany) produced a group of finds related to the refining of silver: cupellation hearth material, crucible fragments and crucible slags. Chemical and mineralogical analysis of these finds has led to the reconstruction of a hitherto unknown process of silver refining involving the re-use of litharge or cupellation hearth material from a previous cupellation. It was fused with sand in crucibles to form a lead oxide slag, to which debased silver scrap was added. Tin as the most deleterious impurity in debased silver, together with zinc, went to the slag, while copper, silver and probably lead formed a ‘bullion’ regulus underneath the slag at the bottom of the crucible. The possible use of the slag produced in this operation as an enamel material will be discussed, based on a few finds from elsewhere. The archaeological context demonstrates that this refining took place in a private workshop located in a craft quarter of the settlement.

Introduction

The Roman Empire saw several periods of significant debasement of its silver coinage, followed by various attempts to re-establish a reliable silver-based currency. Silver debasement was also a matter of concern for local craftsmen working the metal. While the monetary silver stock was allowed to drop to ever lower levels, though still being closely controlled, silver used in tableware remained at a rather high degree of fineness even during the later Roman period. Smaller items of jewellery, by contrast, exhibited a wide range of compositions, though still mostly at a higher level than the more heavily debased coinage. It is generally assumed that the refining of debased silver was done by cupellation, resulting in a silver with as little as 1% copper and lead, though to increase the mechanical properties of the silver it was usually alloyed with c.5% copper by weight.

Recent excavations in the Colonia Ulpia Traiana have produced archaeological evidence for how a silversmith far from primary sources of metal dealt with the problem of debased silver, namely by immersing it in a lead-silica bath prior to cupellation. The first step, carried out in a crucible, served to reduce the content of deleterious elements present, notably tin, while collecting the silver in a ternary copper-silver-lead alloy. During cupellation this alloy is then remelted under a draft of ambient air. The lead and copper oxidize to cupriferous litharge, while the silver becomes more and more enriched, unaffected by the attack of oxygen. Cupellation is typically conducted on a hearth lined with porous materials such as marl or bone ash which readily absorb the metal oxides. The silver metal, having a much higher surface tension than the oxides, stays on top of the hearth, from where it is collected once the process is completed. The ease and success with which the cupellation could be performed depend greatly on the composition of the bullion used. Both tin and copper are deleterious elements when present in too high concentrations relative to lead. Severe debasement of the silver, in particular if bronze is used instead of pure copper, therefore increases the difficulties experienced later in refining the metal. The hearth material, soaked with the oxides of lead, copper, and any further base metals from the charge, is discarded and may eventually make its

Fig. 1. Map of the Colonia Ulpia Traiana; the finds originate from insula 39 at lower right.
way into the archaeological record.

Cupellation goes back to the Early Bronze Age and is still a commonly-used method in noble metal assaying. Initially a means of primary silver production, cupellation became also a refining process, notably during the Roman period. From the late Middle Ages onwards it also served as an analytical technique to determine the noble metal content of a given sample. Since the basic technique is always the same, the identification of finds from a given excavation may sometimes be a problem. To allow proper assignation of related finds, J. Bayley and K. Eckstein\(^4\) have developed a scheme to differentiate between cupellation débris from primary production, recycling and assaying. While the material found at Xanten accords well with other Roman finds (e.g., from Silchester or the Rhineland\(^5\)), being high in copper relative to lead and with significant silver inclusions, the step of crucible melting is unique to this site.

**Colonia Ulpia Traiana (Xanten) and the context of the finds**

Colonia Ulpia Traiana is situated just north of modern Xanten on the left bank of the Rhine some 40 km south of the Dutch border. A little to the south-east, and in sight of the legionary fortress of Vetera II, is the confluence of the Lippe with the Rhine. The course of the river is accompanied by a wide levée above which the Roman colony lies on a higher piece of ground.\(^6\)

The town with 40 \textit{insulae} was founded probably in A.D. 99. Although reduced in size to 16 central \textit{insulae} at the end of the 3rd c., its smaller successor (Tricensimae) probably survived into the opening years of the 5th c., if the sparse pottery evidence is any indication. Continuing excavations at Xanten are dictated by the concept of an Archaeological Park, comprising some 40% of the Roman town of c.73 ha. The main emphasis has been placed on the structures of the \textit{colonia} itself. Following extensive excavations near the town wall on the east, it was expected to find an artisans’ quarter in \textit{insula} 39 (fig. 1) that would shed light on everyday life. This presumption was based on the presence in \textit{insula} 38 of a hostel with attached bath-house, and on the proximity to the harbour. Recent investigations\(^7\) have shown that \textit{insula} 39 possessed direct access to the shipping lane, thereby offering good opportunities for commerce and trade.

The first trenches in \textit{insula} 39 revealed only sparse traces of buildings on the E side of the \textit{insula}. Following the levelling of the preceding settlement, construction of buildings of the \textit{colonia} was begun at the start of the 2nd c. A few foundations of bricks and tile surrounded a large courtyard which probably served for the delivery and storage of goods. Later, the \textit{insula} was divided into plots. First, an L-shaped structure was built in the middle of the N side. Several decades later further buildings were added to this structure. Shops and manufacturing establishments fronted onto the \textit{decum anus} between \textit{insulae} 38 and 39. The living quarters were situated at the rear or in an upper storey. A small tannery or dye-works, a drying-kiln for grain and flax, as well as a silversmith’s workshop prove that small-scale manufacturing activities were carried out. The houses were renovated, extended and remodelled well into the 3rd c. Since the relevant layers are found either just under or even within the modern ploughzone, conditions of survival are poor. The material discussed here was found mostly in the courtyard of one of the buildings and in layers dating to the 2nd c.


\(^7\) D. Charlier and S. Leih, “Der Flußhafen vor der Colonia Ulpia Traiana,” in \textit{Archäologie im Rheinland} 1995 (Köln 1996) 54-56.
Fig. 3. Crucible slag fragment (no 37) from *insula* 39. The full profile with the original melt surface and the countermark of the regulus at the bottom is visible here. Note the many gas bubbles near the top and the thin ceramic wall. See also in colour after p. 240.

**Physical description of the material**

*Cupellation hearth material*

Three pieces of convex-concave shape, roughly 2 cm thick and some 5 x 7 cm square, were identified as cupellation hearth material, the originally porous lining of a cupellation hearth, soaked with lead oxide. Their density is remarkable, more than twice that of stone or ceramic. The dull gray appearance clearly distinguishes them from metal finds. The upper, concave surface is sprinkled with black incrustations. The lower surface is smooth and the edges are irregular and broken. The interior is dark brown with some metallic inclusions in a macroscopically homogeneous matrix (fig. 2 in colour following p. 240). The shape of the cupellation hearth material is functional, resulting from the lining of a hearth. A semicircular rim, partly preserved on some fragments, indicates the size of the silver button which formed on top.

*Crucible slag fragments*

Fourteen major lumps of crucible slag have been found up to now, all solidified within individual crucibles. They often have parts of the crucible walls still adhering to them, and larger pieces show the original upper and/or lower surfaces of the slag, or both (fig. 3 in colour). The typical thickness of the slag ranges from 4 to 6 cm, the upper diameter of the cakes being 4 to 7 cm. Fragments vary in size from a few grams to massive lumps weighing more than 250 gr. The slag is glassy and dense, with vesicles sometimes occurring near the upper surface and in the central part. Colors range from dull brown to bright red, typically with a thin coating of green corrosion products (fig. 4 in colour). Small inclusions of metallic prills, i.e. tiny spheres, are ubiquitous, and several samples show partly resorbed grains of quartz floating in their upper parts.

*The crucibles*

The crucibles are mostly made of a coarse, light-colored ceramic, tempered with quartz. The material is similar to the local coarse ware. Only two samples differ, consisting of a very thin, fine-grained ceramic, but
Fig. 6. The most complete smelting crucible yet found in insula 39. The vessel is filled with slag to about half its volume. Note the regulus countermark at the very bottom and the second layer of less refractory clay only in the lower half of the crucible. The spout has been used, as can be seen by a thin interior cover of slag, just visible at the rim.

of the same whitish color. Most of the crucibles are of the shape typical of later Roman crucibles though with a significantly thicker bottom, while others are apparently re-used domestic ware, fitted with a secondary, outer layer of clay to adjust them for the specific use. Except for the thicker bottom, neither the shape nor the fabric of the crucibles seem to be related specifically to this process and will not be discussed here.

Function-related information can be gained by looking at the way the crucibles broke after use. Three classes of crucible fragments were found. The bottom pieces are usually conical in shape, with a wall thickness of more than 1 cm, and covered externally by a second layer of dark, less refractory and heavily vitrified clay (fig. 5). The inner surface of these sherds is only slightly slagged with a thin film of yellow glass. The middle fragments are almost always intimately fused to massive lumps of slag. The wall thickness decreases here to about 5 mm, while the second, outer clay layer normally tapers off. Finally, the upper wall and rim pieces are c.8 mm thick, with a padded edge and clearly visible striations from turning on a wheel. The exterior is free of adhering material, and the inner surface mostly shows only splashes of slag. The one complete rim contains a spout (fig. 6) which is covered internally with a thin film of slag from pouring.

The main type of crucible to be reconstructed from these fragments is pear-shaped with a thick conical bottom, the lower half wrapped by an outer layer of less refractory, darker ceramic. The opening is about two-thirds of the maximum width, and at least one vessel was equipped with a spout. Flat bottoms do occur but less frequently, indicating the use of local domestic ware. Despite the overall similarity in the shape of the conical-bottomed crucibles, they differ significantly in their actual size and in the ratio of height to width. Every crucible seems to have been made individually. The charge always solidified in the crucibles, resulting in the need to smash the vessels to recover the metal, even though the one preserved spout had obviously been used.
Crucibles of this kind, whether purposely-made pear-shaped or re-used flat-bottomed domestic vessels, and often covered with an external secondary ceramic layer, are quite typical of Roman base-metal workshops, but they do not exhibit such a thick base nor do they bear more than a thin film of internal slag.  

The reconstructed metal charge

The lower ends of the slag pieces never take the form of the crucible bottoms but show a very flat and smooth surface with some downward bending at the edges. This is interpreted as the countermark (meniscus) of a liquid metal bath of high surface tension resting under the slag cover. The crucible bottoms, with only a thin film of glassy slag covering the inside, were all found empty. Thus there must have been a metal pool underneath the slag that solidified to produce a regulus easy to remove. The shape and size of the reguli were defined by the conical internal profile of most crucible bottoms and the flat interface of the metal's upper surface to the liquid slag, resulting in an inverted cone. The composition of this metal can be estimated from the numerous prills scattered in the slag as a bullion of roughly equal parts of copper and silver, with some lead.

Chemistry

The cupellation hearth material

The cupellation hearth material consists mainly of lead and copper oxide; silica, lime, and phosphorous are minor elements, while all other elements analysed were below 1%. The first seven elements in Table 1, silica to phosphorous, are interpreted as residual initial hearth-lining material, while the base and noble metal oxides are due to the cupelled charge. The data are very similar to analyses of Roman cupellation hearth material reported earlier, all being characterised by a high overall copper content. Earlier (Bronze Age) and later (mediaeval) such finds, by contrast, typically have less than 1 wt% copper oxide.

<table>
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<tr>
<th>Sample</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>MgO</th>
<th>CaO</th>
<th>P₂O₅</th>
<th>CuO</th>
<th>PbO</th>
<th>SnO₂</th>
<th>ZnO</th>
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<td>0.9</td>
<td>0.2</td>
<td>0.4</td>
<td>6.1</td>
<td>0.4</td>
<td>13.0</td>
<td>69.8</td>
<td>0.3</td>
<td>0.04</td>
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<tr>
<td>28l</td>
<td>8.2</td>
<td>1.0</td>
<td>0.9</td>
<td>0.3</td>
<td>4.5</td>
<td>4.2</td>
<td>7.5</td>
<td>71.0</td>
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<tr>
<td>45l</td>
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<td>0.2</td>
<td>0.3</td>
<td>4.4</td>
<td>1.9</td>
<td>13.5</td>
<td>67.3</td>
<td>0.2</td>
<td>0.03</td>
</tr>
<tr>
<td>49u</td>
<td>5.1</td>
<td>0.5</td>
<td>0.1</td>
<td>0.2</td>
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<td>74.0</td>
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<td>9.4</td>
<td>75.3</td>
<td>0.2</td>
<td>0.03</td>
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TABLE 1: CUPELLATION HEARTH MATERIAL

Chemical composition of cupellation hearth material and crucible slag fragments from a silversmith's workshop at Xanten. The cupellation hearth material has been analysed separately for the upper and lower half of each sample (sample numbers XXu and XXl, respectively). All data in wt% calculated as oxides, including copper, which occurs partly in the metallic state. Detailed silver values are not given due to this element's unhomogeneous distribution. Ag was found between 100 and 10,000 ppm for both cupellation hearth material and crucible slag fragments. Na₂O and K₂O were present in most samples between 1 and 2% each, Ni and As below 100 ppm, Sb around 300 ppm. All results were calculated as oxides, although some of copper is present in its metallic state. Data are Flame Atomic Absorption and Inductively Coupled Plasma Spectrometric analyses from 50-100 mg samples.


9 Gowland (supra n. 5), Bachmann (supra n. 5).
The crucible slag fragments

The main components of the slag samples are lead oxide and silica with c.50% and c.20%, respectively. Lime scatters around 10% and copper oxide around 7%, while the other oxides analysed, including tin and phosphorus, fall between 0.5 and 2.5%. One sample (no. 46) contains unusually high amounts of silica, iron and tin and significantly less copper and lead; it is also unique in its brown color. Macroscopically similar metallurgical lead-silica slags, colored red by copper oxide precipitates, do exist\(^{10}\) though not in this period nor with such high silver contents as our material. Roman red lead glazes and enamels, by contrast, have a very different, soda-rich, base composition\(^ {11}\) and are generally 'cleaner' in respect to minor elements.

Despite the significant differences in composition and appearance between cupellation debris and the crucible slag, they are believed to be related to one another. The main difference between them is the percentage of the main oxides PbO, SiO\(_2\) and CaO, the latter two being present at higher levels in the slags. This is interpreted as the result of the addition of quartz and lime to the cupellation hearth material, leading to a dilution of the lead content and an increase in silica and lime in the slag. Taking an average lead oxide content of 70 wt% for the cupellation hearth material, the addition of one-third of its weight of calcareous sand would reduce the lead oxide to 53 wt%, the average of the slag analyses (Tables 1-2). One-third by weight equals about 1 : 1 by volume, taking the density of the cupellation hearth material as three times the density of the sand, which would have been a convenient ratio for an ancient silversmith. The silica content in the slag is about three times that of the cupellation hearth material, while the lime content is roughly doubled; this indicates about a 50 : 50 mixture of sand and lime, taking into account the loss of weight due to the calcination of the lime. The increased values for alumina, iron, and magnesia in the slag indicate the use of marl rather than pure lime. Phosphorus is diluted to the same extent as lead is, which supports the idea of charge mixing: phosphorus originates only from the cupellation hearth material and thus should experience the same dilution as lead.

While mass balance estimations are consistent with the proposed model, this is not true for the differences in copper, tin, and zinc concentration levels. Copper is reduced in the slag as compared to the cupellation material, while the tin and zinc content of the slag is 5-10 times higher than that of the cupellation hearth material. The only reasonable source for the excess tin and zinc is some kind of base metal containing 'bronze' being added to the charge (i.e., the so-called gun-metal alloy containing tin, zinc and lead in varying amounts, as is typical for the period). The hypothesis of metal being added to the crucible charge is also supported by the volume ratio of slag to metal reconstructed from the better-preserved crucible bases and slag lumps. The volume of the regali is estimated to about 5-10% of the volume of the slag, while the metal content microscopically observed in the cupellation hearth material is less than 1%.

Mineralogy

Polished sections were mounted from slices of cupellation hearth material and crucible slag fragments. Phase identification was done by optical microscopy and EDX analysis. A JEOL 6400 with two wavelength dispersive spectrometers (WDX), controlled by a NORAN Task package, was used for quantitative microanalysis of selected phases. The detailed mineralogical data is presented elsewhere\(^ {12}\) so discussion in this paper is restricted to the information necessary for the reconstruction of the process.

The cupellation hearth material is a porous, highly crystalline material dominated by lead oxide phases. Inclusions of metallic silver and copper are finely dispersed throughout the material, making up for about 1% of the volume. Noteworthy is the occurrence of a mixed calcium-tin oxide phase near the top surface, partly clogging the porosity. The material has never been entirely fluid, but represents a liquid solidified in the porosity of a fine-grained solid matrix. The slag, by contrast, was once completely molten. It has a highly glassy matrix which owes its striking color to the cuprite, finely dispersed as feather-like dendrites.

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Depending on the actual composition and the degree of crystallinity, there are varying amounts of other, colorless phases typical for lead-rich slags. Metallic inclusions are omnipresent; they are always well rounded spheres. Their overall composition varies widely within the ternary system of copper-silver-lead: even drops immediately adjacent often differ dramatically.

The addition of lime and quartz to the charge has already been postulated in the light of the bulk chemistry; this model is further augmented by the higher silica and/or lime content of almost all the oxide phases present in the crucible slag fragments. The lead oxides of the cupellation hearth material are transferred into the calcium-bearing lead silicate glass of the slag; the lead calcium silicates appear now more calcareous. The most telling transition is that from the calcium-tin oxide found near the surface of the cupellation debris to the calcium-tin silicate found in the slag. Both phases are most uncommon and tie together the two groups of materials beyond any doubt.

The totally different texture of the two groups of materials is also in perfect agreement with this model; the cupellation hearth material obviously retained its solidity throughout most of the cupellation; the original porosity is still visible, now filled with lead oxides, and the crystal growth is one of solid state reactions, as known, for example, from metamorphic rocks. The crucible slag fragments, on the other hand, solidified from a nearly complete melt, with euhedral solidus phases and eutectoid structures in the residual melt; the metals had the chance to fuse and to unmix extensively from the slag, and the upper parts often bear floating grains of quartz no longer attacked by the melt already saturated with silica.

Discussion

The loss of silver during cupellation to the hearth lining has been known for a long time, and the technical literature is rich in admonitions and recipes to avoid cracks and fissures in cupellation hearths. The Renaissance mint-master and assayer Lazarus Ercker mentioned it explicitly, as do metallurgists almost 400 years later, and L. Davis reports implicitly the same phenomenon in her investigation into Romano-British silver production. On the other hand, the reworking of the débris of a gold- or silversmith’s workshop has been a common practice probably since the beginning of metallurgy. Clearly there are two complementary strategies to cope with the problem: to avoid the loss as far as possible and/or to rework the débris. The first strategy finds its natural limits in the properties of the materials used and the degree of care taken in their manufacture and use, while the second strategy is controlled more by socio-economic conditions: costs of labour, availability of commodities and fuels and the level of craftsmanship.

Until recently, we had only limited physical evidence for the execution of these strategies during Classical and earlier times — almost nothing beyond occasional grave finds with bags of collected scrap probably pointing to the reworking of débris. At Xanten we find them integrated in a complex process used for a primary refining of scrap silver — first to condition the metal, afterwards to rework the débris anyway. The most probable reason for refining prior to cupellation is to be sought in the tin content of the debased silver. Tin-bearing metal is more difficult to cupel due to the formation of high melting-point oxides which clog the porosity of the hearth and hinder the separation of litharge and silver metal. Therefore, the removal of tin prior to the cupellation is advantageous.

The ‘Xanten process’ appears to consist of the following. Cupellation of a copper-silver alloy led to the formation of a silver regulus on top of the hearth lining made of marl, clay and ashes. Some silver was trapped in fissures of that lining. Instead of laborously hand-picking these tiny bits of precious metal, the lining was crushed, mixed with the same volume of sand and marl and melted down in a crucible. The addition of silica and lime as a flux was necessary for several reasons: the charge became less corrosive against ceramic material, permitting it to be smelted in a crucible; the melting point of the charge dropped by nearly 200°C to a mere

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750°C; and the density of the slag forming was significantly lowered, helping the metal introduced with the cupellation débris to settle at the bottom within a reasonable time. At one stage of this process, fresh scrap silver was added, resulting in a bigger regulus, and a first raffination of the added alloy occurred by slagging its tin and zinc content. Eventually, the melt was allowed to solidify in the crucible, properly segregated into a top slag layer covering a metal regulus. Finally, the crucible was smashed to recover the regulus and the slag was dumped together with the crucible fragments. The refined copper-silver metal than went through cupellation to acquire the pure silver.

To judge by the number of crucibles used and the homogeneity of the slags (Table 2), the process looks to have been well developed and executed, yet the varying shapes and sizes of the crucibles indicate that it was only conducted on a small scale, without a need for the use of standardized vessels. This hints at a private workshop rather than an official establishment, as was already suggested by its setting within insula 39, an artisan and craft quarter. The small scale of the working is further indicated by the find of a spherical casting crucible (fig. 7).

14 More than a dozen individual crucibles used in this process so far have been identified from the excavated material. The total recovery rate is hard to determine, but it may have been low, as several larger fragments were found scattered in the modern plough zone.
Although no metal or slag traces were found in this obviously used crucible, one may assume from its small size and the lack of metal dross that a noble metal (i.e., silver) was melted in it.

The crucible slag as a possible enamel

The bright red opaque color and the low melting-point of some of the slag produced in this 'Xanten process' has been noted above. Given that this material occurred in a silversmith's workshop, one wonders whether it may have been put to some further, ornamental, use — e.g., as an enamel or for making of tesserae. Most published analyses of Roman enamels, though, place them clearly apart from the Xanten material and relate them to the contemporary glass industry. There are, however, two exceptions which make it worth pursuing the idea further.

Recently, some red enamels of the mid to late first millennium A.D. from Britain were investigated at the Department of Scientific Research, British Museum. C. Stapleton et al. have suggested that these enamels were produced using slags of the type found at Xanten. They differ significantly from earlier red enamels which are based on soda-lime-silica glass compositions with lower levels of lead and copper oxide. Using energy-dispersive X-ray analysis of samples removed from a core group of 11 enamels, supported by non-invasive surface X-ray fluorescence analysis of a further 30 objects, these Early Mediaeval enamels were found to be relatively pure lead silicates with high concentrations of copper and alumina; several also were shown to have trace levels of silver, and a copper-silver droplet was seen in one. Stapleton et al. point out that a very similar slag, containing copper-silver prills, was previously found by the British Museum group in crucibles from a Late Saxon site at Netherton, Hants. Based on these two sets of findings, they conclude that there is strong evidence that red enamel was being made from cupellation débris produced by a process similar to that at Xanten.

Less well documented are several objects reported from an excavation in Wange, Prov. Vlaams-Brabant (Belgium). Here a Roman villa dating to the 2nd and 3rd c. yielded remains of a large bronze scrap collection burnt in a destructive fire, which contained a set of enamelled bronze pins. Semi-quantitative analyses showed increased levels of lime, phosphorus and sometimes silver, again pointing to a material very similar to the Xanten slag. A more detailed study of the Belgian finds is necessary to establish this parallel with more certainty.

Conclusion

The investigation of crucible fragments and related finds from an artisan's courtyard in the 2nd-c. colonia has led to our reconstruction of what we may term the 'Xanten process' as a sophisticated means of reworking bronze-debased silver scrap. The process was conducted in crucibles as reaction vessels, in contrast to the more frequent melting of metals in crucibles which does not involve chemical reactions. While the metallurgical setting of this operation is beyond doubt, it may also be related to making red enamel as a by-product. The frequent occurrence of slag lumps at our site indicates that this was not a primary goal of the process, but evidence from other sites hints at this possibility, underlining the necessity for more systematic research into the relationship between the metallurgical and glassworking crafts.

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15 J. Henderson, "Chemical characterization of Roman glass vessels, enamels and tesserae," in Vandiver et al. (supra n.5) 601-7.
Rehren & Kraus fig. 2. Cupellation hearth fragment (no. 28) from insula 39 in the colonia. The front has been cut with a saw. Note the dark interior and the light corrosion rim. The metallic silver inclusion near the upper left-hand corner of the cut appears bright white, while the silver spots at the surface are covered by a black tarnish.

Rehren & Kraus fig. 3. Crucible slag fragment (no. 37) from insula 39. The full profile with the original melt surface and the countermark of the regulus at the bottom is visible here. Note the many gas bubbles near the top and the thin ceramic wall.

Rehren and Kraus fig. 4. Crucible slag fragment (no. 40) from insula 39. The bright red colour of the slag is converted into light green at the corrosion rim. The ceramic of the crucible is the usual coarse, quartz-tempered fabric. The second, outer layer of less refractory and dark material is heavily vitrified.