Staffordshire Hoard Research Report 16

A Comparative Study of XRF and SEM-EDX Analysis of Gold / Silver / Copper Alloys at the Birmingham Museum Trust and the British Museum Laboratories

E. S. Blakelock

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The British Museum

DEPARTMENT OF CONSERVATION AND SCIENTIFIC RESEARCH

A comparative study of XRF and SEM-EDX analysis of gold/silver/copper alloys at the Birmingham Museum Trust and the British Museum laboratories

Science Report PR07444-9

Eleanor Blakelock

Abstract:

As part of a larger English Heritage-funded study of the Staffordshire Hoard, an intercomparison of the analytical data obtained using the X-ray fluorescence (XRF) instrument belonging to the Birmingham Museums Trust (BMT) and that obtained at the British Museum (BM) using either XRF or scanning electron microscopy-energy dispersive X-ray (SEM-EDX) analysis was undertaken to investigate whether the data were directly comparable.

A number of alloys of certified or accepted composition were analysed using all three instruments. The analyses indicated that there was generally good agreement between the results obtained using the BMT XRF and the BM XRF and SEM-EDX.

Although the data obtained from the analysis of Staffordshire Hoard objects using the BMT XRF instrument was not available for direct intercomparison, it is expected that any results on gold objects from the Hoard obtained using any of the three instruments discussed would be within acceptable margins for future qualitative surface or semi-quantitative analysis on areas prepared to reveal the sub-surface. As the objects are not flat even sub-surface data cannot be treated as fully quantitative.

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Introduction

As part of a larger English Heritage-funded study of the Staffordshire Hoard, an inter comparison of the analytical data obtained using the X-ray fluorescence (XRF) instrument belonging to the Birmingham Museums' Trust (BMT) and that obtained at the British Museum (BM) using either XRF or scanning electron microscopy-energy dispersive X-ray (SEM-EDX) analysis was undertaken.

A number of alloys of certified or accepted composition were analysed using all three instruments.

Methodology

A range of alloys of certified or accepted composition were analysed using each instrument (Tables 1 and 2). The British Museum 'SB' alloys were made in-house by Nigel Meeks to represent a range of archaeological gold alloys. These have been analysed using a number of analytical techniques.[1] The 'MAC' standards comprise of three commercially produced certified alloy standards of differing compositions made by Micro-Analysis Consultants Ltd, for the Staffordshire Hoard project. Three sets were produced, two for the Hoard owning institutions (Birmingham Museum Trust (BMT) and the Stoke Pottery Museum and Art Gallery (PMAG)), and one for the British Museum (BM). These alloys and standards are discussed in more detail in Appendix 3 of the report of the pilot study to investigate surface enrichment in gold objects from the Hoard.[2] (Blakelock 2013). All of the standards have previously been ground to a smooth finish to give an optimum surface for analysis and were analysed directly without further preparation.

British Museum XRF

The XRF instrument used at the British Museum was a Bruker Artax spectrometer fitted with a molybdenum X-ray tube operating at a voltage of 50 kV and a current of 500 µA. The data was collected for 200 seconds using a 0.65 mm diameter collimator. The XRF results were quantified by applying the *Staffordshire Hoard High Gold* standardisation method developed during the pilot study to investigate surface enrichment.[3] Energy to channel calibration on the XRF was performed by analysis of a pure silver standard prior to all analytical work.

The precision (reproducibility) of the BM XRF analyses is c. ±1–5% relative for elements present between 10–100%, c. ±5–20% for those present between 1–10%, and c. ±20–50% for those present between 0.1–1%. The detection limit for most elements is c. 0.1%, though is higher for tin (0.4%) due to the presence of overlapping peaks from other elements in the spectra.

Three different areas were analysed for each of the 'MAC' standards from the BMT and BM sets, and two extra analyses were carried out on different days on the 'MAC' standards

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1 Cowell 1977, 1998; Oddy and Blackshaw 1974.

2 Blakelock 2013.

3 Blakelock 2013. When quantifying the data from a spectrum, the BM in-house software uses a maximum of four accepted or certified alloy compositions against which to standardise, producing compositional data in weight percentage. As with all analytical methods it is important that the compositional range of the standards covers that of the samples of interest to minimise the potential error due to extrapolation. Therefore, standards with similar alloy compositions were used to create a method (Cowell 1977). The *Staffordshire Hoard High Gold* standardisation method used MAC1, MAC2, SB11 and SB8A.

owned by the BM. Four areas were analysed on each of the ‘SB’ alloys on different days. The only exception was SB8A where nine areas were analysed over several days.

British Museum SEM-EDX

The SEM used was a Hitachi S-3700N Variable Pressure SEM set at an acceleration voltage of 20 kV and a live time of 150 seconds was used. The EDX compositional data were obtained using an Oxford Instrument INCA EDX microanalysis system with an INCAx-act Silicon Drift Detector (SDD). The data was quantified by standardisation with the BM MAC2 standard. The SEM-EDX was calibrated by analysis of pure cobalt prior to all analytical work.

The detection limits for each element are variable but are typically 0.1-0.3%. Standard errors are typically within 1% for major elements and 5-20% relative for minor elements.

With the exception of the MAC1 and MAC3 standards owned by BMT that were analysed four times, all the standards were analysed at least ten times. Areas approximately 1.5 by 1 mm were analysed to compensate for any potential heterogeneity of the alloys.

Birmingham Museum Trust XRF

The BMT XRF instrument is a Bruker M1 Mistral fitted with a tungsten X-ray tube with a silicon drift detector. The voltage used was 40 kV with a current of 800 µA. The data for the ‘MAC’ standards and the ‘SB’ alloys was collected for 30 seconds with a 0.7 mm collimator. Quantification of the data was carried out using pure elements, and the energy to channel calibration on the instrument was carried out by analysing a pure silver standard prior to use.

At least three different areas were analysed on each of the BM ‘SB’ alloys of known composition. Each of the BM set of ‘MAC’ standards were analysed on the front and back, and across the surface to allow for heterogeneity of the alloy. Nine areas on the fronts of each of the BMT set of ‘MAC’ standards were analysed.

Results and Discussion

The combined results obtained using the three instruments are shown in Tables 1 and 2, and the individual analytical results available in Tables 3-7. All the data from the three instruments were normalised to 100%. The results reveal generally good agreement between the different analytical methods, and with the certified or accepted compositions of the alloys analysed.

The high standard deviations noted for the analyses of some of the standards suggest alloy heterogeneity, particularly for SB17 and SB8A when examined with the BMT XRF, and SB18 when examined by the two XRF instruments. Silver was the element which demonstrated the greatest variability, but this was within acceptable limits of the analytical equipment and software. The SEM-EDX and XRF results for SB17, SB18 and SB39 could have been improved if standardisation was carried out using an alloy with higher silver and copper content; even so the results are within the limits of analytical errors especially for qualitative surface analysis.

Comparison between the BM and BMT ‘MAC’ standards confirmed good agreement between them (Table 2).

When analysing the surface of objects from the Staffordshire Hoard, the XRF or SEM-EDX results will be affected by any gold enrichment present and therefore any of these results should be quoted as qualitative.[4] As the objects are not flat surface preparation to allow analysis of the sub-surface alloy composition is at best only likely to yield semi-quantitative alloy compositions using XRF and SEM-EDX. Therefore, if the instrument settings reported here are used during the surface or sub-surface analysis of objects from the Staffordshire Hoard, the differences in the results obtained using the different instruments may be considered to be negligible.

Conclusion

The comparative analyses indicated that there was generally good agreement between the results obtained using the BMT XRF and the BM XRF and SEM-EDX. Although data obtained from the analysis of Staffordshire Hoard objects using the BMT XRF was not available for direct intercomparison, it is expected that any results on gold objects from the Hoard obtained using any of the three instruments discussed would be within acceptable margins for future qualitative surface or semi-quantitative analysis on areas prepared to reveal the sub-surface. As the objects are neither flat nor homogenous, even the sub-surface data cannot be treated as fully quantitative.

Author: Eleanor Blakelock Counter signed: Duncan Hook

21st August, 2014

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[4] Blakelock 2013.

					BM								BMT			
					Artax XRF				SEM-EDX				Mistral M1 XRF			
					Quantified using “SH High Gold” Method				Standardised using MAC2				Quantified against pure elements			
	Wt% Au	Wt% Ag	Wt% Cu		No.	Wt% Au	Wt% Ag	Wt% Cu	No.	Wt% Au	Wt% Ag	Wt% Cu	No.	Wt% Au	Wt% Ag	Wt% Cu
SB8A	70.98	24.07	4.95	Mean	9	71.1	23.8	5.1	10	70.4	24.5	5.1	7	71.1	24.3	4.6
				StDev		*0.22*	*0.22*	*0.05*		*0.15*	*0.15*	*0.07*		*0.47*	*0.82*	*0.41*
SB11	85.36	9.64	5	Mean	4	85.9	9.2	4.9	10	85.2	9.8	5.0	6	85.8	9.8	4.4
				StDev		*0.26*	*0.25*	*0.04*		*0.13*	*0.11*	*0.07*		*0.10*	*0.32*	*0.26*
SB12	90.02	4.98	5	Mean	4	89.9	5.2	4.9	13	89.4	5.4	5.2	4	89.9	5.1	5.0
				StDev		*0.16*	*0.13*	*0.10*		*0.10*	*0.10*	*0.06*		*0.17*	*0.09*	*0.11*
SB17	65.91	28.22	5.87	Mean	4	66.4	27.6	6.0	15	64.8	29.2	6.0	4	66.5	27.9	5.6
				StDev		*0.13*	*0.19*	*0.06*		*0.23*	*0.21*	*0.07*		*0.34*	*0.68*	*0.48*
SB18	63.4	27.2	9.4	Mean	4	63.8	26.4	9.8	10	63.4	27.3	9.3	4	64.2	26.5	9.3
				StDev		*0.21*	*0.41*	*0.20*		*0.13*	*0.10*	*0.08*		*0.29*	*0.30*	*0.11*
SB39	50	30	20	Mean	4	50.2	28.6	21.2	10	49.0	31.0	20.0	4	50.2	29.6	20.2
				StDev		*0.13*	*0.11*	*0.11*		*0.11*	*0.08*	*0.10*		*0.32*	*0.21*	*0.14*

Table 1. ‘SB’ alloys and their accepted compositions compared to the results obtained using the BMT XRF and the BM XRF and SEM-EDX. No. indicates the number of analyses carried out.

							BM										BMT				
							Artax XRF					SEM-EDX					Mistral M1 XRF				
							Quantified using SH High Gold Method					Standardised using MAC2					Quantified against pure elements				
		Wt% Au	Wt% Ag	Wt% Cu	Wt% Sn		No.	Wt% Au	Wt% Ag	Wt% Cu	Wt% Sn	No.	Wt% Au	Wt% Ag	Wt% Cu	Wt% Sn	No.	Wt% Au	Wt% Ag	Wt% Cu	Wt% Sn
BM Set	**MAC1**	93.9	4.6	1.04	0.54	Mean	5	94.0	4.4	1.0	0.6	11	94.0	4.4	1.1	0.5	12	94.3	4.2	1.1	0.4
						StDev		*0.30*	*0.12*	*0.01*	*0.20*		*0.13*	*0.07*	*0.06*	*0.06*		*0.23*	*0.20*	*0.04*	*0.09*
	MAC2	74.7	19.2	5.1	1.03	Mean	5	75.2	18.6	5.1	1.1	11	74.7	19.2	5.1	1.0	11	75.6	18.5	4.7	1.2
						StDev		*0.21*	*0.18*	*0.02*	*0.21*		*0.17*	*0.10*	*0.08*	*0.08*		*0.43*	*0.37*	*0.12*	*0.11*
	MAC3	59.4	29.8	9.1	1.98	Mean	5	59.4	29.6	9.1	1.9	11	59.3	29.7	9.0	2.0	12	59.3	29.5	8.9	2.3
						StDev		*0.16*	*0.15*	*0.14*	*0.15*		*0.21*	*0.27*	*0.11*	*0.11*		*0.44*	*0.30*	*0.15*	*0.30*
BMT Set	**MAC1**	93.9	4.6	1.04	0.54	Mean	3	94.1	4.4	1.0	0.5	4	93.7	4.5	1.4	0.4	9	94.1	4.3	1.1	0.5
						StDev		*0.10*	*0.06*	*0.02*	*0.04*		*0.19*	*0.15*	*0.04*	*0.08*		*0.08*	*0.06*	*0.04*	*0.07*
	MAC2	74.7	19.2	5.1	1.03	Mean	3	75.0	18.9	5.0	1.1	13	74.3	19.6	5.1	1.0	9	75.3	18.7	4.7	1.3
						StDev		*0.03*	*0.07*	*0.10*	*0.05*		*0.15*	*0.15*	*0.06*	*0.09*		*0.12*	*0.20*	*0.10*	*0.12*
	MAC3	59.4	29.8	9.1	1.98	Mean	3	59.3	29.2	9.5	2.0	4	58.4	29.7	9.8	2.1	9	59.0	29.3	9.2	2.5
						StDev		*0.15*	*0.19*	*0.06*	*0.19*		*0.12*	*0.42*	*0.29*	*0.23*		*0.09*	*0.30*	*0.21*	*0.10*

Table 2. ‘MAC’ standards and their certified compositions compared to the results obtained using the BMT XRF and the BM XRF and SEM-EDX. No. indicates the number of analyses carried out.

Standard	Laboratory	Instrument	Wt% Au	Wt% Ag	Wt% Cu
SB8A	BM	XRF	71.0	23.9	5.1
			70.9	24.0	5.1
			71.3	23.7	5.0
			71.3	23.6	5.1
			71.2	23.7	5.1
			70.8	24.1	5.1
			71.3	23.6	5.1
			71.2	23.7	5.1
			71.0	24.0	5.0
		SEM-EDX	70.6	24.3	5.1
			70.5	24.4	5.1
			70.4	24.4	5.2
			70.5	24.5	5.0
			70.1	24.8	5.1
			70.4	24.5	5.1
			70.4	24.6	5.0
			70.2	24.7	5.1
			70.2	24.6	5.2
			70.5	24.5	5.0
	BMT	XRF	71.5	23.8	4.7
			70.8	25.1	4.1
			70.1	25.9	4.0
			71.4	23.9	4.7
			71.2	23.9	4.9
			71.5	23.7	4.8
SB11	BM	XRF	85.7	9.3	5.0
			85.6	9.5	4.9
			86.2	8.9	4.9
			85.8	9.3	4.9
		SEM-EDX	85.0	9.8	5.2
			85.1	9.9	5.0
			85.2	9.8	5.0
			85.3	9.8	4.9
			85.1	9.9	5.0
			85.4	9.6	5.0
			85.3	9.7	5.0
			85.0	9.9	5.1
			85.1	9.9	5.0
			85.2	9.7	5.1
	BMT	XRF	85.7	10.0	4.3
			85.9	9.8	4.3
			85.9	9.8	4.3
			85.7	10.0	4.3
			85.6	10.1	4.3
SB12	BM	XRF	90.0	5.2	4.8
			90.0	5.0	5.0
			89.7	5.3	5.0
			90.1	5.1	4.8
		SEM-EDX	89.5	5.3	5.2
			89.5	5.4	5.1
			89.3	5.5	5.2
			89.3	5.4	5.3
			89.7	5.1	5.2
			89.4	5.4	5.2
			89.3	5.5	5.2
			89.5	5.3	5.2
			89.4	5.4	5.2
			89.4	5.3	5.3
			89.4	5.5	5.1
			89.5	5.4	5.1
			89.4	5.5	5.1
	BMT	XRF	89.8	5.1	5.1
			90.1	4.9	5.0
			90.1	5.1	4.8

Table 3. Normalised raw data for SB8A, SB11 and SB12.

Standard	Laboratory	Instrument	Wt% Au	Wt% Ag	Wt% Cu
SB17	BM	XRF	66.3	27.7	6.0
			66.4	27.6	6.0
			66.6	27.3	6.1
			66.3	27.7	6.0
		SEM-EDX	65.1	28.9	6.0
			65.0	28.9	6.1
			65.2	28.9	5.9
			64.5	29.4	6.1
			64.7	29.3	6.0
			64.6	29.4	6.0
			64.4	29.6	6.0
			64.7	29.2	6.1
			64.9	29.1	6.0
			65.0	29.1	5.9
			65.0	29.0	6.0
			64.7	29.3	6.0
			64.7	29.2	6.1
			64.7	29.4	5.9
			64.7	29.2	6.1
	BMT	XRF	66.3	28.8	4.9
			66.6	27.4	6.0
			67.0	27.3	5.7
SB18	BM	XRF	64.0	26.1	9.9
			63.6	26.9	9.5
			63.8	26.4	9.8
			64.0	26.1	9.9
		SEM-EDX	63.2	27.4	9.4
			63.4	27.4	9.2
			63.6	27.1	9.3
			63.4	27.3	9.3
			63.3	27.3	9.4
			63.3	27.3	9.4
			63.6	27.2	9.2
			63.6	27.1	9.3
			63.4	27.3	9.3
			63.4	27.3	9.3
	BMT	XRF	64.5	26.2	9.3
			64.3	26.3	9.4
			64.0	26.8	9.2
SB39	BM	XRF	50.1	28.7	21.2
			50.2	28.5	21.3
			50.3	28.6	21.1
			50.3	28.5	21.2
		SEM-EDX	48.9	31.0	20.2
			49.0	31.1	19.9
			49.0	31.1	19.9
			49.1	30.9	20.0
			49.1	31.0	20.0
			48.8	31.1	20.1
			49.0	31.0	20.0
			49.1	31.0	19.9
			48.9	31.0	20.1
			48.9	31.0	20.1
	BMT	XRF	50.1	29.7	20.2
			49.9	29.7	20.4
			50.6	29.3	20.1

Table 4. Normalised raw data for SB17, SB18 and SB39.

Standard	Laboratory	Instrument	Wt% Au	Wt% Ag	Wt% Cu	Wt% Sn
BM MAC1	BM	XRF	94.6	4.4	1.0	0.8
			94.5	4.5	1.0	0.7
			94.6	4.4	1.0	0.7
			94.8	4.2	1.0	0.4
			94.7	4.3	1.0	0.3
		SEM-EDX	94.5	4.4	1.1	0.4
			94.5	4.4	1.1	0.4
			94.4	4.5	1.1	0.5
			94.5	4.4	1.1	0.5
			94.6	4.3	1.1	0.5
			94.3	4.5	1.2	0.5
			94.6	4.3	1.1	0.6
			94.5	4.4	1.1	0.4
			94.7	4.3	1.0	0.5
			94.6	4.4	1.0	0.4
			94.4	4.4	1.2	0.4
	BMT	XRF	94.5	4.4	1.1	0.4
			94.9	4.0	1.1	0.4
			94.7	4.2	1.1	0.6
			94.7	4.2	1.1	0.4
			95.1	3.9	1.0	0.4
			95.0	3.8	1.2	0.3
			94.7	4.2	1.1	0.4
			94.5	4.4	1.1	0.5
			94.6	4.3	1.1	0.4
			94.5	4.4	1.1	0.6
			94.9	4.1	1.0	0.6
			94.6	4.3	1.1	0.5
BM MAC2	BM	XRF	76.1	18.7	5.2	1.3
			75.8	19.0	5.2	1.2
			75.9	18.9	5.2	1.2
			76.0	18.8	5.1	0.8
			76.3	18.5	5.2	1.2
		SEM-EDX	75.6	19.3	5.2	1.0
			75.6	19.4	5.0	0.9
			75.3	19.6	5.2	1.1
			75.4	19.5	5.2	1.0
			75.3	19.5	5.3	1.1
			75.3	19.4	5.4	1.1
			75.6	19.4	5.0	0.9
			75.5	19.4	5.1	0.9
			75.4	19.5	5.1	0.9
			75.3	19.6	5.1	1.1
			75.5	19.4	5.1	0.9
	BMT	XRF	76.2	19.0	4.8	1.2
			76.1	19.0	4.9	1.2
			76.4	18.8	4.8	1.2
			76.6	18.4	5.0	1.3
			77.5	17.8	4.7	0.9
			76.2	19.0	4.8	1.3
			76.4	18.8	4.8	1.1
			76.7	18.6	4.7	1.2
			76.4	18.9	4.7	1.1
			76.5	18.5	5.0	1.3
			76.5	18.5	5.0	1.3

Table 5. Normalised raw data for the MAC1 and MAC2 standards owned by the BM.

Standard	Laboratory	Instrument	Wt% Au	Wt% Ag	Wt% Cu	Wt% Sn
BM MAC3	BM	XRF	60.4	30.2	9.4	1.9
			60.6	30.2	9.2	1.7
			60.5	30.0	9.5	2.1
			60.5	30.3	9.2	1.9
			60.6	30.0	9.4	1.9
		SEM-EDX	60.3	30.4	9.3	2.1
			60.3	30.4	9.3	2.0
			60.4	30.2	9.4	1.9
			60.9	30.0	9.1	2.1
			60.2	30.5	9.3	2.0
			60.6	30.1	9.3	2.1
			60.3	30.4	9.3	2.2
			60.8	30.1	9.2	2.0
			60.3	30.5	9.2	2.0
			60.3	30.4	9.3	2.0
			60.1	31.0	9.0	1.8
	BMT	XRF	60.7	30.4	8.9	2.5
			60.5	30.5	9.0	2.5
			60.6	30.4	9.0	2.1
			60.9	29.8	9.2	2.8
			60.6	30.1	9.3	2.0
			60.8	30.1	9.1	2.6
			60.6	30.5	8.9	2.2
			60.6	29.9	9.4	2.6
			60.5	30.2	9.3	2.7
			61.6	29.4	9.0	1.6
			60.5	30.3	9.2	2.5
			60.4	30.5	9.1	2.7

Table 6. Normalised raw data for the MAC3 standard owned by the BM.

Standard	Laboratory	Instrument	Wt% Au	Wt% Ag	Wt% Cu	Wt% Sn
BMT MAC1	BM	XRF	94.6	4.4	1.0	0.5
			94.7	4.3	1.0	0.5
			94.6	4.4	1.0	0.6
		SEM-EDX	94.3	4.3	1.4	0.4
			94.1	4.6	1.3	0.3
			94.1	4.5	1.4	0.5
			93.9	4.7	1.4	0.4
	BMT	XRF	94.6	4.3	1.1	0.6
			94.7	4.3	1.0	0.5
			94.5	4.4	1.1	0.4
			94.6	4.3	1.1	0.5
			94.7	4.2	1.1	0.6
			94.6	4.3	1.1	0.6
			94.6	4.3	1.1	0.5
			94.7	4.2	1.1	0.5
			94.6	4.4	1.0	0.4
BMT MAC2	BM	XRF	75.9	19.2	4.9	1.1
			75.9	19.0	5.1	1.1
			75.9	19.0	5.2	1.0
		SEM-EDX	75.4	19.5	5.2	1.0
			74.8	19.9	5.3	1.0
			75.1	19.8	5.1	0.9
			74.9	19.8	5.3	1.1
			74.8	20.1	5.1	1.0
			75.0	19.8	5.2	0.9
			75.1	19.7	5.2	1.1
			75.1	19.8	5.2	1.0
			74.8	20.0	5.2	1.0
			74.9	19.9	5.2	1.0
			75.1	19.7	5.3	1.0
			74.9	19.9	5.2	0.9
			74.9	19.8	5.2	0.8
	BMT	XRF	76.2	19.1	4.7	1.2
			76.2	19.0	4.8	1.5
			76.3	19.0	4.7	1.3
			76.2	19.1	4.7	1.2
			76.7	18.6	4.8	1.4
			76.3	18.8	4.9	1.4
			76.3	18.7	5.0	1.3
			76.2	18.9	4.9	1.2
			76.2	18.9	4.9	1.2
BMT MAC3	BM	XRF	60.3	30.0	9.7	1.9
			60.6	29.8	9.7	1.9
			60.5	29.7	9.8	2.2
		SEM-EDX	59.7	30.5	9.8	1.9
			59.6	30.5	9.9	2.0
			59.7	29.8	10.5	2.5
			59.6	30.4	10.0	2.1
	BMT	XRF	60.4	30.3	9.3	2.5
			60.7	29.9	9.3	2.6
			60.6	30.1	9.3	2.6
			60.7	29.6	9.8	2.7
			60.6	29.8	9.6	2.6
			60.8	29.6	9.7	2.7
			60.6	30.2	9.2	2.4
			60.6	30.2	9.2	2.5
			60.7	30.2	9.1	2.4

Table 7. Normalised raw data for the MAC1, MAC2 and MAC3 standards owned by the BMT.

Staffordshire Hoard Research Reports

Staffordshire Hoard Research Reports were produced by the project

Contextualising Metal-Detected Discoveries: Staffordshire Anglo-Saxon Hoard

Historic England Project 5892

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