

Staffordshire Hoard Research Report 19

XRF Study of Silver Objects from the Staffordshire Hoard

E.S. Blakelock

2015

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Information about this report

This report was produced in 2015 as part of Stage 2 of the project. At that time the catalogue had not been finalised and the specimens in the tables of data are identified by their K numbers. The concordance of the K numbers given in the report to the catalogue numbers as they appear in the final publication is as given below. The list also includes the names of the objects as used in the final publication.

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K	Catalogue	Name in publication
number	number	
13	379	Half of a hilt-plate cast in silver.
39	69	Pommel in cast silver, of cocked-hat form, with gilded low relief
		decoration and niello inlay
63	409	Pair of hilt-guards in cast silver, with panels of gilded interlace, and
		gold mounts with filigree decoration and a gem setting.
64	571	Fragments possibly from a single strip-mount in silver with pointed
		turned ends, geometric niello inlay and a gilded border.
138	371	Hilt-plate in cast silver of oval form with gilding
176	614	Edging in silver-gilt, with a U section.
179	375	Hilt-plate cast in silver with line ornament.
1734 ¹	593	Helmet-band in cast silver-gilt, inset with a silver-gilt sheet band,
		showing a continuous procession of kneeling or running warriors.
239	372	Hilt-plate cast in silver of oval form.
241	570	Fragments from possibly two strip-mounts in silver with geometric
		niello inlay and gilded borders.
248	385	Side of a hilt-plate in silver-gilt.
274	684	Silver 'arm' with one expanded semi-circular terminal.
286	64	Pommel in cast silver, of cocked-hat form, with gilding and a garnet
		set one side
290	75	Pommel in cast silver, of cocked-hat form with double sword-rings,
		with cast interlace, niello and glass decoration.
294	73	Pommel in cast silver, of round-back form, with low relief decoration,
		and one side a mount with filigree and cloisonné ornament.
298	182	Hilt-collar in cast silver, of high form, with gilded low relief
		decoration.
302	580	Pyramid-fitting in cast silver with gold mounts, with filigree and
		garnet cloisonné decoration
304	186	Hilt-collar in cast silver, of narrow form, with gilded low relief
		decoration and black niello.
		Continued

¹ Renumbered from K235

K	Catalogue	Name in publication
number	number	
306 310	63 567	Pommel in cast silver, of cocked-hat form, with filigree decoration. Eye-shaped mount in silver with geometric niello inlay and filigree trim
363	590	Helmet-crest section, cast in silver and gilded, with animal ornament and animal-head.
369	184	Hilt-collar in cast silver, of high form, with line and bunch decoration, gilding and black niello.
397	590	Helmet-crest section, cast in silver and gilded, with animal ornament and animal-head.
453	591	Helmet cheek-piece, cast in silver and gilded, with animal ornament and a gold collar.
456	66	Pommel in cast silver, of cocked-hat form, with gilding.
522	375	Hilt-plate cast in silver with line ornament.
543	82	Sword-ring in cast silver of fixed form.
559	67	Pommel in cast silver, of cocked-hat form.
577	533	Mount in cast silver, of tongue-shaped form, with gilded low relief
		interlace ornament and niello inlay.
593	371	Hilt-plate in cast silver of oval form with gilding.
711	68	Pommel in cast silver, of cocked-hat form, with gilded low relief
		decoration and niello inlay.
744	75	Pommel in cast silver, of cocked-hat form with double sword-rings,
		with cast interlace, niello and glass decoration.
762	77	Pommel in cast silver, of cocked-hat form with double sword-rings,
		gilded low relief decoration, and mounts with filigree and a gemsetting.
776	608	Remains of a bracket in silver-gilt sheet, trimmed with reeded strip.
787	685	Socketed bracket in silver with wood remains.
791	536	Mount in cast silver with a bird head and low relief decoration.
827	65	Pommel in cast silver, of cocked-hat form.
904	75	Pommel in cast silver, of cocked-hat form with double sword-rings,
		with cast interlace, niello and glass decoration.
959	587	Buckle in silver with filigree decoration.
995	379	Half of a hilt-plate cast in silver.
1007	69	Pommel in cast silver, of cocked-hat form with gilded low relief
		decoration and niello inlay.
1026	188	Remains probably of a pair of hilt-collars in cast silver, of high form,
		with gold filigree mounts.
1029	372	Hilt-plate cast in silver of oval form.
1132	615	Edging in silver with a U-section.
1169	188	Remains probably of a pair of hilt-collars in cast silver, of high form,
		with gold filigree mounts.
1185	75	Pommel in cast silver, of cocked-hat form with double sword-rings,
		with cast interlace, niello and glass decoration.
1331	611	Reeded strip in silver gilt, 5mm.
1447	72	Pommel in cast silver, of round-back form.
		Continued

K	Catalogue Name in publication							
number	number							
1493^2	381	End of a hilt-plate cast in silver.						
1509	591	Helmet cheek-piece, cast in silver and gilded, with animal ornament						
		and a gold collar.						
1534	382	End fragment of a hilt-plate cast in silver.						
1566	613	Reeded strip in silver-gilt, 8mm wide.						
1623	76	Pommel in cast silver, of cocked-hat form with double sword-rings,						
		with cast interlace and niello inlay, and mounts with cloisonné and						
		filigree decoration.						
1684	71	Pommel in cast silver, of round-back form, with an animal-headed						
		band and incised ornament.						
1700	569	Mount with fantail in silver with geometric niello inlay and gilded						
		borders.						
1823	381	End of a hilt-plate cast in silver.						

² Renumbered to become K2083



E. S. Blakelock

Abstract:

At the British Museum a pilot study of die-impressed sheet fragments was carried out to determine whether surface X-ray fluorescence (XRF) analysis can be used to help identify groups of sheet fragments from individual panels or bands, and therefore aid in the conservation work. ¹ The analysis of the backs of the sheets showed a range of compositions, which overlapped between panels and therefore did not help in the identification of compositional groups linked to specific panels or bands.

This study was followed by a larger pilot study of silver hilt-plates at the Birmingham Museum and Art Gallery.² (Blakelock 2015). The XRF analysis of the fronts of hilt-plates revealed that half had been mercury gilded. The composition of most of the hilt-plate fragments fell in the range of c.75-88 wt% silver, 5-12 wt% copper, 2-3% tin and 1-5 wt% gold. In this study a thorough study of an additional 43 silver objects was undertaken. Surface analysis was carried out on various components of each object to investigate any gilding present. Sub-surface analysis was undertaken to allow comparison between silver objects of differing date, style and function.

The results have shown that the silver objects in the hoard demonstrated a range of compositions between 75-98% silver and 0-20% copper. Within many of the objects there was up to 4% tin, lead and gold and some had traces of zinc up to 1.5%.

There are few clear workshop groups within the silver objects analysed, the possible exception are the helmet components and the niello mounts. Complex pommels and the set of hilt-collars and pommel do not share the same composition. It is possible that there is an increase in lead and tin, and decrease in zinc, over time, although this is not entirely clear. However there are some clear technical choices when the data is grouped based on the method of manufacture and the applied decoration. Objects that are cast tend to have more lead than those that were worked, i.e. die-impressed sheets.

Within the assemblage of die-impressed sheets it is clear that there are several groups of foils, possibly representing different melts. The horseman and kneeling warrior panels have more zinc present than the beaked quadrupeds and the warriors moving left and right, although all are similar to the helmet components. Analysis of silver interlace and the forward facing warrior fragments confirmed that they had no gilding present and revealed a different ratio of copper to lead from the other panels which may suggest a different melt.

¹ Blakelock 2014a.

² Blakelock 2015.

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Introduction

At the British Museum a pilot study of die-impressed sheet fragments was carried out to determine whether surface X-ray fluorescence (XRF) analysis can be used to help identify groups of sheet fragments from individual panels or bands, and therefore aid in the conservation work.³ With the exception of the interlace die-impressed sheets XRF analysis of the fronts of the sheets revealed the presence of mercury gilding which prevents direct access to the silver alloy below. The analysis of the backs of the sheets showed a range of compositions, which overlapped between panels and therefore did not help in the identification of compositional groups linked to specific panels or bands. There was also evidence for contamination from the gilding process on the back of the die-impressed sheets although it was not always visibly present. The previous study demonstrated that a rapid surface XRF survey is not the appropriate technique to identify panel or band groups.

This was followed by a larger pilot study of silver hilt-plates at the Birmingham Museum and Art Gallery. The XRF analysis of the fronts of hilt-plates revealed that half had been mercury gilded. The composition of most of the hilt-plate fragments fell in the range of c.75-88 wt% silver, 5-12 wt% copper, 2-3% tin and 1-5 wt% gold. Lead and tin were present in all of the fragments. The surface analysis of the inside of the hilt-plate fragments showed a range of compositions due to surface depletion and presence of corrosion products, mostly silver chlorides. Sub-surface analysis provided more consistent and reliable results of the bulk alloy. When the sub-surface results for the different hilt-plate groups were compared to each other, it became clear that the majority overlapped.

This study forms part of a larger English Heritage-funded research project on the Staffordshire Hoard, and was funded by a grant from the Esmée Fairbairn Collections Fund. It consists of a thorough study of an additional 43 silver objects. Surface analysis was carried out on various components of each object to investigate any gilding present. Subsurface analysis was carried out to allow comparison between silver objects of differing date, style and function. There is, however, no agreed method for determining whether a silver or gold alloy is from the same 'stock', particularly when relying on major elements alone, most studies tend to rely on trace elements.

The initial pilot study of the die-impressed sheets was based on the frieze types assigned by curator David Symons and the work carried out at the British Museums reconstructing the die-impressed sheets.⁷. However during the grouping exercise held in February 2014, and another focusing on the die-impressed sheets held in April 2015, the number of panels or bands and their potential relationships were reviewed. Surface analysis of a larger number of die-impressed sheets was therefore carried out to compliment the above silver study, and to extend the smaller pilot study undertaken at the British Museum.⁸

Methodology

Instrument

The XRF analysis was carried out using a Bruker Mistral M1 fitted with a tungsten X-ray tube with a silicon drift detector. The voltage used was 40 kV with a current of 800 μ A. Experiments were carried out during the pilot study to determine the count times required for

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³ Blakelock 2014a.

⁴ Blakelock 2015.

⁵ Cool 2015

⁶ Martinón-Torres and Uribe-Villegas 2015.

⁷ Shearman *et al.* 2014.

⁸ Blakelock 2014a.

ach collimator. Surface analysis was undertaken with the 0.5mm collimator for 240 seconds and a 0.2mm collimator was used for sub-surface analysis with a longer 300 second count time.

Standards and quantification

The XRF instrument applies the fundamental parameter calculation for quantification, using pure element standards. The energy to channel calibration on the instrument was carried out by analysing a pure silver standard prior to use. The spectrum for each analysed area was examined to confirm the presence of elements being quantified especially where the peaks overlap, e.g. gold, zinc, mercury and bromine (Figure 1). Sulphur was not quantified as the air in the chamber would likely influence any results however examination of the spectra revealed no traces but the limits of detection for sulphur is likely to be high. The XRF software automatically normalised the data collected.

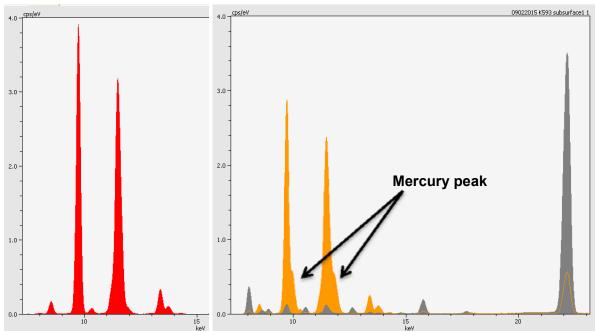


Figure 1. Screen shots of XRF spectra showing the spectra for gold (left red), compared to gilded silver (orange) with the shoulder representing the mercury peak, and a silver alloy (grey).

During the pilot study¹⁰ three silver (AGA) standards were analysed, these revealed that the Bruker XRF was overestimating the copper and lead contents when calculating the totals for the AGA standards. The largest discrepancy was the lead total given for standard AGA3, which was double that of the reference amount which may be probably partially due to the dendritic coring of the silver alloy and segregation across the sample. All XRF methods struggled to accurately quantify elements such as zinc, antimony, bismuth and iron when they were below 0.5%.

The MAC 2 standard was analysed each day the XRF was used. However the MAC2 gold standard bought for the Staffordshire Hoard project standard was not representative of the composition of the silver objects in the Hoard. This standard was therefore used for monitoring analytical precision, Table 2 and Table 3 in the appendix 1 show the daily results. Analysis was carried out with both the 0.5 collimators at 240 seconds and 0.2 collimators at 300 seconds.¹¹

¹⁰ Blakelock 2015.

⁹ Blakelock 2015.

¹¹ Blakelock 2014b.

Method and samples

Surface analysis was carried out on all the objects chosen for the silver study (Table 4 in Appendix 1). This was to confirm the absence or presence of gilding on the object. Analysis was undertaken on a range of components such as beaded or twisted wire, pins or other features. The results of this qualitative survey will be discussed for each object in appendix 3. Surface analysis was carried out on over 100 different areas from 61 different silver pieces from the hoard. In addition the fronts and backs of 53 silver die-impressed sheets fragments from the full range of panels and bands were examined.

The sub-surface areas were prepared by scraping the surface in a similar manner to the gold analysis project. ¹² In the majority of cases the sub-surface area was prepared on the inside or back of the object but some areas were also prepared on the edge. The sub-surface areas were also analysed using the 0.2 collimator for 300 seconds. Sub-surface areas were prepared on 60 individual pieces from the hoard (Table 2 in the appendix), where individual objects were constructed from multiple parts, i.e. main body, shoulders and pommel rings each was analysed for comparison. This total includes the hilt-plates analysed in the pilot study. ¹³

Results and discussion

Standards and quantification

The results from the analysis of the MAC2 standards are shown in Table 1. The results given by the BMT XRF when using the 0.5 collimator was similar to those obtained using the British Museum XRF. The SEM-EDX system at the British Museum appears more accurate than the XRF systems, but this is because the MAC2 standard was used to calibrate the SEM-EDX. There are some differences between the XRF systems; the Mistral M1 XRF produces a lower copper reading compared to the British Museum XRF. Analysis using the 0.2 collimator showed a reasonable correlation to the 0.5 collimator, with the exception of tin which had a bigger range of compositions and often a lower total than the 0.5 collimator. Even so the results are within expectable margins for semi-quantitative analysis.

		Colimator (mm)	Count time (seconds)	Au (%)	Ag (%)	Cu (%)	Sn (%)
				74.7	19.2	5.1	1.03
BMT	XRF	0.5	240	75.7	18.6	4.5	1.2
				0.12	0.06	0.06	0.05
		0.2	300	76.2	18.6	4.5	0.7
				0.31	0.27	0.07	0.31
BM	XRF	0.65	200	75.0	18.9	5.0	1.1
				0.00	0.09	0.08	0.05
BM	SEM-EDX		150	74.3	19.6	5.1	1.0
				0.14	0.14	0.06	0.08

Table 1. Shows the results from the analysis of the MAC2 standard.

Gilding

The majority of the objects chosen for the silver project had some gilding present, and in all cases mercury was present confirming mercury amalgam method of gilding had been used

¹² Blakelock 2014c.

¹³ Blakelock 2015.

to decorate the Staffordshire Hoard silver. This is typical for the Anglo-Saxon period and has been observed in other studies. ¹⁴ Even where no gilding was visible on the surface of objects there was often an increased gold and mercury content to indicate gilding.

There are two methods of gilding with mercury: hot and cold. Pliny writing in 77-79 AD describe a gilding method where a coat of mercury is applied underneath the gold leaf, and there is no mention of heat applied, instead the mercury acts as an adhesive. ¹⁵ 'after a thorough polishing with a mixture of pumice and alum, it is able to take the gold leaf laid on with quicksilver'. ¹⁶ Over time the mercury would evaporate slowly from the surface of the gilded object. ¹⁷ Amalgam gilding was carried out by grinding together small quantities of gold with excess mercury, to form a paste of gold amalgam. ¹⁸ Examples of mortars for this process dated to the middle-late Saxon period have been identified at Hamwic. ¹⁹ The paste was applied to the object to be gilded and then heated which burns off the excess mercury leaving a firmly bonded porous layer of gold. ²⁰ This layer is then burnished smooth. ²¹ Such methods have been documented in the seventh-eighth century *Codex Lucensis 490*, the ninth century Mappae Clavicula (see below), Theophilus's 'On Diver Arts' and Biringuccio's 'Pirotechnia'. ²²

Take an extremely thin gold sheet, shear it into tiny pieces and put it in a mortar. Add a little quicksilver and leave it for a short time. Afterwards, add some natron and vinegar; rub it thoroughly with a pumice stone until it has the consistency of glue on account of the abundance of quicksilver. And now you put it in a clean cloth and squeeze it, so that most of the quicksilver comes out. Then you take the vessel [that is to be gilded], polish it with fine pumice, heat it, and while it cools coat it with the amalgam, and you heat the vessel a second time and again coat it and put it on the fire. And the gold alone becomes enriched. After a little when the colour pleases you, heat the vessel and put it into a blacking liquid, i.e. the boot dressing, with which leather things are blackened. And then rub it.

Excerpt from the Mappae Clavicula on gilding copper and silver vessels.²³

Scientifically, it is difficult to distinguish between cold or hot gilding methods, as in both cases analysis will detect some mercury in the layer of gilding. There are no contemporary goldworking documentary sources, however the Leyden Papyri dated to the third to fourth century AD applies embers to the surface of a gilded object so may be describing fire gilding. ²⁴ In addition, the sub-surface analysis also revealed some objects with higher mercury present below the surface. This does suggest that the objects were mercury gilded and then heated, as the heat would allow the mercury to bond with the core metal. This layer formed in the metal is a known phenomenon and has been noted in previous metallographic analysis of gilded silver objects where the diffusion layer can be up to 10 µm thick. ²⁵ Ideally to achieve the core composition further scrapes would be required until no further mercury was detected, however in some cases the damage caused by deeper scrapes, or larger areas would be too destructive.

²¹ Northover and Anheuser 2000.

 $^{^{14}}$ Northover and Anheuser 2000, 13.

¹⁵ Oddy 2000, 5.

¹⁶ Rackham 1952, 51, Natural History XXX.20.

¹⁷ Oddy 2000, 5.

¹⁸ Northover and Anheuser 2000, Oddy 2000.

¹⁹ Bayley and Russel 2008.

²⁰ Oddy 2000.

²² Burnham 1920, 127-8; Hawthorne and Smith 1979; Smith and Gnudi 1990; Smith and Hawthorn 1974.

²³ Smith and Hawthorne 1974, 36.

²⁴ Caley and Jensen 2008, 30-31.

²⁵ Dandridge 2000; Northover and Anheuser 2000.

The compositional differences seen between gilded fragments are likely to be related to the thickness of the gilding layer, with part or most of the silver detected being located in the silver underneath the gilding layer. The variable thickness of the gilded layer may be affected by different manufacturing processes, but is equally likely to relate to wear during use or in the post-depositional environment.

Silver compositions

The silver objects in the hoard demonstrated a range of compositions between 75-98% silver and 0-20% copper (Table 5 in Appendix 1). Within many of the objects there was up to 4% tin, 4% lead and 4% gold; some had traces of zinc up to 1.5%. However the pilot study showed that when standards with low concentrations (below 0.5%) were analysed the XRF tended to overestimate these totals. It is therefore possible that totals given for alloys with less than 0.5% tin, lead, zinc or gold may not be accurate.

The analysis has shown that there is a consistent presence of gold within the silver (Figure 2, top left), even taking into account the few objects contaminated due to the gilding. There are two possible explanations. The first is that auriferous silver was being used and this had not been parted to remove the gold after the cupellation process. Alternatively the silver stock has been contaminated with gold, perhaps from the recycling of gilded silver.

There is an apparent relationship between lead and tin contents in the silver alloys, and when the sums of both are plotted against copper there is also a good correlation (Figure 2). This suggests that the lead and tin may have entered along with the copper, in the form of a leaded bronze alloy. Leaded bronze alloys were the most common copper alloy in the hoard, ²⁶ and therefore it is not too unexpected. The presence of zinc in the silver suggests that it also entered with the copper, confirmed by the plot (Figure 2), as any zinc present in the silver ore would have evaporated during smelting and cupellation processes, since zinc has a boiling point lower than the silvers melting point. It is therefore likely that pieces of brass or gun metal were also occasionally added to the silver.

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²⁶ Blakelock 2016

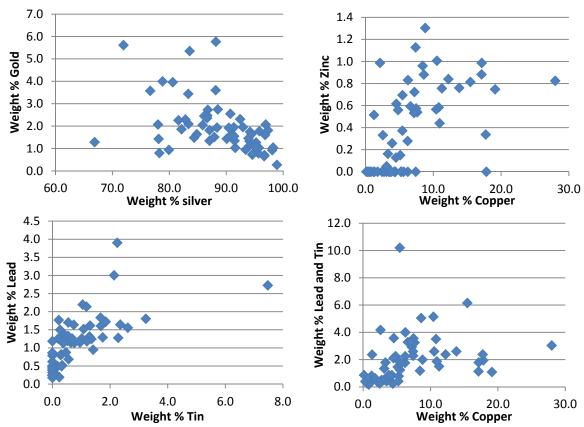


Figure 2. Gold/silver, zinc/copper, lead/tin and lead and tin/copper bi-variate graphs showing all the data collected during this study.

Given the above observations the silver analysed from the Staffordshire Hoard is remarkable in its homogeneity, compared to the gold in the Hoard which had a much larger range of silver-gold compositions (Figure 2).²⁷ However the results suggest that the silver in the Hoard is the result of recycling and mixing of silver, gilded silver and probably fragments of copper alloys.

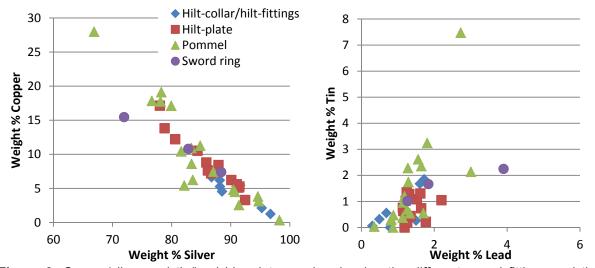


Figure 3. Copper/silver and tin/lead bi-variate graphs showing the different sword fittings and the overlap.

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²⁷ Blakelock 2014c

When the results from the various sword fittings are plotted it is clear that the data overlaps (Figure 3). The pommels show the largest variability and spread, whereas the hilt-plates and hilt-collars tend to cluster together. There appears to be no clear choice of alloy for any one type of sword fitting. The only possible exception are the hilt-collars analysed which are generally higher in silver than the hilt-plates and pommels (Figure 3). However this may be due to other factors as the majority of hilt-collars examined had niello decoration.

When the remaining object data are plotted alongside the sword fittings (Figure 4, Figure 5 and Figure 19 in Appendix 2) it is clear that there is much more variability in composition in the sword fittings when compared to the niello mounts, helmet fragments and other miscellaneous objects (such as the buckle, silver bracket, sword pyramid). The frequency distribution histogram shows quite clearly the normal distribution seen in the data collected for the sword fitments, which is quite different to those for the niello mounts and helmet fittings (Figure 20 in Appendix 2). The niello mounts are almost pure silver with very few impurities, the copper and lead contents often below 1% with a relatively high gold content between 0.9-2.1%. The helmet components form a separate group, with a high silver content and between 2.7-4.4% copper. Like with the niello mounts there is no tin or zinc present and only small quantities of lead c. 0.4% and gold c. 1-2% present. This is remarkable considering the evidence for recycling seen in the silver alloy compositions and suggests that the Anglo-Saxon goldsmiths recognised and separated purer silver for when it was required.

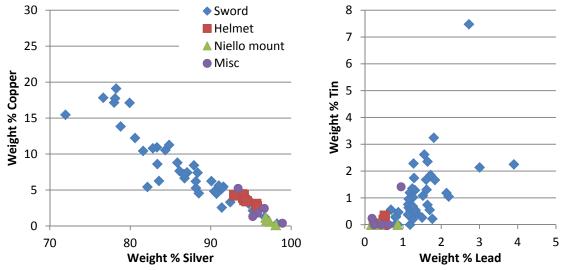


Figure 4. Copper/silver and tin/lead bi-variate graphs showing the difference between the sword fittings and the niello mounts and helmet pieces.

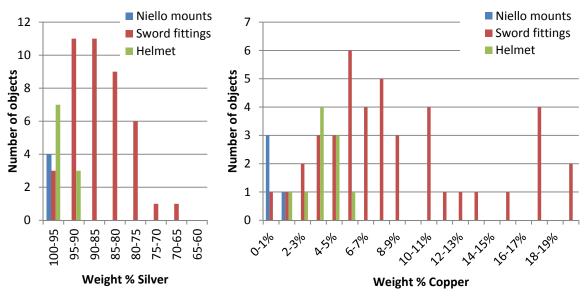


Figure 5. Frequency distribution histograms for silver and copper showing the normal distribution for the sword fittings, compared to the helmet and niello.

Workshop groups and sets

Since the grouping exercise a number of complex pommels have been pieced together, these consist of a main body, but with attached shoulders, rings, gold panels and inlays (Figure 6 and Figure 7). These objects appear to have been cast in multiple parts (body, shoulder, ring, etc) before being joined. Therefore during the analysis a number of parts from these pommels were examined.

The results show that the parts that make up pommel catalogue no. 77 are very similar, whereas there are clear differences between the parts that make up pommels catalogue nos. 76 and 75 (Figure 8). The internal panel of catalogue no. 75 is higher in copper, which may have been decorative (Figure 7) since the alloys used in the other objects tend to be more similar (Figure 8).



Figure 6. Pommels catalogue no. 77 (left) and 76 (right).



Figure 7. Pommel catalogue no. 75 and a detailed photo of the copper rich decorated part.

The sword rings from these pommels generally have a different composition from their respective pommel, suggesting they were a different melt. However considering that different components of the same pommel had distinctive compositions there is not enough evidence to confirm whether the rings were intentionally part of the original pommel construction.

A pair of silver hilt-collars (catalogue numbers 186-7) and a pommel (catalogue no. 69) were noted for having a similar design, decorated with niello set into channels and gilding. To determine whether they were a set, and constructed at the same time they were analysed. The potential set shares similar concentrations of lead, tin, zinc and gold. The pommel consists of two fragments which had similar silver and copper concentrations, however the collars, while similar to each other, had a higher silver content (Figure 8) than the pommel. The results suggests that the set was created during two different melts, but due to the differences it cannot be ascertained whether they were constructed at the same time, although the similar design and decoration suggests at least the same workshop.

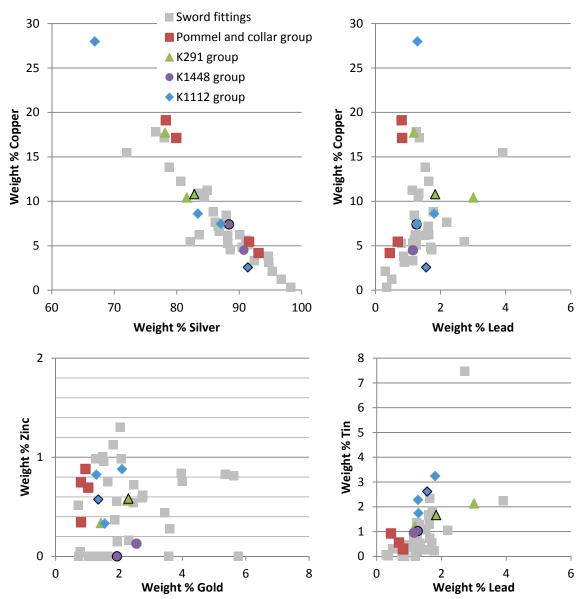


Figure 8. Copper/silver, copper/lead, zinc/gold and tin/lead bi-variate graphs showing the pommel and collar group (red squares), or the complex multi-component pommels with their sword rings (show using a black outline). (Note K291 group is pommel catalogue no. 76; K1448 group is catalogue no. 77 and K1112 group is catalogue no. 75)

Dates

Figure 6 shows the data grouped the objects based on the phases suggested by Chris Fern. Initial observation of the objects dated to 600-650 AD suggests that they have a specific composition, but this relates to the niello and helmet groups noted previously (Figure 9). This is perhaps confirmed by the results from K1684 (catalogue no. 71) which was also dated to the 625-650 and is the single outlier to this group. If the niello mounts and helmet are removed from the dataset, as a distinct group, there is a possible increase in lead and tin, and decrease in zinc, over time (Figure 10).

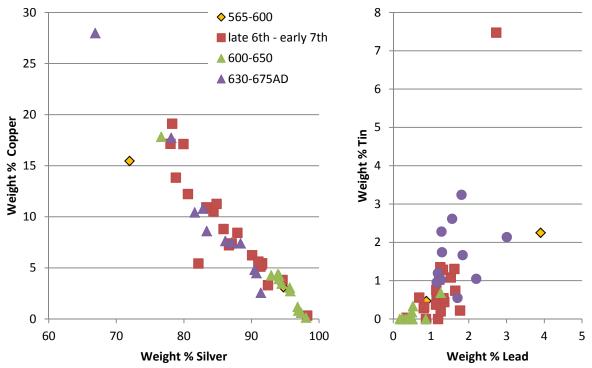


Figure 9. Copper/silver and tin/lead bi-variate graphs showing the objects through time.

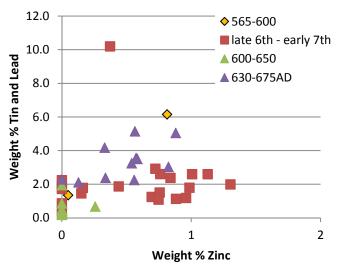


Figure 10. Tin and lead against zinc bi-variate graph showing the objects through time, excluding the helmet pieces and niello mounts.

Decoration types

When the data is grouped based on the type of applied decoration it is clear that there are some differences (Figure 21 in Appendix 2). The first most obvious pattern is that the objects with niello decoration tend to be higher silver (Figure 11 here and Figure 21 in Appendix 2). In the copper histogram the niello objects form two separate groups, one at 0-3% copper and another at 5-9% copper, whereas the objects without niello form a positive skew distribution at 3-8% copper (Figure 11). However, the box and whisker plots show that there is no significant difference in the silver and copper contents of objects with niello to those without (Figure 12). The range of lead and zinc contents are similar but the averages are different, which may suggest a deliberate choice of alloy for some of the niello objects, such as the mounts.

Pure silver-sulphide niello will melt at 861°C, whereas silver with few other alloying elements (up to 10%) would melt at over 900°C. However niello heated at 820°C in an oxidising environment will begin to decompose to pure silver, before it reaches the melting temperature. When the niello is over 600°C it becomes softer and it would be possible to compact it into any channels without the need to melt the niello into place. (Untracht 1975), therefore having a purer silver is not required. It is therefore more likely it was an aesthetic choice made by the goldsmith.

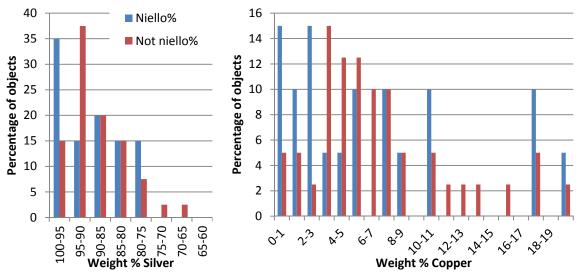


Figure 11. Frequency distribution histograms for silver and copper showing the distribution for the objects without niello compared to those decorated with niello.

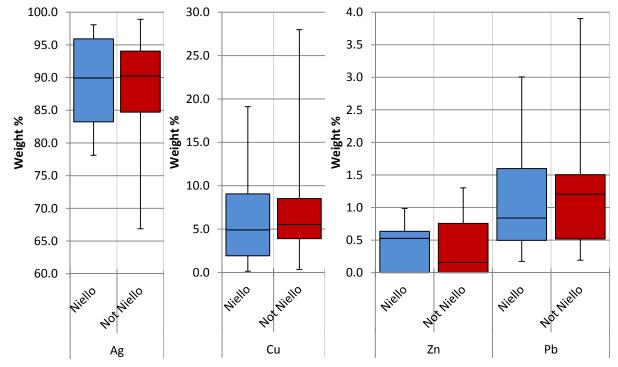


Figure 12. Box and whisker plots showing no significant between objects with niello compared to objects without niello.

²⁸ La Niece 1983; Ogden 1982.

²⁹ Untracht 1975.

At first glance there appears to be no obvious relationship between core composition and gilding (Figure 21 in Appendix 2). However the frequency distribution histograms suggest that the gilded objects tend to have a higher silver content, with lower quantities of copper and zinc, than those objects that are not gilded (Figure 13 and Figure 14). The box and whisker plots for the gilded objects compared to those objects without gilding show that there is clear overlap between the two groups (Figure 15). They do, nevertheless, confirm that there are also differences in the distribution of the two groups, similar to those seen in Figure 13 and Figure 14. Many of the past descriptions of gilding³⁰ advise to remove the surface layers formed by 'the fire and the hammer. If the skin is not scraped off, when the silver is gilded and repeatedly coloured over the fire for a long time, tiny blisters will be raised up here and there, and the silver will show through when they break'. Fire gilding will therefore work better on a metal that has fewer impurities that will oxidise. Theophilus is particularly concerned when leaded brass is gilded, and suggests 'it should be completely pure and purged of lead', however it is likely that this is due to the lead segregation that occurs in copper alloys, and is not necessarily relevant to silver.

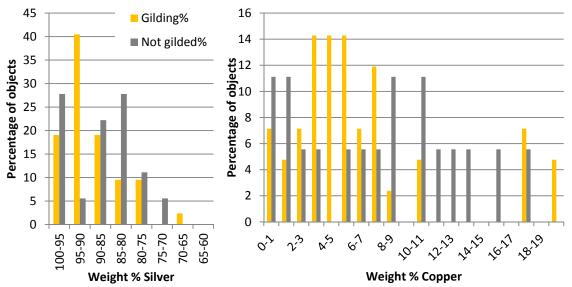


Figure 13. Frequency distribution histograms for silver and copper showing the distribution for the objects without gilding compared to those gilded.

³⁰ Smith and Gnudi 1990, 96; Smith and Hawthorne 1974, 60.

³¹ Hawthorne and Smith 1979, 118.

³² Hawthorne and Smith 1979, 145.

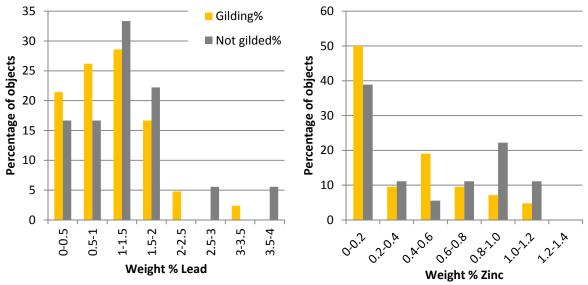


Figure 14. Frequency distribution histograms for lead and zinc showing the distribution for the objects without gilding compared to those gilded.

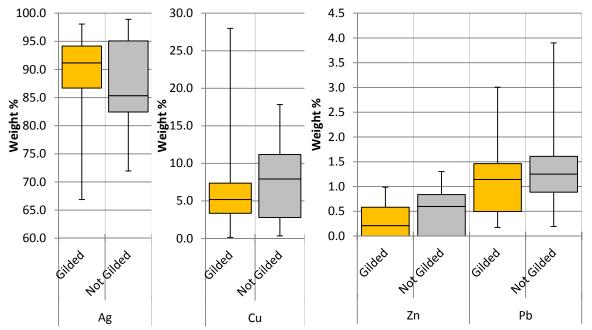


Figure 15. Box and whisker plots showing overlap between the compositions of gilded objects compared to objects without gilding.

Lead is present in the majority of the other objects, particularly the objects that were cast. However the helmet die-impressed sheets, discussed below, have low concentrations of lead present; lead is detrimental to cold working so the goldsmith most likely made a choice not to add this to the alloy.

Silver die-impressed sheets and the helmet

Analysis was undertaken of a small number of die-impressed sheets at the British Museum. This new study however analysed the surface of a larger number of fragments from the full range of designs. It was carried out alongside the die-impressed sheets grouping, reconstruction and conservation work carried out at Birmingham Museum and Art Gallery. As no sub-surface analysis was carried out the results from this survey should be treated as

qualitative. However there were several differences observed in the die-impressed sheets examined.

The majority of the sheets had been gilded, the only exceptions were the zoomorphic interlace and the forward facing warrior fragments. Analysis of the back of these fragments also revealed that they had a different ratio of copper to lead from the other panels, which may suggest a different melt (Figure 16 and Figure 17). The other observation was that the horseman and the kneeling warrior sheets had a higher concentration of zinc present; however the concentrations were only slightly above the detection limits of the XRF so the values given are unlikely to be accurate (Figure 16). The presence of zinc in these sheets may suggest that they represent a different melt from the other sheets; however the similarity in the other elements present probably suggests that they are still part of the same helmet.

The analysis of the moustached warriors revealed that they also had higher concentrations of zinc. However the gilding present is much thicker, and there was gold and mercury present on the backs of these sheets. The gilding present on the front and the backs makes it impossible to accurately determine the composition of the sheet metal used for comparison. Therefore based on these results it is not possible to determine whether they are part of the helmet, and therefore they have not been included in the following plots.

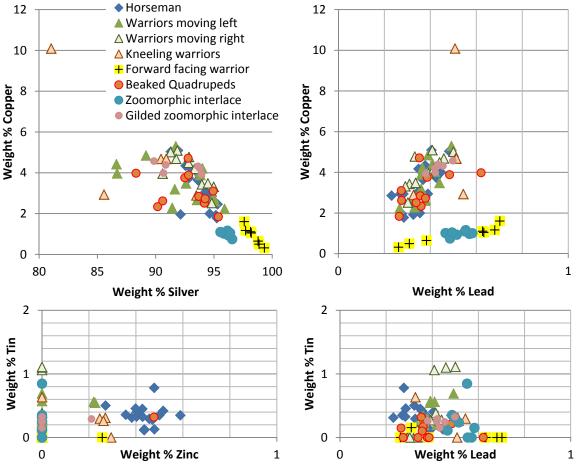


Figure 16. Copper/silver, copper/lead, tin/zinc and tin/lead bi-variate graphs showing the different die-impressed sheets

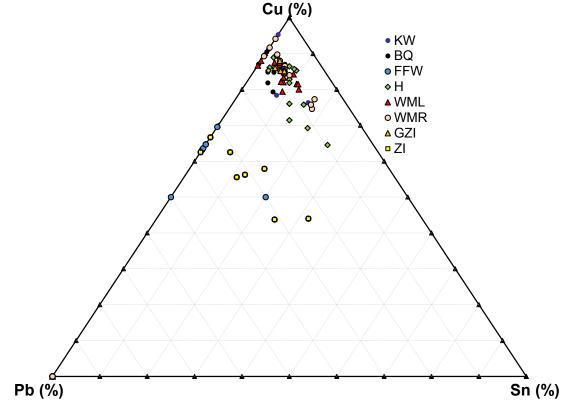


Figure 17. Ternary copper-lead-tin diagram of the die-impressed sheets showing two distinct groups.

The die-impressed sheets and the other helmet components are a similar composition (Figure 18 and Figure 22 in Appendix 2). The analysis of the 8mm, 11mm, and 14mm reeded strips revealed a similar composition to the helmet parts as well as the silver and gilded sheets. The surface analysis of the 5mm reeded strips suggested that they were related to zoomorphic and forward facing warriors, but there were high levels of mercury detected on the back which has resulted in a higher gold and decreased copper content (Figure 22 in Appendix 2). The sub-surface analysis confirms that some contamination has taken place, with an increase of gold at the surface most likely due to the mercury gilding. The core composition suggests that the 5mm strip more likely related to the other reeded strips (Figure 18).

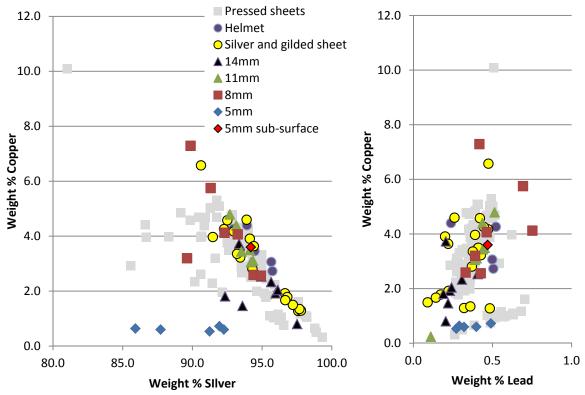


Figure 18. Copper/silver and copper/lead showing the different die-impressed sheets, the ribbed strips and other helmet components. The arrow highlights the sub-surface results from the 5mm ribbed strip which is clearly different to the surface analyses.

Conclusion

The silver objects in the hoard demonstrated a range of compositions between 75-98% silver and 0-20% copper. Within many of the objects there was up to 4% tin, lead and gold and some had traces of zinc up to 1.5%.

There are few clear workshop groups within the silver objects analysed, the possible exceptions are the helmet components and the niello mounts. Different components on the same complex pommels and the set of hilt-collars and pommel do not share the same composition. It is possible that there is an increase in lead and tin, and decrease in zinc, over time, perhaps a result of natural loss each time the alloy is re-melted, although this is not entirely clear. However there are some potential technical choices when the data is grouped based on the method of manufacture and the applied decoration. Objects that are cast tend to have more lead than those that were worked, i.e. die-impressed sheets.

Within the assemblage of die-impressed sheets it is clear that there are several groups of foils, possibly representing different melts. The horseman and kneeling warrior panels have more zinc present than the beaked quadrupeds and the warriors moving left and right, although all have a similar composition to the helmet components. Analysis of silver interlace and the forward facing warrior fragments confirmed that they had no gilding present and revealed a different ratio of copper to lead from the other panels which may suggest a different melt.

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Eleanor Blakelock October 2015

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Appendix 1. Tables of data

Date	Au (%)	Ag (%)	Cu (%)	Sn (%)
26/03/2015	75.6	18.6	4.6	1.2
26/03/2015	75.8	18.6	4.5	1.1
26/03/2015	75.6	18.7	4.6	1.1
26/03/2015	75.7	18.7	4.5	1.1
27/03/2015	75.7	18.7	4.5	1.1
27/03/2015	75.6	18.7	4.6	1.1
27/03/2015	75.7	18.7	4.4	1.2
27/03/2015	75.6	18.7	4.5	1.2
27/03/2015	75.5	18.7	4.6	1.2
27/03/2015	75.8	18.6	4.5	1.1
27/03/2015	75.6	18.6	4.6	1.2
27/03/2015	75.7	18.6	4.5	1.2
19/05/2015	75.7	18.7	4.5	1.1
21/05/2015	75.7	18.7	4.4	1.2
22/05/2015	75.5	18.7	4.6	1.2
26/05/2015	75.6	18.7	4.5	1.2
29/05/2015	75.7	18.7	4.4	1.2
10/06/2015	75.7	18.7	4.4	1.2
11/06/2015	75.8	18.6	4.5	1.1
12/06/2015	75.6	18.7	4.5	1.2
15/06/2015	75.8	18.5	4.5	1.2
16/06/2015	75.6	18.7	4.5	1.2
17/06/2015	75.6	18.7	4.5	1.2
18/06/2015	75.6	18.7	4.5	1.2
22/06/2015	75.6	18.8	4.5	1.1
24/06/2015	75.5	18.7	4.5	1.3
26/06/2015	75.8	18.6	4.5	1.1
Average	75.6	18.7	4.5	1.2
Standard Deviation	0.08	0.06	0.06	0.05

 Table 2. Results from the daily analysis of standard MAC2 with the 0.5 collimator for 240 seconds.

XRF study of silver objects from the Staffordshire Hoard

Date	Au (%)	Ag (%)	Cu (%)	Sn (%)
18/02/2015	76.5	18.3	4.5	0.7
24/02/2015	76.2	18.6	4.4	0.8
25/02/2015	76.5	19.0	4.5	0.0
03/03/2015	76.7	18.4	4.4	0.5
04/03/2015	76.1	18.6	4.6	0.7
05/03/2015	75.8	18.7	4.4	1.1
30/04/2015	76.3	18.8	4.5	0.4
01/05/2015	76.6	19.0	4.4	0.0
22/05/2015	76.4	19.0	4.6	0.0
29/05/2015	76.4	18.3	4.4	0.9
10/06/2015	76.1	18.9	4.4	0.6
11/06/2015	76.4	18.4	4.4	0.8
12/06/2015	76.1	19.0	4.5	0.4
15/06/2015	76.7	18.8	4.5	0.0
16/06/2015	76.4	18.4	4.5	0.7
17/06/2015	76.2	18.3	4.5	1.0
18/06/2015	76.7	18.7	4.3	0.3
24/06/2015	77.1	18.3	4.6	0.0
26/06/2015	76.3	18.7	4.4	0.6
Average	76.3	18.6	4.4	0.7
Standard Deviation	0.30	0.27	0.07	0.36

Table 3. Results from the daily analysis of standard MAC2 with the 0.2 collimator for 300 seconds.

					De	ecoration
Object	Object type	Set/groups	Date/Phase	Gilding	Niello	Other
K179	Hilt-plate	Hilt-plate pair A	late 6th - early 7th			
K522	Hilt-plate	Hilt-plate pair A	late 6th - early 7th			
K13	Hilt-plate	Hilt-plate pair B	late 6th - early 7th	У		
K995	Hilt-plate	Hilt-plate pair B	late 6th - early 7th	y		
K1029	Hilt-plate	Hilt-plate pair C	late 6th - early 7th	•		
K239	Hilt-plate	Hilt-plate pair C	late 6th - early 7th			
<138	Hilt-plate	Hilt-plate pair D	late 6th - early 7th	у		
K593	Hilt-plate	Hilt-plate pair D	late 6th - early 7th	ý		
K1534	Hilt-plate	Hilt-plate pair E	late 6th - early 7th	,		
K1493	Hilt-plate	Hilt-plate pair E	late 6th - early 7th			
K248	Hilt-plate		late 6th - early 7th	У		
K1823a	Hilt-plate	Hilt-plate pair F	late 6th - early 7th	y		
K1823b	Hilt-plate	Hilt-plate pair F	late 6th - early 7th	ý		
K63	Hilt-guard	Time process process	630-675AD	y	у	
K298	Hilt-collar/hilt-fittings		000 01 01 12	y	?	
K369	Hilt-collar			y	у	
K577	Hilt-fitting			y	y	
K791	Hilt-fitting			У	,	
K1026	Hilt-collar			У	у	
K1169	Hilt-collar			У	,	
K304	Hilt-collar	Group 1	late 6th - early 7th	y	У	
K306	Pommel	Cioup i	late 6th - early 7th	,	,	
K286	Pommel		late 6th - early 7th	some		Garnet
K1447	Pommel		iate our carry rur	301110		Gamet
K456	Pommel		late 6th - early 7th	some		
K827	Pommel		late 6th - early 7th	301110		
K294	Pommel		630-675	у		Gold panel
K559	Pommel		late 6th - early 7th	у		Gold pariel
K1684	Pommel		625-650			
K39	Pommel	Group 1	late 6th - early 7th	٧	У	
K1007	Pommel	Group 1	late 6th - early 7th	,	-	
K1623	Sword ring	K291	630-675AD	y V	y V	Gold panel, garnet, glass
C1023	Pommel rivet	K291	630-675AD	,	,	Gold panel, garnet, glass
K1385	Pommel body	K291	630-675AD	y y	y V	Gold panel, garnet, glass
<1303 <1448	Pommel ring	K1448	630-675AD	•	У	Gold panel, rock crystal
<762	Pommel body	K1448	630-675AD	y V		Gold panel, rock crystal
K744	Pommel decorative panel	K1446 K1112	630-675AD	,	V	Gold panel, glass
K904	Pommel shoulder	K1112 K1112	630-675AD	У	У	Gold panel, glass Gold panel, glass
K1185	Pommel body	K1112 K1112	630-675AD	у	У	Gold panel, glass Gold panel, glass
11185 1290	, , , , , , , , , , , , , , , , , , ,			У	У	1 , 0
K290 K711	Sword ring	K1112	630-675AD	У	У	Gold panel, glass
	Pommel Sword ring	K711?	565-600AD	у	у	
<543	Sword ring	N/TI!				

 Table 4. List of objects that were analysed by sub-surface analysis.

					Decoration			
Object	Object type	Set/groups	Date/Phase	Gilding	Niello	Other		
K310	Niello mount		600-650	some	У			
K64	Niello mount		600-650	border	у			
K1700	Niello mount		600-650	border	у			
K241	Niello mount		600-650	border	у			
K787	Silver bracket							
K302	Sword pyramid			y		Gold panel, garnet		
K274	Misc							
K959	Buckle					Gold wire		
K176	c-tubing			у				
K1132	c-tubing							
K1331	5mm ribbed			y				
K776	14mm ribbed		600-650	у				
K1566	8mm ribbed		600-650	у				
K453	Helmet cheek piece	K453		у	у			
K1509	Helmet cheek piece	K453	600-650	y	у			
K363	Helmet animal head		600-650	у				
K397	Helmet animal head		600-650	y				
K546	Helmet crest		600-650	у				
K1734	Helmet tray	K235	600-650	У				

Table 4 cont. List of objects that were analysed by sub-surface analysis.

							Aver	ages				
	Object type	Date/Phase	Description	No of Analyses	Ag (%)	Cu (%)	Sn (%)	Zn (%)	Pb (%)	Au (%)	Total	Notes
K1331	5mm ribbed		Gilding	6	94.2	3.6	0.0	0.0	0.5	1.7	99.8	
K776	14mm ribbed	600-650	Gilding	5	94.1	4.3	0.0	0.0	0.3	1.3	99.4	0.5-0.7% Hg
K1566	8mm ribbed	600-650	Gilding	4	94.0	3.9	0.2	0.3	0.5	1.1	99.8	0.1-0.2% Hg
K363	Helmet animal head	600-650	Gilding	4	93.9	4.4	0.0	0.0	0.2	1.5	98.9	0.8-1.3% Hg
K397	Helmet animal head	600-650	Gilding	5	94.5	3.5	0.0	0.0	0.4	1.6	99.3	0.2-1.4% Hg
K1509	Helmet cheek piece	600-650	Niello & gilding	6	95.7	3.1	0.0	0.0	0.5	0.8	99.3	0.5-0.8% Hg
K546	Helmet crest	600-650	Gilding	4	95.7	2.7	0.0	0.0	0.5	1.0	100.0	
K235 (K1734)	Helmet	600-650	Gilding	10	92.9	4.3	0.3	0.0	0.5	2.0	99.7	0.2-0.3% Hg
K453	Helmet cheek piece		Gilding	4	62.6	2.1	0.0	0.0	0.4	34.9	94.3	4-7% Hg
K298	Hilt-collar/hilt-fittings		Niello & gilding	4	96.7	1.3	0.3	0.5	0.5	0.7	100.0	
K369	Hilt-collar		Niello & gilding	3	95.2	2.1	0.1	1.0	0.3	1.3	99.7	
K577	Hilt-fitting		Niello	4	88.2	5.3	0.0	0.0	8.0	5.8	96.0	3-4% Hg
K791	Hilt-fitting		Gilding	4	88.1	6.2	0.3	0.3	1.5	3.6	95.2	0-0.5% Fe, 3-5.5%Hg
K1026	Hilt-collar		Gilding	5	88.5	4.5	1.9	0.6	1.7	2.7	99.7	
K1169	Hilt-collar		Gilding	8	86.8	6.6	1.7	0.6	1.6	2.7	97.3	1.5-3% Fe
K304	Hilt-collar	late 6th - early 7th	Niello & gilding	5	91.6	5.5	0.6	0.7	0.7	1.0	98.6	0.1-0.2% Hg, 0.4-0.7% F
K179	Hilt-plate	late 6th - early 7th	Plain	5	87.9	8.4	0.0	1.0	1.2	1.5	100.0	
K522	Hilt-plate	late 6th - early 7th	Plain	4	85.9	8.8	0.2	1.3	1.8	2.0	100.0	
K13	Hilt-plate	late 6th - early 7th	Gilding	5	91.3	5.1	0.2	0.2	1.3	1.9	99.9	
K995	Hilt-plate	late 6th - early 7th	Gilding	5	92.4	3.3	0.6	0.2	1.1	2.3	99.9	
K1029	Hilt-plate	late 6th - early 7th	Plain	5	78.8	13.8	1.1	0.8	1.5	4.0	98.9	0.9-1.3% Fe
K239	Hilt-plate	late 6th - early 7th	Plain	3	80.6	12.2	0.7	0.8	1.6	4.0	99.2	0.7-0.8% Fe
K138	Hilt-plate	late 6th - early 7th	Gilding	4	78.0	17.1	0.4	1.0	1.3	2.1	97.6	2-3% Fe
K593	Hilt-plate	late 6th - early 7th	Gilding	5	86.7	7.2	1.3	0.7	1.6	2.5	100.0	
K1534	Hilt-plate	late 6th - early 7th	Plain	6	84.4	10.5	1.3	1.0	1.3	1.5	99.9	0-0.4% Fe
K1493	Hilt-plate	late 6th - early 7th	Plain	4	87.1	7.4	1.4	1.1	1.2	1.8	99.3	0.5-0.8% Fe
K248	Hilt-plate	late 6th - early 7th	Gilding	5	90.1	6.2	1.0	0.0	1.2	1.4	99.3	0.5-0.9% Hg
K1823a	Hilt-plate	late 6th - early 7th	Gilding	4	91.5	5.3	0.8	0.0	1.1	1.3	99.6	0.3-0.5% Hg
K1823b	Hilt-plate	late 6th - early 7th	Gilding	5	91.0	5.6	0.5	0.0	1.2	1.6	99.3	0.5-0.8% Hg
K63	Hilt-guard	630-675AD	Niello & gilding	7	86.1	7.6	1.0	0.5	2.2	2.4	90.4	8-10% Fe
K310	Niello mount	600-650	Niello & gilding	6	97.3	0.6	0.0	0.0	0.3	1.8	100.0	
K64	Niello mount	600-650	Niello & gilding	4	96.8	1.2	0.0	0.0	0.4	1.6	99.4	0.3-1% Hg
K1700 group	Niello mount	600-650	Niello & gilding	7	98.1	0.1	0.0	0.0	0.9	0.9	99.9	•
K241	Niello mount	600-650	Niello & gilding	8	96.9	0.8	0.0	0.0	0.2	2.1	99.9	

 Table 5. Sub-surface XRF data from the silver study.

						Averages						
	Object type	Date/Phase	Description	No of Analyses	Ag (%)	Cu (%)	Sn (%)	Zn (%)	Pb (%)	Au (%)	Total	Notes
K306	Pommel	late 6th - early 7th	Front	5	94.6	3.8	0.0	0.0	0.9	0.7	100.0	
K286	Pommel	late 6th - early 7th	Plain, some gilding	5	84.8	11.3	0.4	8.0	1.1	1.6	99.5	0-0.6% Fe
K1447	Pommel		Plain	5	83.6	6.3	2.4	8.0	1.6	5.3	100.0	
K456	Pommel	late 6th - early 7th	Plain, some gilding	4	98.2	0.3	0.0	0.0	0.4	1.0	99.6	
K827	Pommel	late 6th - early 7th	Plain, some gilding	5	82.1	5.4	7.5	0.4	2.7	1.9	100.0	
K294	Pommel	630-675	Gold panels, gilding, punched	5	90.5	4.8	0.6	0.6	1.7	1.9	99.8	0.1-0.2% Hg
K559	Pommel	late 6th - early 7th	Plain	5	83.3	10.9	0.5	0.4	1.3	3.4	99.6	0-0.5% Fe
K1684	Pommel	625-650	Gilded	6	76.6	17.8	0.7	0.0	1.3	3.6	100.0	
K39	Pommel	late 6th - early 7th	Niello & gilding	5	78.3	19.1	0.3	0.7	8.0	8.0	91.4	6-12% Hg
K1007	Pommel	late 6th - early 7th	Niello & gilding	3	79.9	17.1	0.3	0.9	0.8	0.9	99.4	0.3-0.6% Hg
K1623	Sword ring	630-675AD	Niello, gold panels & gilding	5	82.8	10.8	1.7	0.6	1.8	2.3	98.8	0.5-1.3% Fe
K242	Pommel rivet	630-675AD	Niello, gold panels & gilding	5	78.1	17.7	1.2	0.3	1.2	1.4	99.1	0.8-1% Fe
K1385	Pommel body	630-675AD	Niello, gold panels & gilding	5	81.6	10.4	2.1	0.6	3.0	2.3	98.1	0.2-0.3% Hg, 1.4-1.9% Fe
K1448	Sword ring	630-675AD	Niello, gold panels & gilding	5	88.4	7.4	1.0	0.0	1.3	1.9	100.0	
K762	Pommel body	630-675AD	Niello, gold panels & gilding	5	90.7	4.5	1.0	0.1	1.1	2.5	99.7	
K744	Pommel decorative panel	630-675AD	Niello, gold panels & gilding	5	66.9	28.0	1.7	8.0	1.3	1.3	99.5	0.5-0.9% Fe
K904	Pommel shoulder	630-675AD	Niello, gold panels & gilding	6	87.1	7.5	2.3	0.6	1.3	1.3	95.7	3-4% Fe
K1185	Pommel body	630-675AD	Niello, gold panels & gilding	3	83.4	8.6	3.2	0.9	1.8	2.1	97.6	1.2-1.5% Fe
K290	Sword ring	630-675AD	Niello, gold panels & gilding	7	91.4	2.6	2.6	0.3	1.6	1.5	99.6	0-0.5% Fe
K711	Pommel	565-600AD	Gilded	5	94.7	3.1	0.5	0.0	0.9	0.8	95.5	1.5-3.5% Fe
K543	Sword ring		Plain	5	72.0	15.5	2.3	0.8	3.9	5.6	99.1	0.7-0.9 Fe
K787	Silver bracket		Plain	6	95.2	1.3	1.4	0.0	1.0	1.1	100.0	
K302	Sword pyramid		Gilding, gold panel	5	86.6	7.2	1.2	0.5	2.1	2.4	100.0	
K274	Misc		Plain	3	98.9	0.4	0.2	0.0	0.2	0.3	99.1	0.6-1.4% Fe
K959	Buckle		Gilding	5	96.7	2.4	0.0	0.0	0.2	0.7	100.0	
K176	c-tubing		Gilding	5	93.4	5.2	0.0	0.0	0.4	0.9	99.9	
K1132	c-tubing		Plain	5	95.8	1.8	0.0	0.0	0.6	1.8	100.0	

Table 5 cont. Sub-surface XRF data from the silver study.

Appendix 2. Extra graphs

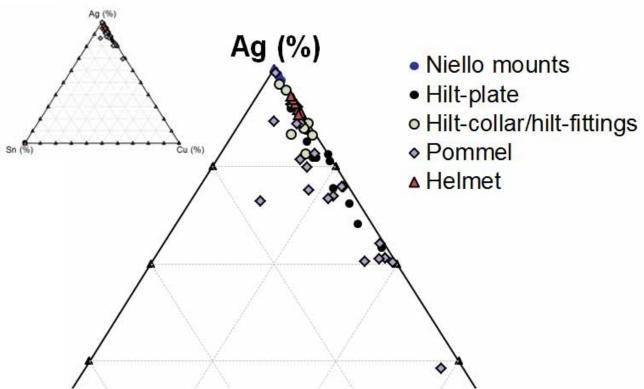


Figure 19. Ternary copper-lead-tin diagram of the die-impressed sheets showing two distinct groups.

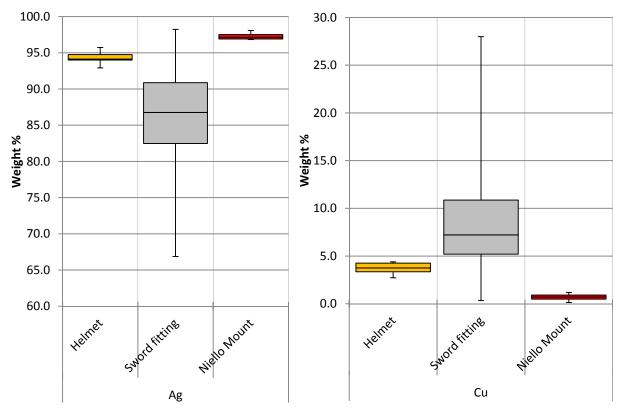


Figure 20. Box and whisker plots showing the significant differences between the silver alloy used for the helmet, niello mounts and the rest of the sword fittings.

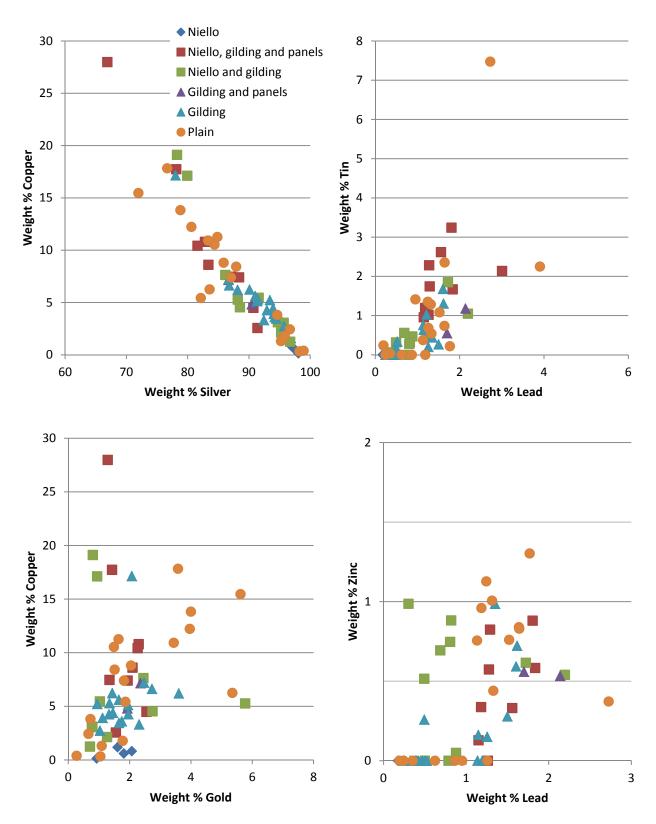


Figure 21. Copper/silver, tin/lead, copper/gold and zinc/lead bi-variate graphs showing the different types of applied decoration

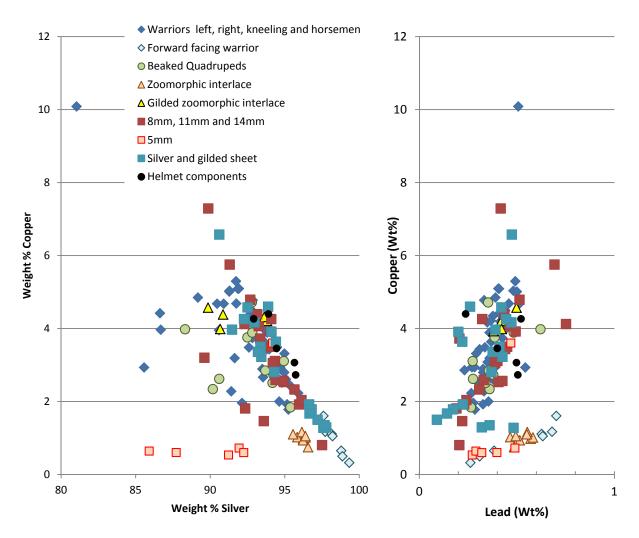


Figure 22. Copper/silver and copper/lead showing the different die-impressed sheets, the ribbed strips and other helmet components.



Staffordshire Hoard Research Reports

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Contextualising Metal-Detected Discoveries: Staffordshire Anglo-Saxon Hoard

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