

LEGGE'S MOUNT,
THE TOWER OF LONDON, LONDON
SCIENTIFIC EXAMINATION OF THE CUPELS

TECHNOLOGY REPORT

Harriet White



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SUMMARY

A small but important collection of used and unused cupels were excavated from the surrounds of a 16th-century furnace situated at Legge's Mount, the north-west bastion of the Tower of London. The cupels were examined by XRF and SEM-EDS to shed light on the raw materials used in their manufacture and the reasons for their use. The investigations revealed the cupels were fabricated from pure bone ash and faced with a layer of finely ground bone ash, reflecting the descriptions of cupel manufacture by 16th-century authors. It was further shown that they were used for refining silver in which the main contaminant was copper.

ACKNOWLEDGEMENTS

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ARCHIVE LOCATION

Mounted samples are archived at Fort Cumberland, Fort Cumberland Road, Eastney, Portsmouth, PO4 9LD

DATE OF RESEARCH

2007-2010

CONTACT DETAILS

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INTRODUCTION

During 1976 archaeological excavations were carried out at Legge's Mount, the north-west bastion of the Tower of London. The excavations revealed a furnace structure with two key-shaped hearths and an adjoining ash pit. Archaeomagnetic dating of the furnace remains indicate it received its last heating between the years 1530 and 1560 (Parnell 1993). A large number of industrial vessels and metalworking debris were recovered from in and around the ash pit. The vessels included crucibles, long-necked globular flasks identified as nitric acid distillation flasks, and fragments of ceramic dishes. A small but important collection of cupels (the object of this report) were also recovered from the furnace surrounds. The variety of vessels present suggests that a range of metallurgical processes were carried out at the site, and it is likely these were associated with the Royal Mint. It is known that the Mint was situated close to Legge's Mount until 1560 when it was moved to the Salt Tower area of the Tower (Barter 1978). This report presents the results of scientific investigations of the Legge's Mount cupels, which were undertaken to determine methods of cupel manufacture and cupel use.

BACKGROUND: CUPELS AND CUPELLATION

Cupellation is the metallurgical process by which noble metals are refined. The impure noble metal (gold or silver) is heated with lead in an oxidising atmosphere so that the lead is oxidized to litharge (PbO). The litharge reacts with and oxidizes the base metals mixed with the gold or silver. It also forms fusible compounds with them which separate from the melt. The noble metals, which do not react with oxygen or litharge, remain as a discrete button or *regulus* when the base metals have been oxidised and absorbed by the cupel (Bayley 1991, 125). Cupellation is carried out for a number of purposes. These include ore assaying to test the quantity of metal carried in an ore and so can be associated with mining, metal assaying to determine alloy compositions or to check the purity of metals, for example in a mint or alchemical laboratory, or for recycling to recover noble metals from scrap metal. Gold or silver were routinely alloyed with copper, for example in the manufacture of jewellery or coinage (Campbell 1991; Bayley and Eckstein 1997; Martín-Torres *et al*/2008).

Small scale cupellation is carried out in porous ash-based vessels known as cupels. A number of 16th-century treatises describe the cupellation process and manufacture of cupels. These texts include Lazarus Ercker's *Treatise on Ores and Assaying* (Sisco and Smith 1951), Agricola's *De Re Metallica* (Hoover and Hoover 1950) and Biringuccio's *Pirotechnia* (Smith and Gnudi 1959)). According to the authors cupels were manufactured using ground and washed ash that is moistened with a binder such as water, beer or water mixed with egg white and beaten into a mould with a wooden stamp to produce a shallow cavity on the upper surface (Figure 1). The cavity is then faced with finely ground bone ash. Though a variety of ashes (plant, bone and horn), and other raw materials such as clay were used, bone ash was thought to be the most effective material for cupel manufacture. Bone ash is an inert material which does not react with the oxidised metals, but absorbs them by capillary action and provides efficient separation (Bayley 1991; Martínón-Torres *et al*/2003; Martínón-Torres *et al*/2008).

A total of ten cupels and four cupel fragments were recovered during the excavations. The most complete examples have a diameter of between 30 and 32mm and a height of 10 to 12mm (Figure 2 and Appendix 1). Four are white and friable (samples 399 to 403) and the remainder are dark grey to black and are consolidated (samples 403 to 412).

Exploratory X-ray fluorescence (XRF) analysis of each of the cupels' surfaces showed the grey examples were rich in lead while the white examples contained only trace amounts. The trace amounts of lead in conjunction with their white colour and friable character indicate that samples 399 to 402 were unused (Appendix 2). Discoveries of unused cupels in the archaeological record are extremely rare. The only other example reported to date is a single cupel recovered in Oberstockstall, Austria (Martínón-Torres *et al*/2008). Their poor survival can be explained by the method of manufacture; dampened ash is tamped into moulds and allowed to dry (see above), meaning they are too fragile to be preserved archaeologically. Used cupels are preserved since the litharge absorbed into the body acts as a cement binding the raw ingredients together. As it is used cupels that are most commonly recovered archaeologically, the approach taken to investigate original raw material composition has been to subtract the lead and other metal oxide

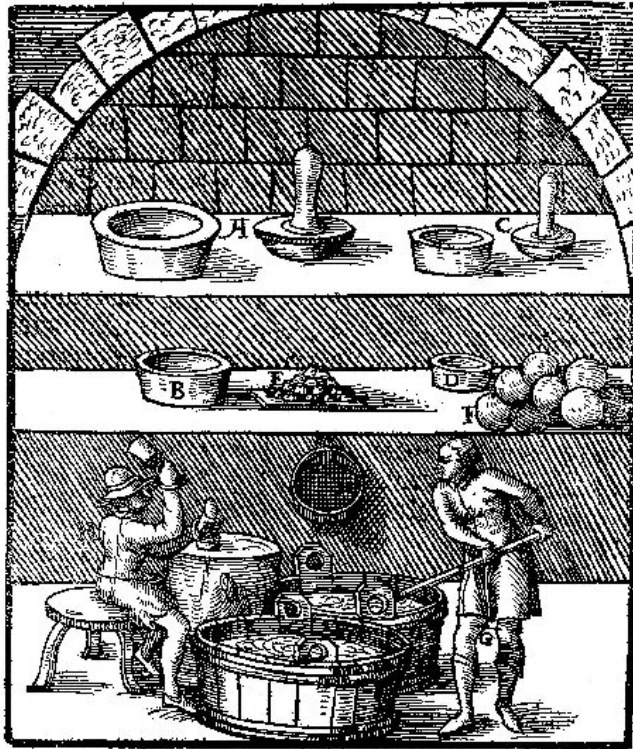


Figure 1. Assayers washing ashes and moulding them into cupels as depicted in Lazarus Ercker's Treatise on Ores and Assaying (1580, Plate 5). The picture also illustrates cupel moulds and stamps, balls of prepared bone ash and cupels made in the moulds.



Figure 2. Collection of cupels recovered from Legge's Mount.

concentrations from the compositional results and normalize the remaining element oxides to 100% (for example Rehren and Eckstein 2002; Martín-Torres *et al* 2008). The presence of used and unused cupels at Legge's Mount provides an opportunity to examine cupel manufacture and compare the results with methods detailed by 16th-century authors and to investigate the purpose of cupellation carried out at the site. It also provides an opportunity to test the methodology applied by others for estimating original cupel compositions.

METHOD

Cross-sections of two cupel samples (Table 1) were mounted in epoxy resin, polished down to 1µm particle size and carbon coated for scanning electron microscopy (SEM). The analysis was undertaken using a Zeiss-Stereoscan 440 SEM with a tungsten filament, and attached Oxford Instruments germanium energy dispersive (ED) X-ray detector, and ISIS software. The germanium detector allows for the simultaneous detection of all elements from boron to uranium providing they are present in concentrations above the detection limits of the system. The operating conditions of the SEM were 25kV accelerating potential with a probe current of 1nA, a process time of 5 and each spectrum was collected for 100s livetime. The spectra were calibrated using a cobalt standard, and deconvoluted with the phi-rho-z correction procedure using the Oxford Instruments SEMQuant software. This utilizes element profiles derived from single element or simple compound standards (eg pure iron, jadite, wollastonite etc.). Since ED X-ray spectrometry provides no direct information on the valence states of the elements present in the analysed material appropriate valence states were selected and the oxide weight percents were calculated stoichiometrically. The minimum detection levels for each element, determined by previous analyses, are presented in Appendix 3. Spectra were collected from areas approximately 1 x 1.5mm in size in points from the top centre of the cupels to the cupel bases. Analytical totals were typically in the range of 98.5 to 99.5% wt% and were normalised to 100 wt%.

Table 1. Cupels sampled for analysis

Sample Number	Colour	Diameter (mm)	Height (mm)
402	White	31.5	12.4
412	Dark grey to black	30.0	9.8

Four experimental cupels manufactured by Nicholas Thomas (University of Paris I) using mixtures of bone ash, vegetable ash and clay were also analysed using SEM-EDS to provide comparative compositional data.

RESULTS

The body of cupel 402 is calcium phosphate, with only trace amounts of other element oxides present (Table 2), and the composition was shown to be constant through the profile. It is comparable to the experimental cupel manufactured from 100% bone ash. The experimental cupels manufactured from vegetable ash or mixtures of bone ash and vegetable ash, and bone ash and clay contain significant concentrations of other element oxides, most notably alumina, magnesia, silica and potassium oxide (Table 2).

Cupel 402 is made from angular to sub-angular fragments of bone ash up to 400µm in size (Figure 3). The upper surface of the cupel has a compact layer of finely ground bone ash approximately 150µm thick. The upper and lower surfaces of the cupel both show some lead oxide enrichment, though it is more pronounced in the lower surface where it extends about 250µm into the body (Table 2 and Figure 3 (right)). The lead oxide content is much less than that observed for cupel 412 and it appears to have been absorbed by the bone ash fragments themselves rather than being inter-granular (compare Figures 3 and 5) and so is most likely a result of post-burial contamination.

Cupel 412 contains significant quantities of lead oxide and minor amounts of silver and copper oxides (<2%) in addition to calcium phosphate (Figure 4 and Appendix IV). Concentrations of silver and copper oxides are greatest at the cupel surface, while the highest concentration of PbO occurs towards the middle of the cupel (Figure 5). In contrast to cupel 402 the lead oxide surrounds the grains rather than being absorbed by

Table 2. The composition of cupel 402 compared with four modern examples made from a variety of materials.

Sample	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₂	K ₂ O	CaO	PbO
Mean*	<0.5	0.4	<0.3	0.3	40.3	0.2	0.0	58.0	0.4
Sdev	0.1	0.1	0.1	0.1	0.4	0.1		0.5	0.4
Surface	<0.5	0.3	<0.3	<0.3	38.9	0.8	0.0	55.2	4.1
Base	<0.5	<0.3	<0.3	<0.3	34.0	<0.2	0.0	45.8	19.5
100% Bone ash	<0.5	1.0	0.4	1.3	41.3	0.1	0.0	55.5	0.0
100% Vegetable ash	<0.5	1.5	4.7	30.9	1.6	0.2	6.4	51.0	0.0
50% bone ash / 50% vegetable ash	<0.5	1.8	4.1	17.3	20.8	0.3	3.3	47.8	nd
75% bone ash / 25% dry clay	<0.5	1.0	3.7	8.3	36.6	0.3	0.2	49.5	nd

* Eight areas were analysed, starting just below the top centre and finishing just above the bottom centre. The composition was consistent throughout.

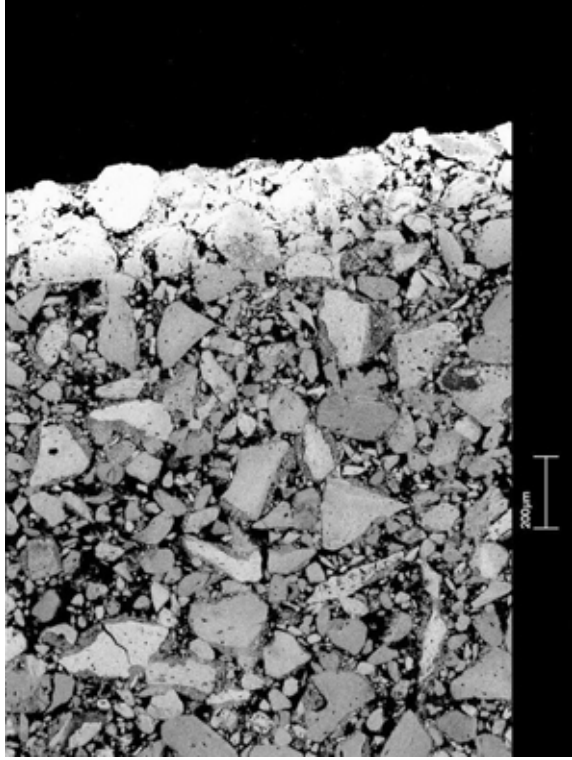
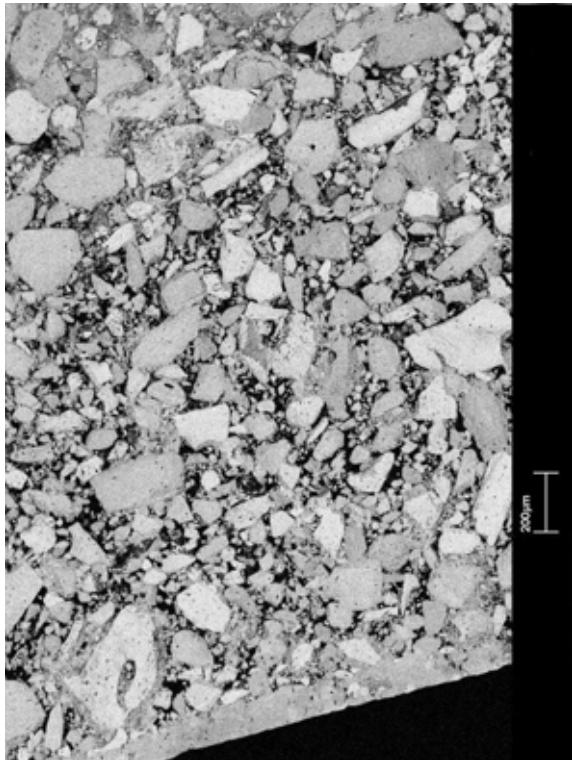


Figure 3. SEM micrographs (BSE) of cupel 402 showing angular fragments of bone ash (max 400µm). The upper surface (left) shows a compact layer of finely ground bone ash, while the cupel base (right) is brighter showing lead has been absorbed by the bone ash fragments.

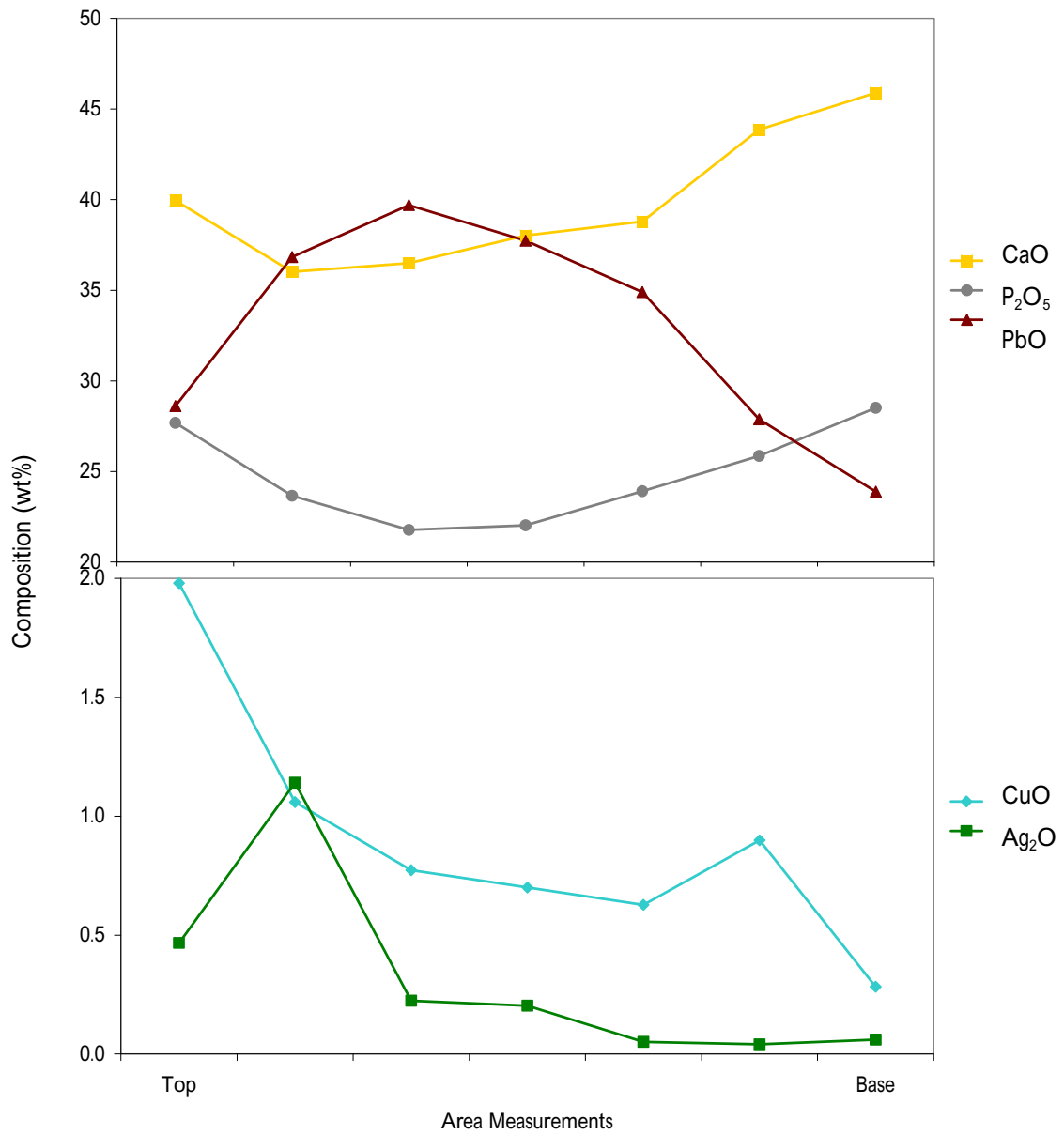


Figure 4. Compositional profile through cupel 412. Left of graph is cupel surface, right of graph is cupel base.

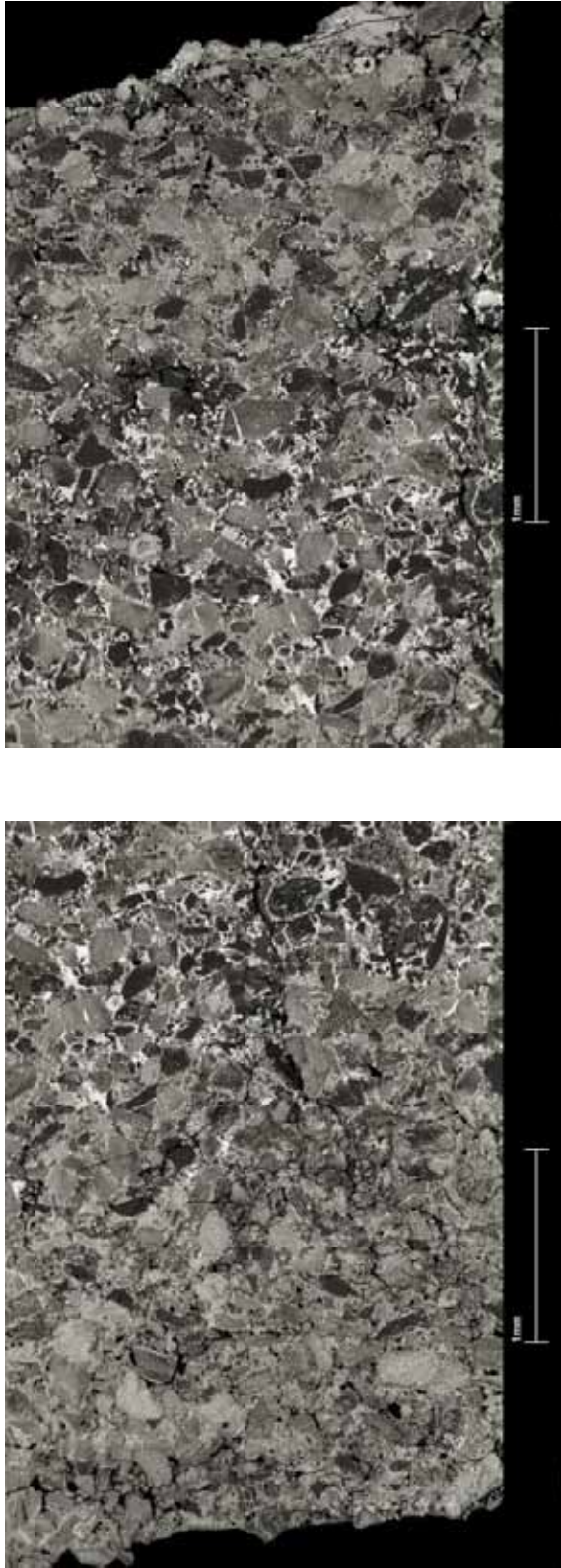


Figure 5. SEM micrographs (BSE) of cupel 412. Left image is upper surface of cupel and right image is cupel base. The lead oxide is concentrated in the central area of the cupel and surrounds the bone ash grains.

Table 3 Comparing the re-normalised composition of cupel 412 with the composition of the unused cupel 402.

	Sample	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₂	CaO
402	Mean*	<0.5	0.4	<0.3	0.3	40.3	0.2	58.0
	Sdev	0.1	0.1	0.1	0.1	0.4	0.1	0.5
412	Mean**	<0.5	0.3	0.4	0.5	37.5	0.5	60.4
	Sdev	0.1	0.2	0.1	0.2	1.6	0.3	1.4

* Eight areas were analysed, starting just below the top centre and finishing just above the bottom centre. The composition was consistent throughout.

**Seven areas were analysed starting just below the top centre and finishing just above the bottom centre. The metal oxides were removed from the analytical totals and the remaining element oxides re-normalised to 100% for each area. The results were consistent throughout the cupel body and so the mean of the seven points analysed is given.

them. The fine facing observed on upper surface of cupel 402 was also preserved in places on cupel 412.

The original composition of the cupel matrix is indicated by the subtraction of the metal oxides from the cupel composition and re-normalising the remaining elements oxides to 100% (Table 3). Like cupel 402, the main component is calcium phosphate (98%) with only trace amounts (<2%) of alumina, silica and magnesia present, and this composition is consistent throughout the cupel body. This shows cupel 412 was also manufactured from pure bone ash.

DISCUSSION

The unused cupel and re-normalised cupel compositions demonstrate that these cupels from Legge's Mount were fabricated from pure bone ash without the addition of any other "excipient" or bulking material such as vegetable or wood ash or clay. As recommended by contemporary authors, for example Ercker (Sisco and Smith 1951) and Biringuccio (Smith and Gnudi 1959), the cupels were finished with a fine bone ash facing. The bone ash facing helps the *regulus* separate more cleanly from the cupel.

The heavy metal oxides present in the used cupel (lead oxide, copper oxide and silver) indicate the cupels were used for processing silver in which the main contaminant was copper. It should be noted that the silver concentrations present (up to 1%) are high in comparison to other reported concentrations of silver loss in medieval and post-medieval cupellation (see for example Martín-Torres *et al*/2009). Bayley and Eckstein (2006) and Martín-Torres *et al* (2009) suggest an insufficient Pb:Cu ratio may account for silver loss, when some free cuprous oxide (Cu_2O) may form if not enough lead is present in the melt. Cuprous oxide can dissolve up to 44% silver and so carry it into the cupel body. This explanation is not likely to be the case in the instance of cupel 412 where, apart from the uppermost area scanned, the Pb:Cu ratio is above the recommended ratio of 16 (Appendix IV), (see Martín-Torres *et al* (2008) for a discussion of this). A more likely explanation for the high silver content is the presence of cracks on the cupel surface which, if formed during the drying stage of cupel manufacture, would allow some molten silver to seep into the body during the cupellation process.

The presence of both used and unused cupels at Legge's Mount provided the opportunity to test the standard method of determining original raw material composition of used cupels applied by other authors. To recap, the very fragile nature of unused cupels mean they rarely survive archaeologically, and it is normally used cupels that are available for study. The assumption is made that if the contaminants associated with cupel use (lead, copper and so on) are subtracted from the total composition and the remaining element oxides are re-normalised to 100% an indication of original cupel composition is given. In applying this methodology to cupel 412 and comparing the re-normalised results to that of cupel 402 it was found that despite heavy contamination with PbO through use the re-normalised composition matched closely the composition of the unused cupel. This demonstrates the method applied to cupel investigations is reliable.

To conclude, this research has established the metallurgists operating out of Legge's Mount during the 16th century were refining silver in which the main contaminant was copper. The process of silver refining would have been undertaken at the Royal Mint so the results of these analyses support the association of the Legge's Mount cupels with the operation of the Tudor Mint. The analytical data and SEM imaging substantiates the 16th-century descriptions of cupel manufacture.

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APPENDIX I

Catalogue of Legge's Mount cupels



Sample Number: 399
Colour: white
Diameter: 32.1mm
Height: 11.7mm
Weight: 16.32g
Comment: the body is granular and friable and the upper surface is smooth to granular. Sand and soil are adhering. The cupel is unused.



Sample Number: 400
Colour: white
Diameter: 32.2mm
Height: 12.5mm
Weight: 15.58g
Comment: the body and upper surface are granular and friable. The cupel is unused.



Sample Number: 401
Colour: body – white, upper surface - greyish white
Diameter: 32.4mm
Height: 12.2mm
Weight: 14.15g
Comment: the body is granular and friable and the upper surface is smooth and compact. The cupel is unused.



Sample Number: 402
Colour: body – white, upper surface – greyish white
Diameter: 31.5mm
Height: 12.4mm
Weight: 12.77g
Comment: the body is granular and friable and the upper surface is smooth and compact. The cupel is unused. Sampled for SEM-EDS analysis.



Sample Number: 403
Colour: body and upper surface – dark grey, base - white
Diameter: 33.0mm
Height: 16.6mm
Weight: 30.79g
Comment: the upper body is smooth, hard and consolidated with a depression in the centre of the upper surface. The base is white and friable. The cupel is used but not completely impregnated.



Sample Number: 404

Colour: yellow grey

Diameter: 31.1mm

Height: 9.1mm

Weight: 15.95g

Comment: while the majority of the cupel is hard and consolidated, there is a white granular area on the edge. The cupel is used but not completely impregnated.



Sample Number: 405

Colour: grey and yellow

Diameter: 30.1mm

Height: 9.4mm

Weight: 17.94g

Comment: like cupel 404, the majority of cupel 405 is hard and consolidated and has a white granular area on the edge. The cupel is used but not completely impregnated. The yellow discoloration is associated with an Fe-rich aggregate adhering to the edge.



Sample Number: 406

Colour: white grey, with dark grey to black circular mark in centre

Diameter: 32.9mm

Height: 12.7mm

Weight: 20.34g

Comment: while the cupel is relatively friable, the dark grey to black depression is hard and consolidated. The cupel is used but not fully impregnated.



Sample Number: 407

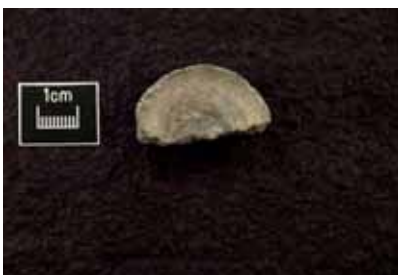
Colour: dark grey

Diameter: approx. 30.0mm

Height: 11.7mm

Weight: 4.34g

Comment: less than $\frac{1}{4}$ of the cupel survives. It is hard, consolidated and completely impregnated.



Sample Number: 408

Colour: dark grey

Diameter: 30.0mm

Height: 16.9mm

Weight: 14.91g

Comment: the cupel body is smooth and hard, with a black circular depression in the centre of the upper surface. It is used and fully impregnated.



Sample Number: 409
Colour: dark grey with pink and black traces.
Diameter: 29.8mm
Height: 9.1mm
Weight: 16.40g
Comment: the surface is hard and smooth. The cupel is used and fully impregnated.



Sample Number: 410
Colour: body – dark grey to black, base – white.
Diameter: not enough preserved.
Height: 13.5mm
Weight: 12.53g
Comment: the surviving body is smooth and hard and the base is granular and friable. The cupel is used but not fully impregnated.



Sample Number: 411
Colour: dark grey
Diameter: 30.0mm
Height: 11.4mm
Weight: 13.44g
Comment: the cupel is hard and consolidated, but has cracks through the body. It is used and completely impregnated.



Sample Number: 412
Colour: dark grey to black.
Diameter: approximately 30.0mm
Height: 9.8mm
Comment: The cupel is hard and consolidated, but with cracks across the surface. Sampled for SEM-EDS analysis.

APPENDIX II

Results of Exploratory XRF-Analysis of the Legge's Mount Cupels

Sample	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	CuO	PbO	AgO
399	0.6	2.6	23.1	0.76	71.7	0.1	0.1	0.2	0.0	0.85	0.0
400	0.7	3.2	24.6	0.89	70.0	0.1	0.1	0.1	0.0	0.38	0.0
401	0.8	2.2	16.6	1.16	78.9	0.09	0.1	0.2	0.0	0.06	0.0
402	0.7	2.0	17.2	0.71	76.3	0.06	0.0	0.2	0.0	2.68	0.0
403	3.0	7.0	10.2	1.26	26.6	0.1	0.0	0.8	1.87	49.04	0.2
404	3.9	6.6	11.9	3.95	46.3	0.11	0.0	0.9	1.22	24.85	0.3
406	4.6	5.8	11.7	1.16	40.5	0.33	0.0	3.3	0.83	31.49	0.2
407	3.0	9.5	12.9	1.4	32.3	0.07	0.1	0.5	2.3	37.38	0.6
408	9.3	16.8	13.8	1.8	29.7	0.25	0.1	0.8	0.9	26.14	0.2
409	2.5	5.4	11.9	1.2	52.3	0.08	0.0	0.5	1.12	24.63	0.3
410	3.6	9.4	13.56	1.7	22.4	0.11	0.0	0.8	2.59	45.09	0.7
411	3.6	14.9	10.89	0.8	24.3	0.42	0.	1.8	1.18	41.38	0.4

APPENDIX III

Minimum detection limits (MDL) for the SEM-EDS system

	MDL	Error (1sdev)
NaO ₂	0.5	0.5
MgO	0.3	0.3
Al ₂ O ₃	0.3	0.3
SiO ₂	0.3	0.7
P ₂ O ₅	0.2	0.1
SO ₃	0.2	0.1
K ₂ O	0.1	0.4
CaO	0.1	0.3
TiO ₂	0.1	0.1
MnO ₂	0.1	0.1
Fe ₂ O ₃	0.1	0.1
CoO	0.1	0.1
CuO	0.1	0.1
ZnO	0.1	0.1
As ₂ O ₃	0.5	0.3
SnO ₂	0.3	0.3
PbO	0.3	0.3

APPENDIX IV
Composition of cupel 412

Area	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₂	CaO	Fe ₂ O ₃	CuO	Ag ₂ O	PbO	Pb/Cu ratio
1 (top)	<0.3	0.3	0.4	27.7	0.3	39.9	0.3	2.0	0.5	28.6	14.4
2	<0.3	0.3	0.3	23.7	0.1	36.0	0.4	1.1	1.1	36.8	33.4
3	0.3	0.3	0.2	21.8	0.1	36.5	0.1	0.8	0.2	39.7	49.6
4	<0.3	0.2	0.3	22.0	0.6	38.0	<0.1	0.7	0.2	37.7	53.8
5	0.3	0.2	0.6	23.9	0.4	38.8	0.1	0.9	<0.2	34.9	38.7
6	0.5	0.2	0.4	25.9	0.4	43.9	0.1	0.9	<0.2	27.9	31.0
7 (base)	0.3	0.3	0.2	28.8	0.4	45.9	0.1	0.3	<0.2	23.9	79.6



ENGLISH HERITAGE RESEARCH DEPARTMENT

English Heritage undertakes and commissions research into the historic environment, and the issues that affect its condition and survival, in order to provide the understanding necessary for informed policy and decision making, for sustainable management, and to promote the widest access, appreciation and enjoyment of our heritage.

The Research Department provides English Heritage with this capacity in the fields of buildings history, archaeology, and landscape history. It brings together seven teams with complementary investigative and analytical skills to provide integrated research expertise across the range of the historic environment. These are:

- * Aerial Survey and Investigation*
- * Archaeological Projects (excavation)*
- * Archaeological Science*
- * Archaeological Survey and Investigation (landscape analysis)*
- * Architectural Investigation*
- * Imaging, Graphics and Survey (including measured and metric survey, and photography)*
- * Survey of London*

The Research Department undertakes a wide range of investigative and analytical projects, and provides quality assurance and management support for externally-commissioned research. We aim for innovative work of the highest quality which will set agendas and standards for the historic environment sector. In support of this, and to build capacity and promote best practice in the sector, we also publish guidance and provide advice and training. We support outreach and education activities and build these in to our projects and programmes wherever possible.

We make the results of our work available through the Research Department Report Series, and through journal publications and monographs. Our publication Research News, which appears three times a year, aims to keep our partners within and outside English Heritage up-to-date with our projects and activities. A full list of Research Department Reports, with abstracts and information on how to obtain copies, may be found on www.english-heritage.org.uk/researchreports

For further information visit www.english-heritage.org.uk

