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Viking silver metallurgy. Determining the function of ceramic dish fragments from Sigtuna in Sweden & Fyrkat in Denmark

Angela Wallace

Small flat slagged ceramic dishes connected with metalworking, are known from several Scandinavian Viking period contexts. As part of a MSc thesis for University College London, a comprehensive archaeological survey was compiled and detailed scientific analysis carried out on two assemblages of fragments from three sites of 10th and 11th century date. Ten fragments came from Fyrkat in Denmark, and ten from Sigtuna in Sweden, while one complete dish from Trelleborg in Denmark was also included. The results are here summarised and some new findings and theories concerning the function of these dishes are presented based on the results of this study.

Characterising the studied material

The objects from Fyrkat, Sigtuna and Trelleborg allow for a good comparative study of a similar artefact type from contemporary regional and urban centres, that can provide evidence for shared cultural technological processes across the Viking region.

The Sigtuna assemblage consisted of ten fragments, all deriving from contexts connected with King Olof Eriksson's 10th–11th century mints or royal workshops. They consist of mainly diagnostic rim and rim and body sherds, and derive from flat ceramic dishes. These are commonly referred to as 'melting plates' in the literature and will be referred to as such in this work to avoid confusion. Two of them appear unused, the other eight have glassy or vitrified upper surfaces with splashes of red, green and black, while three fragments also have one or two small metallic prills visible within the vitrified layer. There was no vitrification in evidence on the bases of the plates.

The Fyrkat assemblage consisted of ten fragments, also from diagnostic rim, or rim-and-body sherds. These fragments were very similar in appearance to those from Sigtuna, with vitrification of various colours on the upper surface and the occasional visible metallic prills.

One complete melting plate from the Danish fortress of Trelleborg was also examined but not sampled. This plate was several millimetres thicker than many of the broken fragments and had no metallic prills visible within the slag that was concentrated on only one side of the plate – an important point to consider when analysing broken fragments of this artefact type, since many fragments that appear unused, could still belong to a used vessel. The slag was glassy and light greenish in colour, unlike the more colourful deep reds observed on some of the other fragments.

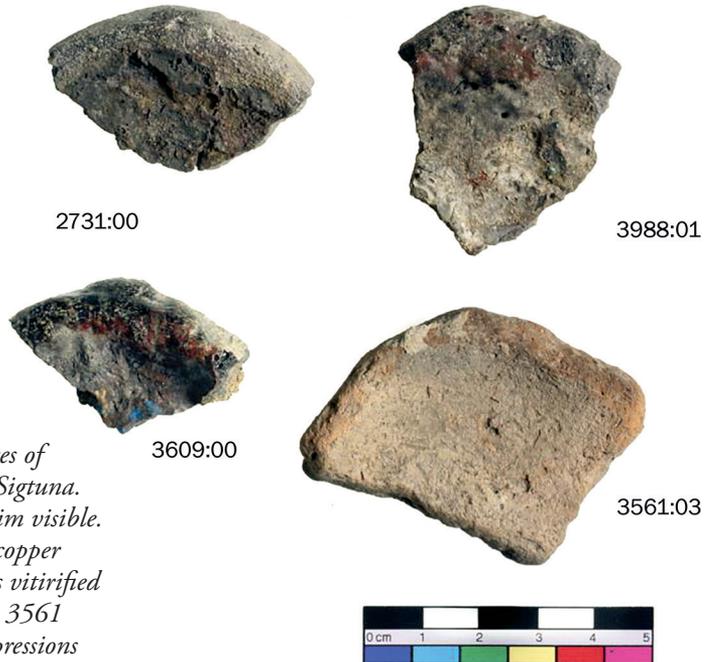


Figure 1. View of inner surfaces of melting plate fragments from Sigtuna. 2731 has a distinctive outer rim visible. Vitrification and small green copper prills visible on 3988. 3609 is vitrified across the entire inner surface. 3561 appears unused and plant impressions are visible on the fabric.

Previous interpretations regarding vessel function

There exist several theories regarding the function of these vessels, some based on the archaeological context from where the artefact was retrieved and also the appearance of tiny metallic prills adhering to the inner surfaces. The vessels have been described as heating/melting and soldering trays. They have been associated with jewellery manufacture, for filigree and granulation work (Hulthén 1995; Brorsson 1998), and have also been interpreted as used for melting metals prior to casting. Roesdahl was one of the earliest authors to link heating trays with refining as outlined by Theophilus (Hawthorne & Smith 1963), but later, based on experimental work, she



Figure 2. Image of several Fyrkat fragments.



Figure 3. Image of complete melting plate from Trelleborg.

suggested that filigree and granulation work was a more likely function (Roesdahl 1977:196). In many cases this artefact type has been recovered on high status sites associated with fine metalworking with precious metals.

The artefact type is found from the 6th century onwards throughout Scandinavia, Ireland and Britain. Flat-bottomed crucibles from Garranes in Ireland were believed to have 'been used for enamel and glass manufacture' (O'Riordain 1942). This interpretation is probably due to the glassy appearance of the inner surfaces. Flat-bottomed crucibles were also identified at 8th–9th century Dunadd in Scotland, where it is suggested they were used for soldering or to collect filings and trimmings of precious metals (Lane & Campbell 2000:134).

It was recognised by Lamm that open crucibles from the 6th century site at Helgö in Sweden were heated from above, linking this to the use of blow-pipes. A small iron blowpipe was found at Helgö. 'Traces of gold are preserved on four whole and seven fragments of open crucibles at Helgö' (Lamm 1980:103). Lamm has drawn the conclusion that they were used for refining or assaying (Lamm 2008:191ff). In 8th century Hamwic, gold was detected on one vessel, while other examples linked with gold working are known from 10th century York, (Bayley 1992a; 1996) and also at Winchester (Bayley & Barclay 1990). Analytical evidence from York suggests that these flat-bottomed ceramic dishes are more commonly associated with silver, but are also associated with gold. One of the aims of this study was to determine if the collection for analysis contained any evidence for gold.

Initial analytical work carried out on a large assemblage of this vessel type found high quantities of lead on the inner surfaces, along with traces of silver and copper. This led to the theory that they were used in small-scale cupellation operations for the refining of small quantities of silver. This theory has led to the term 'cupels' being applied to these dishes. The most suitable material for cupellation is bone ash as it absorbs the lead oxide. Ceramic tends to block absorption, as the lead combines with the fabric to form lead silicates. The cupellation method also 'suffers from serious problems due to the loss of silver, dissolved in copper oxide, into the litharge', the molten lead oxide (Bayley & Eckstein 1997).

Cupellation carried out for the specific purpose of determining levels of impurity, as opposed to refining, to obtain a qualitative or quantitative analysis of an alloy containing a precious metal would be called assaying. This has led to the term 'assaying trays or assaying dishes' also being used.

The term 'scorifier' could also be applied to these dishes. This refers to a vessel used in the first heating in assaying to remove the earth and gangue and to concentrate the gold and silver in a lead button. It is difficult to assess from the slag on the vessels which process can most closely be associated with these vessels. Given the small quantities, assaying seems the most plausible explanation, however it could only have been an indicative process given the loss of silver into the slag.

The use of these vessels, and their connection with jewellery-making and their possible relation to goldsmithing and minting has been further discussed by Eilbracht (2004 & 2016), on whose initiative this study was conducted.

Focus of analysis and research objectives

The analyses were focused on testing the ‘refining’ hypothesis concerning function, and establishing whether the interpretation of these vessels as soldering plates linked to jewellery production has any foundation. Analyses thus focused on determining the elemental composition of the slag or vitrified layer, as well as the metallic prills therein.

Establishing technological choices in the manufacture of the melting plate fabrics was also a consideration but this was not explored in detail. Characterisation of the fabrics was done using optical microscopy and elemental analyses.

Examination of the microstructure of the fabrics was carried out to determine what types of clay were used, levels and types of inclusions and to establish the effects of high temperatures. The fabric analyses were also used to determine whether there was any contamination from the fuel ash in the areas where heat was applied, and to document any elements absorbed into the fabric from the reactions, as also from the later burial environment.

The main research objectives were to place the material within its wider archaeological context and to look at links with coinage and jewellery manufacture, and to examine these metallurgical ceramics in the light of other work from a similar time period, as well as the type of residues left and to determine what they can reveal about function. The results would confirm or dismiss earlier hypotheses put forward for the function of these artefact types.

Methodology

Six fragments from Sigtuna, ten from Fyrkat and the complete vessel from Trelleborg, were subjected to non-destructive small area analyses using a Spectro 2000 XRF (X-ray fluorescence) instrument on their inner and outer surfaces. XRF analyzers determine the chemistry of a sample by measuring the fluorescent (or secondary) X-rays emitted from a sample when it is excited by a primary X-ray source. This method provided a good indicative reading on the surface composition of the slag on the inner surfaces and the fabric on the outer surfaces. The results of this type of analysis can only be seen as qualitative.

Samples were cut from a total of seven melting plates: four from Sigtuna and three from Fyrkat. Two separate samples were obtained from fragment 3609:01. Small pieces were cut with a circular saw and mounted in epoxy resin. Once solidified, the samples impregnated in resin required polishing in order to create a flat surface. The samples were polished to the 1µm level.

The polished resin samples were examined using optical microscopy, and digital images were taken using plain and cross-polarised light. Images were taken documenting fabric types, porosity, vitrification, slag residue and any metallic prills trapped within. This initial microscopic characterisation proved a useful tool in identifying areas of interest for analysis before using SEM-EDS, energy-dispersive X-ray spectrometry.

The examination of mounted samples in the scanning electron microscope facilitated a more comprehensive visual examination of the fabrics and the slag layer in cross-section, and the attached energy-dispersive analyser also allowed for targeted chemical analysis within various zones.

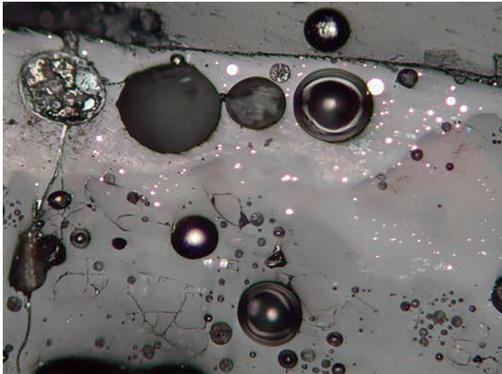


Figure 4. 3988. Plan Polarised Light (PPL) slag layer with large spherical bloating pores with small copper and silver prills. Image width 1 mm.

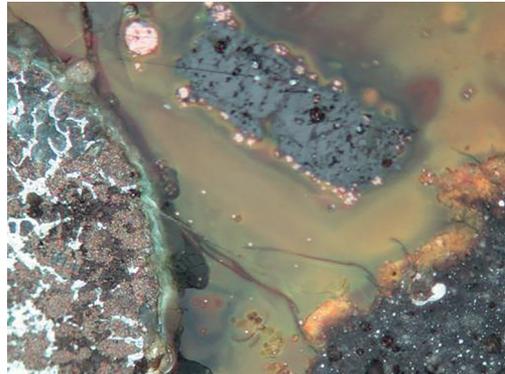


Figure 5. 3988. Cross Polarised Light (XPL) shows small metallic prills, slag and clay layer. Width of image c. 1 mm.

Once optical microscopy on the samples had been carried out and a comprehensive set of images for each sample had been obtained, it became clear that a consistent methodology for SEM-EDS analyses would be necessary in order to obtain a comparative set of results. Analysis was focused on chemical characterisation of the ceramic fabric at the outer surface, central and inner areas. Initial analysis of the fabric at the inner surfaces often showed contamination from the slag and results had to be normalised excluding contaminants. This was done in an attempt to establish whether the inner surfaces of these plates had been modified in any way during manufacture to facilitate the high temperature processes they were designed for.

Bulk analyses were taken at a constant measurement of 120x160 microns. Two to three analyses were taken at the outer, central and inner portions of the fabrics creating an overall total of 6–9 fabric analyses for each sample.

The standard measurement of 120x160 microns was also used for obtaining 3–4 bulk analyses on the slag layer. Spot analyses of 1–2 μm were usually taken on the metallic prills. In cases where prills were very large, bulk analyses were obtained in



Figure 6. Sample 3988 before sampling.

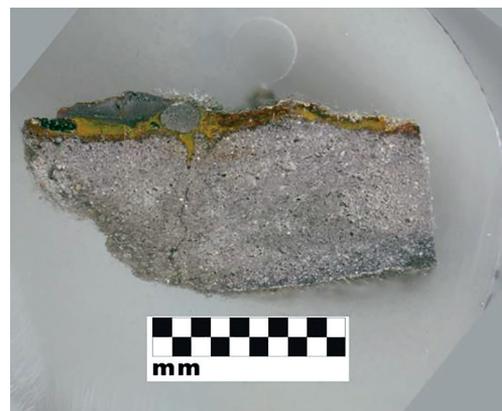


Figure 7. Sample from 3988 mounted in polished block.

order to characterise the overall composition. Usually 2–3 prills were analysed on each sample.

A sheet was designed for each individual sample, documenting an image of each fragment prior to sampling and showing where the sample was taken (see *fig. 6* for pre-sampling image of sample 3988 from Sigtuna and *fig. 7* for an image of a polished block sample).

It is not within the scope of this article to reproduce the comprehensive set of images and analytical results obtained from each individual sample. A selection of analytical results for metallic prills within the slag layer from different samples are presented along with averages of SEM-EDS results for the fabric and slag layer of each individual sample.

Results of XRF analysis

XRF results show that the majority of the elements detected on the inner surfaces are those typically associated with ceramic fabrics. Several metallic elements not associated with clays are also evident and these are associated with a metallurgical use of the vessel. Lead oxide is the largest contaminant averaging at 4.5% of the composition on the Fyrkat plates, 6.9% on the Sigtuna plates and only 0.3% on the Trelleborg plate. There is a lot of variation in the levels of lead oxide in the various dishes ranging from 17.6–0.3%. The very low levels detected in the Trelleborg dish may point to a different function for this vessel; the other contaminants present are CuO at 0.1%, ZnO at 0.4% and Ag at 0.3%, in stark contrast to the ratios of these elements to each other in the other dishes. As was expected no lead or any other metallic contaminants were detected in the Sigtuna sample 3561, as this had appeared unused prior to analysis.

Along with lead, the majority of the samples also show levels of copper oxide ranging from 0.1 to 6.4% and of silver ranging from 0.1–1.4%. Zinc oxide is present from 0–1.2% and levels of tin are also present in some samples from 0.1–0.6%. It is obvious that there is considerable variation between the different types and quantities of metallic elements turning up. Lead oxide is consistently present in all samples, along with lesser levels of copper and silver. Tin, zinc and bismuth turn up as trace elements in many of the fragments but not in a consistent fashion. It is notable that no gold was detected in any of these dishes.

The variation evident in the results may point to different functions of use. The consistent presence of lead and silver lends support to the refining/cupellation theory and perhaps the variation in levels of copper, zinc and tin are an indication of the different types of alloys being mixed with silver at that time. The levels of silver present within the slag also show that there was a certain percentage lost in the process. Perhaps metal-smiths or traders were content to allow this loss in order to gain some knowledge, however vague, of the levels of impurity.

There is also the possibility that the dishes were being used for soldering activities and that the lead was used as a solder for different types of precious metals and alloys in the manufacture of fine jewellery. This process could also have led to the presence of the various elements detected and would also explain the wide variation

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	CuO	ZnO	As ₂ O ₃	Ag	SnO ₂	PbO	Bi	Totals
Fyrkat																				
D1231C-1-1966	1.6	0.7	11.6	68.3	0.2	0.0	0.1	3.3	1.5	0.3	0.1	2.4	5.2	0.1	0.0	0.4	0.1	3.8	0.3	84.1
D1231C-2-1966	3.3	0.9	14.1	59.2	0.6	0.0	0.2	1.9	1.5	0.4	0.1	3.3	6.4	0.1	0.1	1.2	0.0	5.0	1.6	35.4
D2100-1-1966	1.3	1.2	14.2	64.1	0.6	0.1	0.2	3.3	2.4	0.5	0.1	4.2	2.5	0.2	0.1	0.3	0.0	4.1	0.5	86.6
D2100-2-1966	1.6	1.3	15.8	53.7	1.1	0.0	0.4	2.7	2.0	0.5	0.2	4.7	3.7	0.1	0.2	0.9	0.0	9.9	1.1	49.4
D2100-3-1966	1.6	1.3	12.0	66.5	0.4	0.0	0.0	4.9	6.5	0.4	0.1	3.9	0.6	0.0	0.0	0.1	0.0	1.4	0.0	84.1
D2100-4-1966	0.7	1.0	13.4	62.2	0.0	0.0	0.0	2.8	1.1	0.4	0.1	3.7	8.2	0.4	0.0	1.4	0.0	3.8	0.5	54.9
D345B-1-1966	1.4	1.3	18.2	59.4	1.0	0.0	0.0	3.3	2.0	0.6	0.1	7.5	2.3	0.4	0.0	0.1	0.1	1.9	0.2	69.3
D345B-2-1966	1.8	1.1	17.1	57.2	1.8	0.0	0.1	3.1	1.1	0.5	0.1	4.5	2.8	0.0	0.1	0.3	0.0	8.3	0.0	57.7
D522C-1966	0.4	1.0	10.2	49.5	1.7	0.4	0.1	2.6	4.8	0.6	0.2	24.5	0.4	0.0	0.0	0.2	0.1	3.0	0.1	83.1
D561-1966	4.2	1.8	11.0	57.8	2.0	0.0	0.3	3.9	4.3	0.4	0.1	5.6	3.7	0.0	0.0	0.5	0.0	3.9	0.2	104.8
Averages	1.8	1.2	13.8	59.8	0.9	0.1	0.1	3.2	2.7	0.5	0.1	4.4	3.6	0.1	0.1	0.5	0.0	4.5	0.4	
Trelleborg																				
Complete plate	1.8	1.0	11.3	65.3	2.1	1.6	0.0	4.4	6.0	0.4	0.1	5.0	0.1	0.4	0.0	0.3	0.0	0.3	0.0	69.9
Sigtuna																				
3304[R7.15]:02	1.2	0.9	11.0	54.2	4.4	1.8	0.0	3.1	4.4	0.2	0.0	5.7	5.8	0.1	0.1	0.6	0.0	5.5	1.0	78.8
3561/4:06	2.1	1.4	12.5	59.2	4.0	0.9	0.0	4.1	6.2	0.3	0.1	5.6	0.7	0.3	0.1	0.6	0.0	1.7	0.0	81.0
3609[T6.5]:01	0.0	1.1	13.1	57.4	4.3	0.8	0.2	4.0	6.4	0.2	0.1	7.3	2.9	1.2	0.1	0.4	0.6	4.6	0.1	63.8
2731[T4.4]:01	1.2	0.0	10.0	43.7	9.5	2.8	0.5	3.9	3.0	0.2	0.0	11.6	3.6	0.4	0.1	0.7	0.1	11.9	0.9	53.3
3988[57.4]:01	1.1	0.7	7.1	43.1	11.1	1.3	0.1	2.1	7.6	0.2	0.0	8.9	0.8	0.2	0.1	0.5	0.0	17.6	0.1	94.9
3561[57.5]:03	1.2	1.7	17.1	64.7	2.5	0.2	0.0	4.7	4.1	0.4	0.1	4.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	87.4
Averages	1.1	1.0	11.8	53.7	6.0	1.3	0.1	3.6	5.3	0.3	0.1	7.2	2.3	0.3	0.1	0.5	0.1	6.9	0.4	76.5

Table 1. XRF (turbo-quant) Results for inner surfaces of melting plates. All results have been normalised and converted to weight %. Totals represent the original total wt % prior to normalization. Averages for Fyrkat Fe₂O₃ exclude the 24.5% value as it is exceptionally high and distorts overall average.

of contaminants detected on the various dishes. There is very little published on the chemical analysis of solders present on Viking Age jewellery or even on the composition of alloys used in such jewellery. Yet there are ocular analyses of the solders in gold- and silver-filigree from Birka, Sweden, made by Duzcko who concluded that the majority of the analysed objects were not soldered with metallic but with chemical solder, i.e. various copper compounds (Duzcko 1985:25ff). More work would need to be done in this area.

These slagged ceramic plates vary a lot in morphology and in the slag composition found on their upper surfaces. Given that this artefact type is found across a wide geographical area from the 6th to the 12th centuries and in varying different contexts, multi-functional uses cannot be ruled out.

No metallic contaminants were detected at the outer surfaces. Results from the basal surface of vessels can be seen as indicative of the pre-use chemical signature of the fabric. Overall the fabrics from different regions are quite similar. The Sigtuna and Trelleborg plates show higher levels of sodium, this may be due to their deposition in a more saline environment. Higher phosphorus and potassium levels are evident in the Sigtuna fragments, this may also be due to the absorption of these

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	CuO	ZnO	Ag	SnO ₂	PbO	Totals	
Fyrkat																			
D1231C-1-1966	1.2	1.3	18.8	60.8	2.9	0.6	1.2	4.3	2.3	0.9	0.1	4.4	0.1	0.0	0.8	0.0	0.0	0.0	61.5
D1231C-2-1966	1.3	2.0	15.3	66.0	1.5	0.3	0.0	4.3	2.1	0.7	0.1	6.2	0.0	0.0	0.0	0.0	0.0	0.0	62.1
D2100-1-1966	0.8	1.1	14.7	73.7	0.2	0.2	0.0	2.9	1.1	0.8	0.1	4.2	0.0	0.0	0.0	0.0	0.0	0.0	82.2
D2100-2-1966	0.7	1.4	16.0	72.8	0.1	0.2	0.0	2.7	0.6	0.7	0.0	4.3	0.0	0.0	0.0	0.0	0.0	0.0	76.9
D2100-3-1966	1.5	1.1	14.4	72.2	0.2	0.3	0.0	3.9	0.9	0.8	0.0	4.5	0.0	0.0	0.0	0.0	0.0	0.0	91.9
D2100-4-1966	1.1	1.3	15.1	69.3	0.1	0.2	0.0	3.3	0.9	1.0	0.1	7.4	0.0	0.0	0.0	0.0	0.0	0.0	46.0
D345B-1-1966	0.9	1.4	17.7	68.3	0.2	0.2	0.0	4.5	1.0	0.8	0.0	4.7	0.0	0.0	0.0	0.0	0.0	0.0	72.6
D345B-2-1966	1.5	1.6	15.9	70.1	0.2	0.2	0.0	3.1	0.7	0.9	0.1	5.6	0.0	0.0	0.0	0.0	0.0	0.0	81.0
D522C-1966	0.8	1.7	15.9	69.0	0.7	0.2	0.0	3.4	1.8	0.8	0.1	5.5	0.0	0.0	0.0	0.0	0.0	0.0	88.0
D561-1966	1.2	0.7	17.9	63.4	4.1	0.4	0.0	3.1	1.6	0.9	0.1	6.1	0.0	0.0	0.0	0.0	0.0	0.0	108.5
Averages	1.1	1.4	16.2	68.6	1.0	0.3	0.1	3.6	1.3	0.8	0.1	5.3	0.0	0.0	0.1	0.0	0.0		
Trelleborg																			
Complete plate	2.2	1.4	13.7	69.8	1.1	0.4	0.0	3.7	2.9	0.5	0.0	3.9	0.0	0.0	0.0	0.0	0.0	0.0	88.9
Sigtuna																			
3304 [R715]:02	1.5	1.6	14.5	60.7	3.5	0.3	0.0	3.3	8.3	0.5	0.1	5.3	0.1	0.0	0.0	0.0	0.1	0.1	89.3
3561/4:06	1.8	2.0	16.8	65.5	1.5	0.1	0.0	3.2	4.6	0.4	0.1	3.8	0.0	0.0	0.0	0.0	0.0	0.0	80.0
3609 [T6.5]	3.6	1.2	13.5	60.1	2.1	0.3	0.1	6.7	3.7	0.4	0.1	7.8	0.0	0.0	0.0	0.0	0.0	0.0	83.2
2731[T4.4]:01	1.4	1.5	15.6	65.9	3.3	0.0	0.0	3.1	3.7	0.4	0.1	4.0	0.2	0.0	0.0	0.0	16.1	0.1	90.3
3988[57.4]:01	5.0	1.0	16.1	64.8	2.0	0.2	0.1	4.2	3.4	0.4	0.1	4.4	0.0	0.0	0.0	0.0	0.1	0.1	97.0
3561[57.5]:03	1.3	1.8	16.3	59.7	5.8	0.2	0.0	4.1	5.3	0.6	0.2	5.7	0.0	0.0	0.0	0.0	0.0	0.0	84.3
Averages	2.4	1.5	15.5	62.8	3.0	0.2	0.0	4.1	4.8	0.5	0.1	5.2	0.0	0.0	0.0	0.0	0.0	2.7	

Table 2. XRF (turbo-quant) results for outer surfaces of melting plates. All results have been normalised and converted to weight %. Totals represent the original total wt % prior to normalization.

elements within the burial environment. Calcium oxide is a lot higher in the Sigtuna fragments and this may be due to a higher level of calcareous minerals within the clay.

It is notable that the levels of iron oxide are 2.0% lower on the outer surface of the Sigtuna plates than on the inner surface, this could be due to the deliberate creation of a finer clay layer on the inner surface of the vessels. Subsequent analytical evidence from SEM-EDX analyses suggested there is a higher concentration of iron oxide in the clay paste once larger mineral inclusions are excluded from analysis.

Optical microscopy of melting plates

A total of five samples were taken from the Sigtuna melting plates, of which two were from the same artefact 3609 [T6.5]:01, while sample 3561[57.5]:03 was from an unused melting plate. Fabrics of all five samples ranged from light to dark grey in colour, indicating reducing firing conditions. Sample 3988 had a metallic prill clearly visible on the inner surface, 2731 also had a corroded metallic fragment on the inner surface. Samples 3609A and B did not have any visible prills, but the slag layer was very well-defined due to its bright red colour.

The fabrics of the melting plate samples consisted predominantly of a sandy clay (40% sand, 60% clay), and sample 3988 had a higher level of sand grains comprising



Figure 8. Images of polished blocks for Sigtuna melting plate samples L-R 3988A, 2731, 3561, 3609A & 3609B.

c.70% of the fabric. The fabrics were all fine-grained, inclusions were predominantly quartz; crushed quartz with sharp angular edges was common throughout the fabrics. The inclusions ranged from being moderately to well-sorted throughout the fabrics. Mica and other inclusions were also present within the fabrics, but to a lesser extent.

Sample 3988 was slightly different from the others in that the size of inclusions was generally smaller, and also because it had a layer of fine ceramic, with no inclusions visible overlying the slag layer. This may indicate that the melting plates were deliberately coated with a layer of fine clay on the inner surface prior to use. There was no visible evidence for this on the other samples, and if originally present it could have become absorbed within the slag during the reaction stage.

The used melting plates all showed vitrified slag layers with fine to medium bloating pores on the inner surface. Immediately below the slag layers, extensive vitrification with irregular porosity (20-30%) could be seen within the fabric, to depths of 2 mm below the slag. The central area usually showed evidence for extensive to initial vitrification. The outer surface showed no vitrification, and inclusions could be clearly distinguished from the surrounding clay matrix; porosity at the outer edges usually ranged from 3–5%. The vitrification at the inner surfaces of these samples indicated that heat was applied from above. The vitrification levels at the inner surfaces pointed to temperatures in excess of 1000 °C, and in the central areas of the fabrics it is estimated that temperatures of c.750–850 °C would have been required to cause the

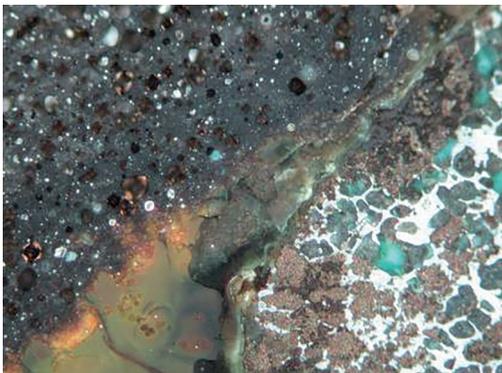


Figure 9. Sample 3988 XPL image of silver prill (right) and fine clay layer overlying the adjacent slag. Image width 1 mm.

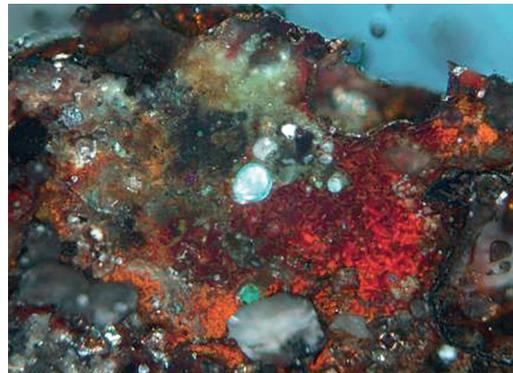
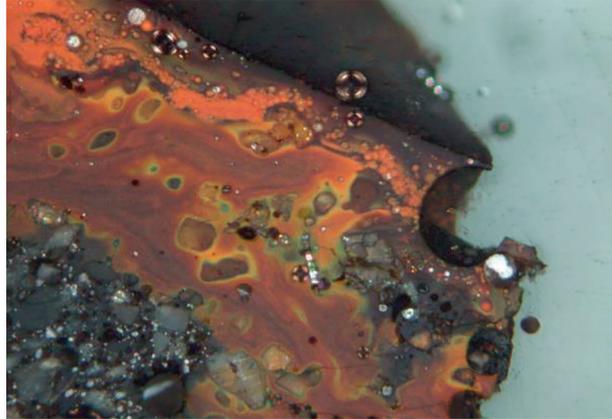


Figure 10. Sample 2731 XPL image of metallic inclusion on the inner surface showing a mixed area of copper metal dendrites, copper oxide and lead silicate crystals. Image width 1 mm.

Figure 11. Sample 3609A, microscopic view (XPL) of copper-rich slag layer at the edge of the break. Image width 1 mm.



increased porosity and initial to extensive vitrification in the fabrics. Vitrification and porosity levels in the samples are described according to parameters outlined by Tite et al. (1990).

All of the used melting plates had a slag layer of varying thickness across the surface of the sample. The slag layer in sample 3609A was thicker than in the other samples and the slag extended down the broken edge of the fragment, perhaps indicating that the reaction of the metals with the ceramic material may have contributed to its breakage. There is also the possibility that this sherd was broken before use. The reuse of broken sherds has been observed by previous authors (Rehren & Kraus 1999; Bayley 1992a). Samples 3609A and B also showed more bloating in the fabric immediately beneath the slag layer than in the other samples, indicating that this fabric may not have been as refractory as the others or that it was subjected to higher temperatures.

SEM-EDS imaging and analyses

The XRF results facilitated some level of characterisation of the ceramic fabrics but were mainly useful in providing an indicative pointer regarding the presence of metallic elements within the slag layer. The XRF results can only be seen as qualitative. The SEM-EDS results are semi-quantitative and are more useful in documenting chemical composition.

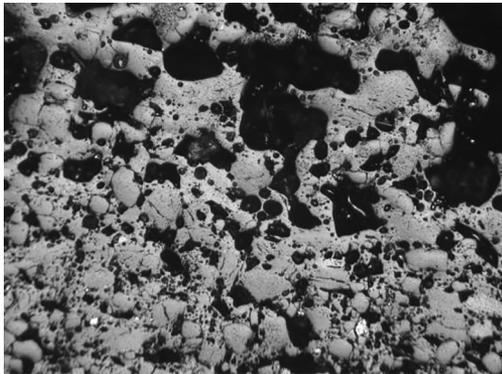


Figure 12. Sample 3609B PPL fabric showing transition from irregular large interconnecting pores to isolated pores. Width of image c.1 mm.

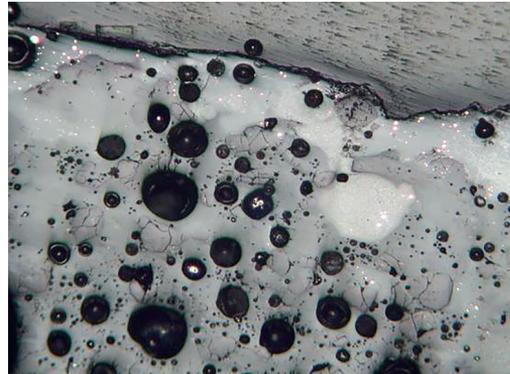


Figure 13. Sample 3609B PPL Vitrified slag layer with rounded pores and copper prills visible as well as small pink flecks. Image width c.1 mm.

Analyses were focused on establishing chemical signatures of the fuel ash within the slag layers, the chemical composition of the fabrics and the slag layers, to document variation between the samples, and to document the chemical composition of metallic prills identified within the slag layers.

*Examining the chemical signature of the fabric,
and examining changes within the reaction zone*

Re-normalising the elemental analyses for the slag layer to exclude metallic elements proved useful in documenting chemical differences between the general fabric of the vessel and the changes that had occurred within the fabric on the inner surface due to the reaction(s) that took place, and perhaps due to deliberate modification of this portion of the vessel prior to use.

Previous analyses of wood ash composition have shown that the major elements are calcium, potassium and magnesium. Sulfur, phosphorus and manganese are present at c. 1% (Misra et al. 1993:111). It is evident from *table 3* that magnesium, potassium and calcium levels are slightly higher in the slag layer. The higher concentration of these elements within the slag layer is most likely produced by the fuel ash used in the reaction within these vessels.

The variation in phosphorus levels is probably due to differential absorption of this element from organic sediments within the burial environment. Absorption of this element within the fabric appears to be concentrated in regions of high porosity.

There is also the possibility that high levels of CaO and P₂O₅ noted in the slag layer could be linked to the use of a fine layer of bone ash across the surface of these dishes. If this were the case such a layer would facilitate the absorption of lead in the event of cupellation or assaying being carried out. It is difficult to establish if this is the case without carrying out some experimental work attempting cupellation on ceramic dishes with and without bone ash to determine the kind of results one might expect.

There were substantial differences between the iron concentrations within the fabric of the Sigtuna vessels and the slag layer, where the average iron value within the fabric was 5.1% as opposed to 8.32% in the slag. In the Fyrkat samples the opposite was the case, the iron values in the fabric were 6.10% and in the slag 5.68%.

It is difficult to explain the higher iron values within the slag in the Sigtuna samples. It may be due to the presence of iron within the metal being melted in the vessel at the reaction stage or it may be due to the presence of a fine clay coating at the inner surface of the vessel prior to the reaction stage. Spot analyses on the clay matrix of some of the Sigtuna vessels seemed to indicate higher iron values being concentrated in the clay matrix (the majority of the bulk fabric analyses consisted of clay and up to 50–60% inclusions). The presence of a fine ceramic layer overlaying the slag in sample 3988 (*fig. 14*) provides some support for this theory. Spot analyses on the clay layer in 3988 also revealed a high iron concentration. Higher Fe values when analysing pure clay matrix, points to high Fe in the clay fabric.

The Fe values are significantly higher when analysing the fine clay matrix than in the bulk fabric compositions. The higher Fe values present in the slag layer were initially thought to be connected to high Fe values in the metal being melted. The

Table 3. Averages were obtained of overall inner, central & outer bulk fabric analyses (120 x 160 µm, clay matrix & inclusions). The bulk fabric analyses from the slag layer were recalculated excluding the metallic elements in order to detect any chemical differences in the ceramic fabric at the inner surface of the vessel within the slag layer. F1-F3: Fyrkat samples, 2731-3609B: Sigtuna samples.

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	FeO
F1 slag	0.9	2.0	10.0	57.3	1.3	0.0	4.3	8.9	0.6	4.8
F1 fabric	1.1	1.5	14.0	72.3	0.1	0.1	3.4	1.3	0.9	6.2
F2 slag	1.3	1.5	7.5	60.7	0.0	0.0	7.0	9.9	0.4	4.0
F2 fabric	1.6	1.3	14.0	72.3	0.1	0.0	3.5	0.9	0.8	5.4
F3 slag	3.0	3.1	9.7	52.6	0.0	2.8	4.2	6.0	0.7	8.2
F3 fabric	1.8	1.2	14.3	66.6	2.2	1.4	3.3	1.4	0.9	6.7
2731 slag	1.18	0.52	11.21	63.18	0	0	7.64	9.19	0	7.86
2731 fabric	2.85	0.7	14.85	70.8	0.4	0	3.7	2.2	0.4	3.85
3988 slag	2.06	1.81	14.21	60.94	1.87	0	4.41	5.91	0	8.79
3988 fabric	1.7	1.45	12.7	73.05	0.3	0	4	1.85	0.45	4.5
3609A slag	0.23	1.47	14.82	63.26	0	0	4.29	8.88	0.3	6.77
3609A fabric	1.7	1.3	13.55	70.4	0.25	0	3.65	1.65	0.45	5.65
3609B slag	0.85	2.59	13.79	58.26	0	0	4.18	10.47	0	9.86
3609B fabric	2.49	1.48	14.43	67.37	1.01	0	3.65	1.93	0.5	6.46

presence of a thin fine clay layer in this sample overlying the slag, points to the possibility of an inner lining of fine clay in advance of the reaction. If this was the case it would explain the overall higher Fe values noted in the slag residues of the other samples as well.

Analyses of the slag layer within the melting plates

Although no silver was detected in the bulk composition of the slag layer for samples 3988, 3609B and F2, there were one or two large (c. 1 mm across) silver prills associated with these samples. It would seem that some samples have tiny prills within the slag while others have only one or two large prills. The overall evidence suggests that the loss of silver into the slag is quite low. However this is all relative to the amount of silver being melted, which is a difficult factor to quantify. Even with experimental work, one would need a clearer idea of which process was most likely being carried out. Experimental work could prove helpful.

The table illustrates quite clearly that lead is the main metallic component within the slag layer. There is considerable variation between the samples, with lead ranging

Figure 14. Sample 3988 view of fine clay layer overlying slag and large silver prill.

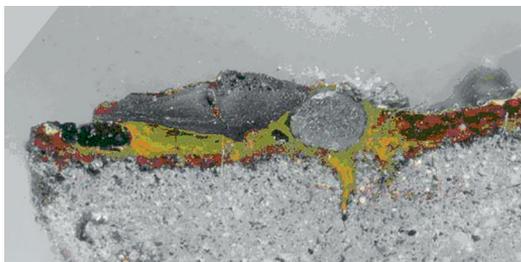


Table 4 below. SEM-EDS, composition of clay matrix.

3988		Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	TiO ₂	FeO
sp15	Spot analysis on internal clay (fabric only)	1.93	2.7	19.04	60.55	4.79	1.77	0.93	8.28
sp3	Bulk composition of fine clay at surface	2.11	2.91	18.68	57.89	6.94	1.84	0.97	8.66

Slag averages	Fabric	CuO	ZnO	Ag ₂ O	PbO
2731	73.6	4.5	0.7	4.9	24.2
3988	45.0	1.7	0.6	0	58.2
3609A	76.3	13.7	0.4	3.7	12.7
3609B	59.1	11.4	4.7	0	31.9
Averages Sigtuna	63.5	7.8	1.6	2.2	31.8
F1	73.1	8.5	4.6	0.4	20.6
F1	60.7	4.1	0.0	0.0	39.7
F3	89.9	1.5	0.0	0.5	16.8
Averages Fyrkat	74.6	4.7	1.5	0.3	25.7

Table 5. Values obtained from bulk sample analyses (120x160 µm), carried out on the slag layer at the inner surface of the melting plates. The elements associated with the ceramic fabric have been totalled in order to illustrate the proportion of ceramic to metallic material within the slag layer. Relative values for ceramic analyses have already been presented in Table 3. Sample 3561 (unused plate) has been excluded.

from 12.7 to 58.2% of the slag, which is probably also caused by the uneven distribution of the various elements across the inner surface at the reaction stage. Copper is usually present as a minor element ranging from 2–14%, and zinc is usually only present in trace quantities (apart from sample 3609B). Silver is usually only detected in trace quantities, apart from samples 2731 and 3609A. All analyses of the slag layers show that they consist of metallic elements that have penetrated into the ceramic fabric; the elements found within the fabric are also found in the slag. No copper, zinc, lead or silver were detected in sample 3561, confirming that this fragment was never used for metallurgical purposes.

The differences in copper, silver, zinc and lead values between 3609A and 3609B show the differential distribution of metallic elements within the slag layer from a single fragment. The values for copper, silver and zinc also vary considerably from sample to sample.

Despite problems of silver loss when using the hypothetical assaying/cupellation process, this technique may have been an option known to traders at the time for providing a relatively good indication of levels of purity, complementing the use of touchstones (cf. Moore & Oddy 1985, Ježek 2017). The levels of copper detected, ranging from 1.5–13.7%, along with trace quantities of zinc and tin, could be taken as an indicator of relative success in removing impurities from the silver, if this was the intention.

Regardless of intention, the variation of the different metallic elements within the slag together with the high levels of copper in many of the samples, would suggest that there were large variations in the quantities of copper and zinc alloyed with the silver.

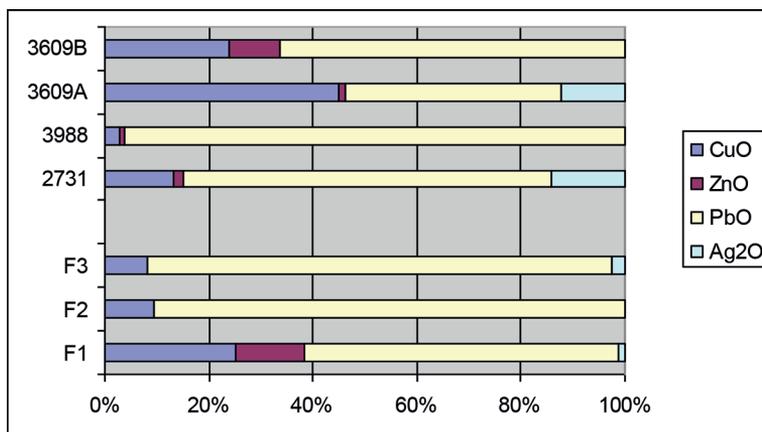


Figure 15. Relative values of copper, zinc, lead and silver between the various samples.

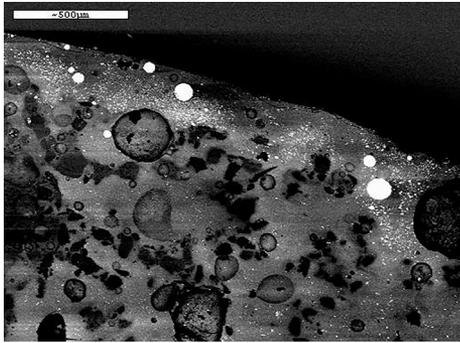


Figure 16. Sample 3609A, SEM-EDS image showing large to small copper & silver prills in the slag.

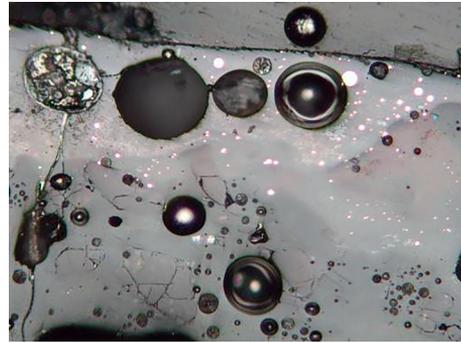


Figure 17. Sample 3988, PPL slag layer with large spherical bloating pores with small copper and silver prills. Image width c.1 mm.

Metallic prills within the slag layer

The metallic prills on the slag surfaces and within the slag layers show a variety of differing results when analysed. They usually consist of either relatively pure copper or silver, or a combination of both. Silver chlorides and sulfides were observed, as well as bismuth and high iron values.

Three silver-copper prills were analysed in sample 3609A. One consisted of 20% copper and 74% silver, a second prill consisted of 32% copper and 70% silver, and a third prill consisted of 92% silver and 1% copper. The results for the 3609A prills were obtained using spot analyses 1–2 μm in diameter.

In sample 3609B, it was difficult to obtain any good analytical data from the prills in the upper slag layer, as many of them were very small. A single spot analysis on one prill in this layer revealed a composition of 31% Cu and 69% Ag. A large lump of residue was adhering to the base and side of this fragment. This lump measured 275 μm in thickness and extended almost 1 mm around the edge of the sherd. Two bulk analyses on this area (x860) revealed it was rich in a silver sulfide with high iron levels, and small traces of copper (table 6).

The bulk slag analyses from samples 3609A and B (table 5) show far higher levels of copper, suggesting that this plate was used for melting a particularly debased silver or perhaps a larger quantity of silver than in the other melting plate samples.

In sample 3988 a large metallic prill was visible, measuring c.1.5 mm in diameter (figs. 8 & 14). Also, within the slag layer frequent very small copper-rich prills were visible (fig. 17). Analyses on the large prill revealed that it consisted mainly of silver chloride with copper metal and copper oxide (table 7).

The prill in sample 2731 (figs. 8, 10 & 18) was a mixed mass of various metallic and non-metallic phases. Analyses revealed areas rich in silver, bismuth, lead and copper as well as high levels of calcium, phosphorus and silica.

Several spot analyses (2 μm diameter) on various areas within the metallic region adhering to the ceramic revealed some of the different phases present. Spectrum 1

	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	CaO	FeO	CuO	Ag ₂ O
Sp5	2.65	1.51	4.99	12.86	1.3	12.43	1.08	58.31
Sp6	4.1	17.65	5.11	11.5	1.43	11.42	0.88	44.89

Table 6. Bulk analysis of silver-rich residue at the base of sample 3609B on the right side.

3988 MP	Mg	Al	Si	P	Cl	Cu	Ag	Pb
Prill 1A x860 bulk		2.72	1.31	1.16	22.74	25.48	45.39	1.2
Prill 1B spot analysis	0,79	1.09	1.85		19.48	1.75	75.05	

Table 7. Analysis of large silver prill in sample 3988.

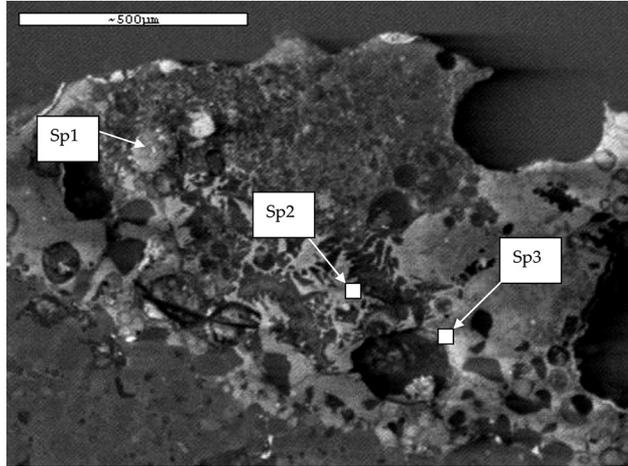


Figure 18. SEM image of metallic prill on upper surface of sample 2731 (refer Table 8 for analyses).

reveals a prill of silver and bismuth, this would have separated from the lead during the reaction stage. Spectrum 2 shows a high proportion of silica, lead, copper, and calcium. The silica is present as part of the original ceramic fabric has reacted with lead oxide to form a lead silicate compound in some areas. The calcium could originate from the fuel ash, the oxidation of the lead would in turn have contributed to the oxidation of the copper, explaining why they are more likely to occur together. Silver tends to separate into isolated prills within the melt, which also is the very nature of the cupellation process. Spectrum 3 is from a mixed region of copper, silver, lead and ceramic phases.

Examination of the Fyrkat samples revealed a similar pattern of metallic prills rich in copper and silver within the slag layer.

A wide variety of metallic and non-metallic, ceramic, phases are visible within the prills, making it difficult to make any conclusive statements about the analyses obtained. Bismuth was only observed in one analysis on the Sigtuna samples (2731), however it was detected within several prills within sample D345B-1-1966 from Fyrkat (table 9).

The method of analysis adopted makes it difficult to quantify the amounts of silver being lost into the melt, perhaps some sort of digital image analysis of the quantity of metallic prills within the slag would help with this. A systematic analysis of metallic elements within the prills could then lead to overall estimates of silver within the prills. This figure could then be related to a percentage of the cross-section

2731 MP	Na ₂ O	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	FeO	CuO	Ag ₂ O	PbO	Bi ₂ O ₃
Sp1		2.9		11.4		4.8	3.8	1.3	38.9		36.9
Sp2		6.1	30.8		4.6	10.6	4.8	12.8		30.4	
Sp3	1.2	9.6	27.1	11.2	1.4	7.4	4.7	3.2	14.8	19.4	
Entire area, fig. 18	0.8	10.1	48.8	3.4	2.9	8.3	6.8	5.2	3.5	11.0	

Table 8: Results of spot analyses in metallic area (2 µm diameter) & bulk analysis of the whole image (refer fig. 18, sample 2731).

	Cu	Ag	Bi
Prill 1	2	91	7
Prill 2	57	38	5
Prill 3	42	49	9
Prill 4	63	37	0

Table 9: Analyses of prills on Fyrkat sample D345B-1-1966.

being analysed, which could in turn be related back to the amount of slag on the sample. It would, however, still prove very difficult to relate the quantity lost into the melt back to the amount of silver that was melted in the first place.

Conclusions & suggestions for future research

Overall, this analysis has helped to characterise the fabrics used in these ceramic dishes. This information is useful, but it was not within the scope of this study to relate that information to other non-metallurgical ceramics from contemporary contexts. A further comparative study of a similar nature would prove useful in documenting technological choices for these fabrics. It was evident that there was very little difference between the fabrics used for the Sigstuna and Fyrkat plates. The systematic bulk analysis of fabrics at the outer, central and inner areas of the ceramic fragments did not reveal any significant changes, and points to a relatively homogenous ratio of elements throughout the fabric.

The bulk analyses of the slag layer, once normalised to exclude contaminating metallic elements from the reaction, did reveal some interesting information regarding the interaction of the fabric with the fuel ash. It was noted that calcium, potassium and phosphorus levels were higher in this area, this may be as a result of elements from fuel ash being absorbed into the fabric. The high calcium and phosphorus levels may also be indicative of a layer of bone ash as mentioned in Theophilus' treatise from the 11–12th centuries. The higher iron levels that were also detected, along with the analyses from the layer of fine clay noted on the surface of sample 3988, point to a possible deliberate modification of the internal surface of melting plates. This layer may have become obscured in the subsequent use of the other vessels.

The overall impression from chemical analyses and visual examination of the slag layer within these samples is that there is considerable variation. Even the two samples taken from the same fragment 3609 show substantial differences. This is due to the differential distribution of the slag across these dishes. There is also a variation within the composition of the metallic prills. This makes it difficult to reach any definitive conclusion regarding the use of these vessels. The investigation found no evidence for gold in any of the samples. However, copper-silver alloys were frequently encountered, demonstrating the use in silver metallurgy, as has been proposed earlier. Refining by cupellation can be excluded, based on the absence of significant phosphorus enrichment beyond what can be explained by fuel ash.

The high lead quantities could still support the refining theory, but the loss of silver prills into the melt suggests it would have been a rather arbitrary method. There has been very little exploration of jewellery-making or soldering hypotheses and the latter still cannot be ruled out as a possible function. Some evidence for the use of lead solders in jewellery manufacture is known.

Analysis of filigree panels on contemporary Insular Irish Sea metalwork has indicated two types of solder, a gold-based brazing alloy or a copper salt derived from

malachite powder, similar to the results from the study of filigree jewellery from Birka. On the Derrynafan paten analysis of joints on a finely decorated panel revealed a variety of elements in excess. These showed combinations of e.g. silver, copper, and occasionally silver and tin, copper and tin and sometimes a little lead occur, suggesting some association with soft solder alloys. These combinations were found mostly on the joints, which suggests that they represent traces of brazing alloys (Whitfield 1993: 133).

The appearance of the ceramic melting plates on high status sites in the 6th century, before the advent of coinage suggests they were initially used in association with the manufacture of fine jewellery, however the link between the use of this artefact type and precious metals may have led to its later use in association with coin production and assaying. Sigtuna was indeed a minting site from the late 10th century and a few decades onwards, and the Sigtuna samples in this study are all connected with the early phase of this minting activity. A study of the contexts where this artefact type is found over time across a wide geographical area, along with a detailed scientific study of the residues on a larger sample, would undoubtedly prove useful in elucidating the single or multi-functional uses of this artefact type.

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Sammanfattning

Denna artikel är baserad på en uppsats som ingick i en MSc (Master of Science) i Technology & Analysis of Archaeological Materials vid Institute of Archaeology, University College of London, 2005. Undersökningen utfördes under handledning av professor Thilo Rehren, och analyserna utfördes vid Wolfson Archaeological Science Laboratories vid den arkeologiska institutionen. Arbetet genomfördes med stöd av ett Marie Curie-stipendium för MSc-studier.

Inom studien har två samlingar av metallurgisk keramik undersökts, en från en plats identifierad som Sveriges första myntverkstad (ca 1000 e.Kr.) i det vikingatida Sigtuna. Den andra samlingen kom från den samtida kungliga ringborgen Fyrkat i Danmark.

Den undersökta metallurgiska keramiken bestod av keramiska plattor eller ”värmeplattor”, associerade till silverraffinering och/eller guldsmede och smycketillverkning. Studiens mål var att utröna kärlets primära funktion. De flesta prover hade slagglager på ytorna, vilket antydde en användning inom metallurgisk aktivitet under höga temperaturer.

De förslagade keramiska plattorna varierar mycket morfologiskt, liksom sammansättningen hos slaggen på deras ovanytor. Variationen hos de olika ämnen som observerades, och de höga kopparvärdena, antyder stora variationer i koppar- och zinkhalt hos det silver som hanteras i kärlet. De höga blyvärden som observerades och en antydning till eventuell användning av benaska i processen ger trovärdighet åt teorin om raffinering, men de silverdroppar som syns ha förlorats som inneslutningar i slaggen antyder att metoden i så fall varit tämligen lyckfull.

En studie av de miljöer där föremålstypen påträffats, över ett vitt geografisk område, tillsammans med detaljerad naturvetenskaplig analys av ämnena i ett större materialprov, skulle tveklöst vara värdefull för att klarlägga föremålstypens användningsområde eller användningsområden. Experimentella studier baserade på föreliggande lödnings- och raffineringshypoteser vore också välkomna.