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Summary

Excavations revealed part of a large quarry pit, which contained quantities of combed slipware waste, metalworking slag, Isleworth porcelain and bone ash cupels. The site, which is associated with the Isleworth pottery works, was dated to the first half of the 19th century. Qualitative analysis of the cupels' surface revealed that they had been used to purify or assay silver. Quantitative analysis of the bone ash cupels and Isleworth porcelain was also carried out and this indicated that the cupels were being crushed and reused in the porcelain fabric.

Keywords

Metal Working-Fe
Metal Working-non Fe
Silver
Pottery
Technology
Post Medieval

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Introduction

Excavations at 223 Hanorth Road, Hounslow, revealed part of a large quarry pit. The upper fills contained considerable quantities of combed slipware waste along with fragments of kiln furniture, slag, Isleworth porcelain and 45 bone ash cupels. The site, which is associated with Isleworth pottery works, was dated to the first half of the 19th century.

These finds posed a number of questions which this report will attempt to answer. The main one is why the cupels were present, in such large numbers, at a pottery works. It is possible they were one of the raw materials used in the production of the porcelain. The other subsidiary questions relate to the cupels themselves: whether they had been used and if so whether it was gold or silver that had been refined or assayed in them.

Background

Porcelain is a fine translucent or semi translucent earthenware that was first produced in China and Japan and imported from the 15th century onwards into Europe. The European porcelain industries carried out many experiments during the development of their own porcelain fabrics and, because true hard paste porcelain proved difficult to reproduce, a number of soft paste porcelains were developed. By 1750 three principle types of soft paste porcelain were in production; these were glassy porcelain similar to French *pâte tender*, soapstone porcelains using soapstone rock from the Lizard peninsula, and bone-ash porcelains (Tite and Bimson 1991, 3-4).

Bone ash porcelain and bone china are characterised by the presence of calcium phosphate which is derived from bone. A recipe for the early 18th century Bow bone-ash porcelains shows that ‘Lynn sand’, ‘blue ball clay’, bone ash and gypsum or alabaster were used. After 1789 ‘hard paste’, a china clay and china stone mix, was used in the production of bone china. The fabric of 18th century bone-ash porcelains and the later bone china both consisted of varying amounts of unreacted quartz and abundant β-tricalcium phosphate (whitlockite $Ca_3(PO_4)_2$) in a glassy matrix. Unlike the bone-ash porcelain, the later bone china also contained abundant anorthite ($CaAl_2Si_2O_8$) due to the large amounts of alumina in the hard paste (Tite and Bimson 1991, 13-17).

Porcelain, slipwares and red stonewares, were all being produced at Isleworth between 1756 and the 1790s. Red earthenware vessels were also being produced at a pottery in Hounslow. A large number of combed slipwares found during the excavations were wasters and may have been produced at either Isleworth or Hounslow. Large amounts of kiln furniture were found, some of which was associated with porcelain production at Isleworth (Keily pers comm. 2004).

It is unlikely that cupels would be used at a pottery as they are associated with precious metal workshops. The presence of cupels at this pottery workshop suggests that they may have been reused in some way, possibly in the manufacture of the porcelain. One possibility is that they were to be crushed and added to the porcelain fabric.

Cupels were used in the cupellation process, either to separate precious metals from base ones or assay (test the purity of) precious metals. The impure precious metal was melted with lead, which formed lead oxide (litharge) that dissolved any base metals present (Bayley *et al.* 2001, 19-20). Bone ash cupels were first known in England in the 16th century. They were very efficient because they did not react with the litharge but just absorbed it (Bayley 1992, 6).The purified silver and/or gold was left on the cupel surface (Bayley *et al.* 2001, 19-20).

Assemblage

Twelve bone ash cupels from context 32 were studied, plus two that were unstratified. The cupels all appear to be of similar size and shape, circular with tapering sides and concave surface. They measure 24-25mm in diameter, 17-20mm in height and they weigh between 19 to 21g each (Figure 1). Seven fragments of unglazed porcelain from the same context were also examined.

Some slag and a possible crucible fragment, recovered from another fill in the same pit, were also studied. These are discussed in appendix 2 as they were found to be waste from processes unrelated to the pottery production or cupellation.

Methods

The surfaces of 12 of the bone ash cupels were analysed using energy dispersive X-ray fluorescence (EDXRF), providing a non-destructive, semi-quantitative method of identifying the elements present and mapping their distribution. Prior to analysis the objects were not cleaned as this would have removed the areas of interest. The distribution of elements, on the surface of the cupels and across a section, was mapped using the XRF by moving the stage in 1mm increments and analysing the surface at each point.

Two cupels were cut in half and mounted in epoxy resin. Small fragments of the porcelain were also mounted in resin. All of the samples were then ground and polished to a 1-micron finish and coated with carbon. Examination and analysis was carried out using a scanning electron microscope (Karl Zeiss Stereoscan 440I) in backscatter electron mode. The composition of a small area (typically 3mm by 2mm) was determined using an energy dispersive spectrometer (with Germanium detector) attached to the scanning electron microscope. Spectra were collected at 25kV and 2nA for 100 seconds live time and quantified using the Oxford Instruments SEMQuant software, calibrated with a cobalt standard. The analytical results are given in appendix 1 with the results for each sample in table 5. Table 6 compares results obtained for standards of known composition. These are generally in good agreement, demonstrating the accuracy of the data in table 5. The results have been presented as if all the elements detected were present as oxides, although there will be some uncombined elements present.

Cupels

Analytical results

Surface mapping (Figure 2) of the cupels revealed that the elements present on the surface of the cupel vary in concentration. The elements present were predominantly calcium and phosphorus (bone ash), lead (litharge), silver in varying amounts and small quantities of copper. The calcium and phosphorus were detected all across the cupel but were concentrated around the edges while the lead and copper were evenly distributed across the majority of the surface but with peaks near the centre. The lead varied in concentration from 18% to 54% and the copper ranged from 0.2% to 1.7% across the surface. The highest concentrations of lead and copper were often located in areas that also had higher concentrations of silver present. The highest concentration of silver formed a ring centred on the small depression in the middle of each cupel. The amount of silver present on the surface of the cupel ranged from 0% to 11% (calculated as an oxide).

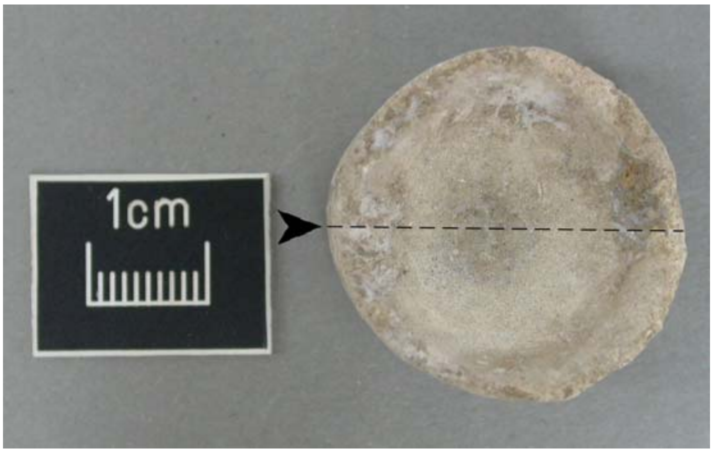

Figure 1 Surface view of one of the cupels that was mapped and sectioned, see below for the distribution of the elements present. The arrow and dashed line indicates where the section was taken.

The element map of a full cross section of a cupel (Figure 3) revealed that calcium, phosphorus and lead were also present throughout the cupel. The silver was concentrated in a small area at the surface of the cupel near the centre and penetrated only about 3mm into the bone ash.

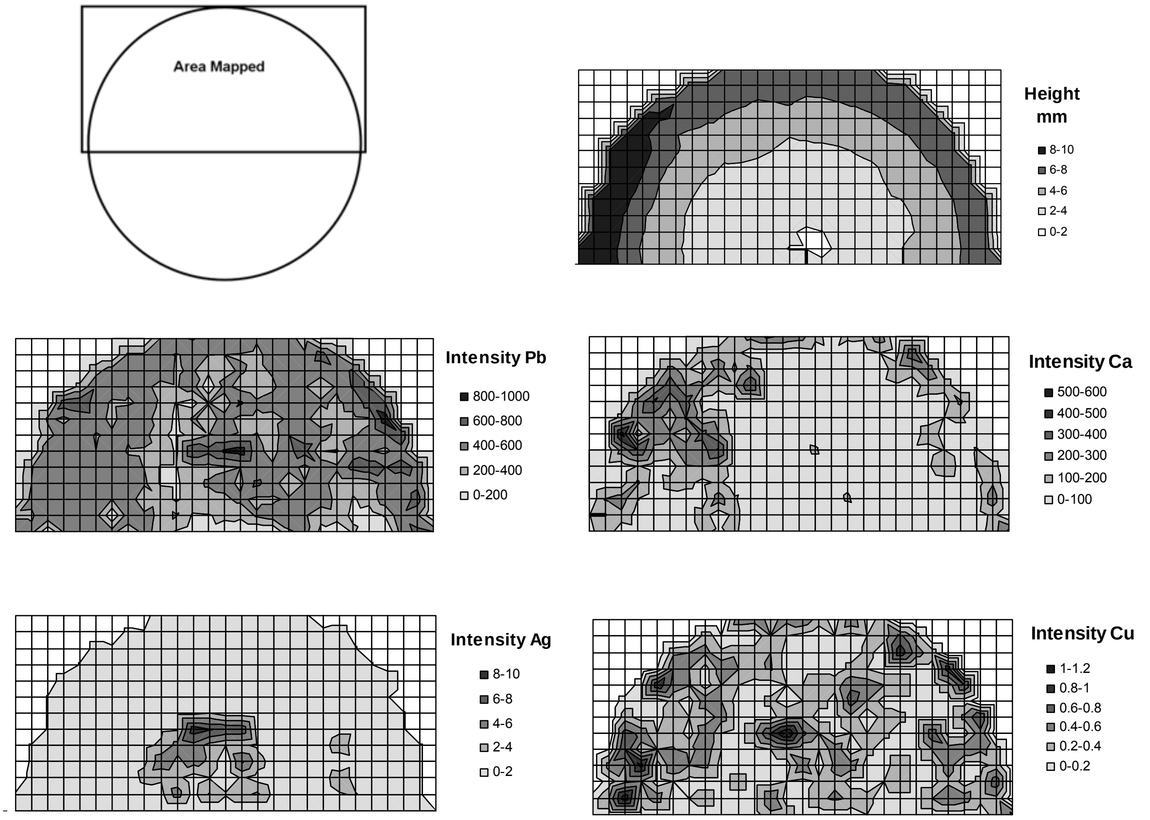

Figure 2 Surface maps of a cupel showing its topography and the relative intensities for each element, in a 1mm square grid. Top-right: topography, middle-left: lead, middle-right: calcium, bottom-left: silver and bottom-right: copper.

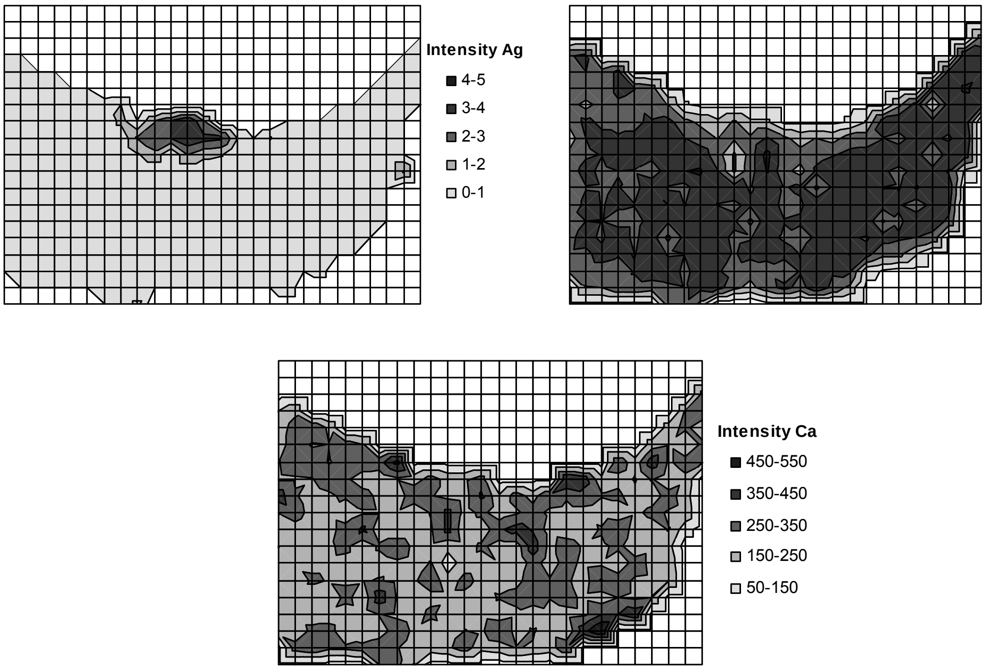

Figure 3 Section maps of a cupel (Figure 1) showing the relative intensities for each element in a 1mm square grid. Top-left: silver, top-right: lead and bottom: calcium. The bottom right edge of the cupel had been heavily eroded.

SEM analysis was carried out on areas in the centre of the cupel which the XRF mapping had shown to be of reasonably uniform composition, so as to obtain a composition representative of the majority of the cupel. This analysis revealed that both cupels contained 40% or more lead oxide and the rest was bone ash with traces of copper, magnesia and silica. The microstructure of the cupels revealed abundant bone ash, some of which was lead-rich, in a lead-rich glass matrix.

Table 1: The chemical composition of two cupels (data from table 5).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	CuO	SO_3	PbO
Cupel 1	<0.5	0.3	<0.3	0.1	21.7	<0.1	36.4	<0.1	0.4	<0.2	40
Cupel 2	<0.5	0.3	0.4	0.3	19.3	<0.1	34.3	<0.1	0.4	<0.2	44.4

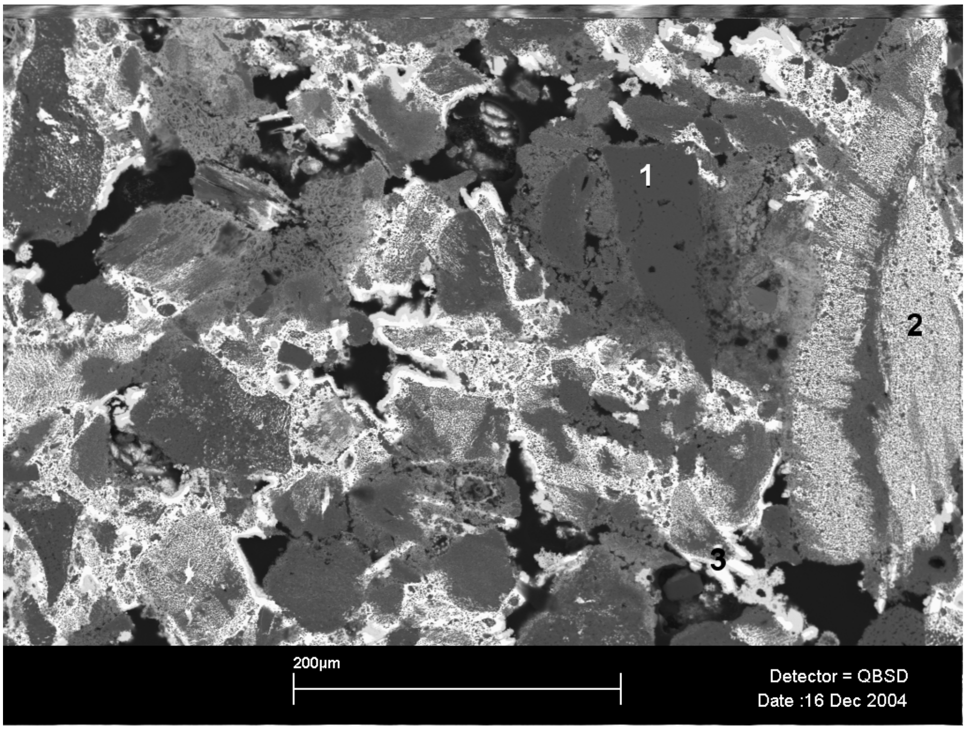

Figure 4 Backscatter SEM image of the microstructure of cupel 2 with abundant amounts of 1) bone ash (dark grey), 2) lead-rich bone ash (grey), probably re-crystallised $Ca_3(PO_4)_2$ mixed with litharge, and 3) lead oxide (white) in a lead-rich glassy matrix (light grey). The black areas indicate voids.

Discussion

XRF analysis of the surface of 12 cupels showed that they were used for refining or assaying silver, as this was the only precious metal detected. The distribution of elements across the surface of the cupel can be explained in terms of the cupellation process. As the litharge is absorbed into the bone ash cupel the silver-rich litharge would coalesce in the centre. The areas around this deposit would be the last places where the litharge could be absorbed. Towards the end of the process there is no longer an excess of litharge present so some silver gets bound up with cuprite (copper oxide) and is trapped in the last of the litharge, leaving a silver-rich ring around the central depression where the droplet of purified silver solidified (Bayley and Eckstein forthcoming). Note the coincidence of elevated levels of copper and silver in the centre of the cupel (Figure 2)

Isleworth porcelain

Analytical results

The Isleworth porcelain had a fairly typical bone-ash porcelain composition (Table 2), although it had more alumina (Al_2O_3), potash (K_2O) and lead but lower quantities of phosphorus than the porcelain analysed by Tite & Bimson (Table 3). Analysis of some other Isleworth porcelain by Freestone *et al*. (2003) revealed a similar composition to the samples in table 5, with over 3% lead, high potash and sulphur. The presence of sulphur in the porcelain may suggest that gypsum ($CaSO_4$) was one of the ingredients being added. The Isleworth porcelain had over 3% lead oxide in the fabric, compared to the 0.4% PbO found in some Bow porcelain by Tite & Bimson (1991). However bone china, produced with hard paste, had higher alumina and phosphorus compared to the Isleworth porcelain (Table 3). This suggests that the porcelain from Isleworth was not made using hard paste but using a combination of sand, clay, gypsum and bone-ash, possibly using crushed cupels to provide part of the bone ash. During Freestone's analysis of Isleworth porcelain angular fragments of frit or devitrified soda-lime-silica glass were identified within the microstructure. None was identified in this study but due to the very similar composition of the porcelain it is likely that it was also an ingredient added to the Isleworth bone-ash porcelain studied.

Two fragments of the analysed porcelain were of different compositions. One (sample 7) was a fragment of bone-ash porcelain that had low levels of lead, alumina and sulphur compared to the others. This fragment was almost identical to the bone-ash porcelains analysed by Tite and Bimson (Table 3). The other fragment (sample 8) was very different from the other pieces of bone-ash porcelain and more similar to glassy porcelains with high concentrations of lead, silica and alumina analysed by Tite and Bimson (Table 3). These two atypical wasters may represent later developments at Isleworth.

Table 2: The chemical composition of porcelain from Isleworth (data from table 5). The average is the mean of samples 3-6 and 9, sample 7 has lower lead and sulphur contents and sample 8 is clearly different.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	CuO	SO_3	PbO
Average	0.9	0.7	8.8	38.0	16.4	1.7	26.2	0.3	<0.1	2.6	3.2
Sample 7	0.6	0.4	6.5	46.3	18.7	1.5	24.4	0.3	<0.1	0.5	0.2
Sample 8	0.8	<0.3	10.4	69.0	0.4	4.1	4.9	0.6	<0.1	0.3	8.4

Table 3: The average chemical composition of bone-ash porcelain, hard paste bone china, glassy porcelain (after Tite and Bimson 1991) and bone-ash porcelain from Isleworth analysed by Freestone et al (2003).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	SO_3	PbO
Bone-ash porcelain	0.7	0.5	7.5	47.4	17.5	1.0	23.8	0.3	2.0	0.4
Bone china	0.8	0.6	13.7	41.8	17.8	2.2	23.1	nm	nm	nm
Glassy porcelain	0.6	0.3	3.6	70.5	1.9	3.4	15.0	0.3	nm	5.8
Isleworth porcelain	0.7	0.6	7.4	40.5	17.0	2.0	25.6	0.4	2.6	3.2

nm = not measured

The microstructure of the porcelain (Figure 5) was very different from that of the cupels. Most of the porcelain consisted of unreacted quartz, β-tricalcium phosphate ($Ca_3(PO_4)_2$) and a small quantity of anorthite ($CaAl_2Si_2O_8$) in a glassy matrix. The presence of anorthite is related to the concentration of alumina in the porcelain (Tite and Bimson 1991, 16). Porcelain sample 8 had a very different microstructure compared to the other fragments, with large clusters of fine grained silica recrystallized in a lead rich glassy matrix (Figure 6). This is typical of glassy porcelain (Tite and Bimson 1991, 10-13) and is a result of the high lead content.

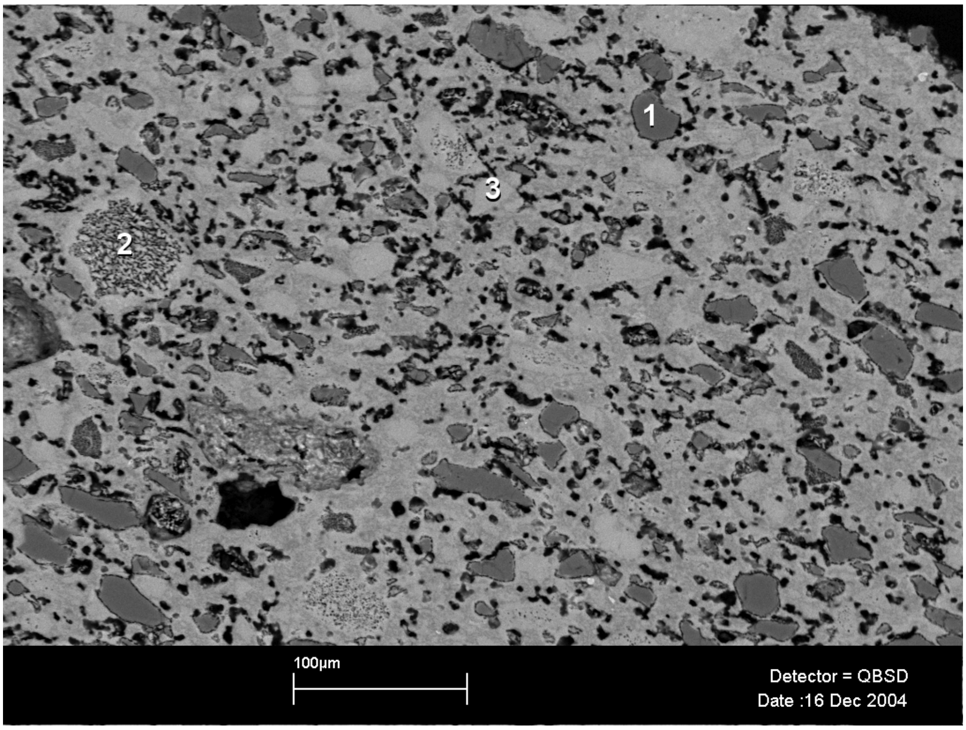

Figure 5 Backscatter SEM image of porcelain sample 9 showing 1) unreacted quartz (grey), 2) β-tricalcium phosphate (grey and white) and 3) anorthite (light grey) in a glassy matrix. The black areas indicate voids.

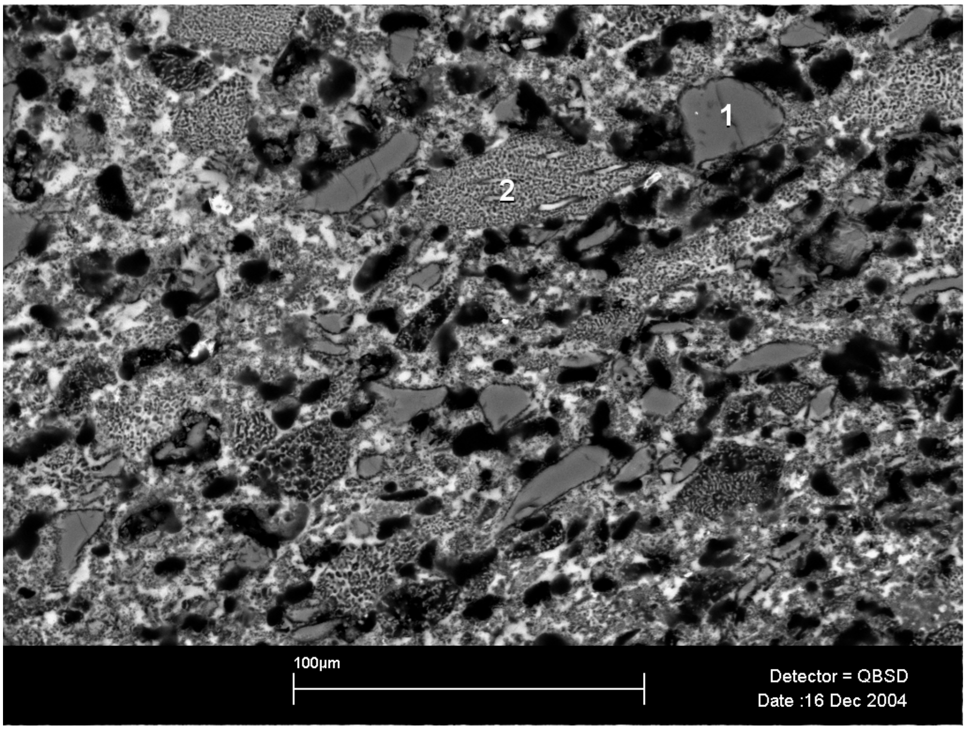

Figure 6 Backscatter SEM image of sample 8, similar to that of glassy porcelain, with 1) unreacted quartz (dark grey) and 2) clusters of fine grained recrystallized silica (grey) in a lead-rich glassy matrix (light grey and white). The black areas indicate voids.

Discussion

The presence of lead in all but one of the bone-ash porcelain fragments indicates that material containing a significant quantity of lead was being added to the porcelain. Freestone *et al.* (2003, 286) suggested that either potter's lead, litharge, or flint glass was added to the porcelain body. In their study, potash was plotted against alumina for various porcelain fabrics and it was noted that the ratio of potash to alumina for the Isleworth porcelains was higher than that for Bow and Liverpool porcelains. This was interpreted as indicative of another source of potash, such as flint glass or glaze frit, being added to the Isleworth porcelain in addition to the clay used. However the results of this study indicate that the high ratio of potash to alumina in the Isleworth porcelains is not necessarily linked to the addition of flint glass but instead may be largely a characteristic of the clay used. This conclusion is supported by the composition of porcelain fragment 7, which contains low levels of lead but has a similarly high ratio of potash to alumina as found in the more typical samples (Table 2, Figure 7). In contrast sample 8, a lead-rich glassy porcelain, does have a significantly higher ratio of alumina to potash than the other samples, consistent with the addition of flint glass or glaze frit containing lead and potash to the fabric. However the ratio of titania to alumina is similar for all of the samples (Figure 7) suggesting that a similar type of clay was used for all three types of Isleworth porcelain fabric.

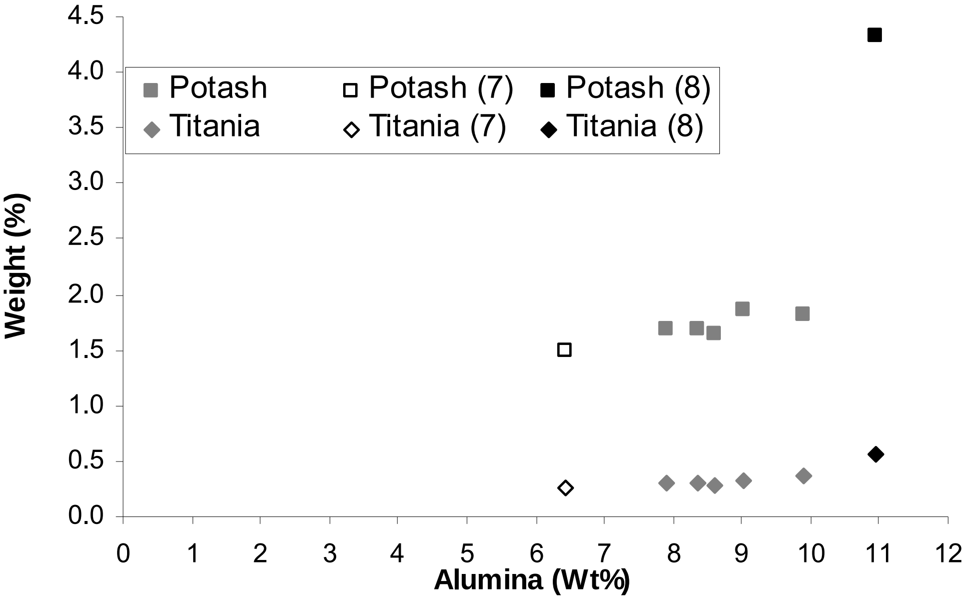

Figure 7: A graph showing alumina (Al_2O_3) against both potash (K_2O) and titania (TiO_2) contents in bone-ash porcelain from Isleworth.

The presence of lead-rich cupels at the pottery works indicates that they were being recycled in some way. An alternative source of lead, such as lead-rich flint glass or glaze frit, discussed above, does not appear to have been used and so the cupels may be the source of lead in the majority of Isleworth porcelain fabrics. Table 4 below uses the estimated chemical compositions, which may vary, of the raw materials that may have been used at Isleworth to determine the possible weight percentages needed to create bone-ash porcelain with a similar composition. If the bone ash cupels were used as a raw material then about 8% of crushed cupel would have been mixed with up to 34% new bone ash to account for the amount of lead and calcium phosphate in the porcelain (Table 4). The remainder of the porcelain would have been made up of clay, sand, soda-lime-silica glass and gypsum. (Although no surviving fragments of soda-lime-silica glass frit were identified in the samples examined in this study, they have been noted in other studies of similar fabrics (Freestone *et al*. 2003, 286) so this material is included in the calculations below).

Table 4: Approximate compositions of ceramic raw materials above (Holdridge 1956, 373; Dungworth forthcoming) and proportions mixed to give the porcelain composition below.

Raw Materials	**Na2O**	**Al2O3**	**SiO2**	**P2O5**	**SO_3**	**K2O**	**CaO**	**PbO**
Crushed cupels	<0.5	0.3	0.2	21.0	0.1	<0.1	35.3	40.9
New bone-ash				43			57	
Gypsum					59		41	
Soda-lime-silica glass	12.7	1.8	66	0.2		5.7	8.7	1.1
Ball clay	0.5	37	56			5		
Sand			100					

	Na2O	**Al2O3**	**SiO2**	**P2O5**	**SO_3**	**K2O**	**CaO**	**PbO**	**% Weight**
Crushed cupels				1.7			2.8	3.2	8
New bone-ash				14.7			19.5		34
Gypsum					2.6		1.8		4
Soda-lime-silica glass	0.8	0.1	4.1			0.4	0.5	0.1	6
Ball clay	0.1	8.8	13.4			1.2			24
Sand			19.5						20
Total	**0.9**	**8.9**	**38.0**	**16.4**	**2.6**	**1.6**	**24.6**	**3.2**	
Porcelain Total	**0.9**	**8.8**	**38.0**	**16.4**	**2.6**	**1.7**	**26.2**	**3.2**	

Conclusion

The cupels were used to refine or assay silver. Element mapping of surfaces and sections revealed interesting distribution patterns. Higher concentrations of silver were found in a ring around the central depression of each cupel and no traces were found near the edges of the cupel. In this central area the silver has been absorbed up to 3mm deep into the cupel. This information on spatial distribution may assist in selecting the best areas for spot analysis of cupels in future research, to determine whether gold or silver was being purified.

Cupels would normally suggest a precious metal workshop but their presence at the Isleworth pottery works suggests that they were being reused in some way. Analysis of fragments of bone china porcelain from the same site revealed that lead was present in sufficient quantities to suggest that the bone ash cupels were being crushed and then added to the porcelain fabric.

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Appendix 1

Table 5: Chemical composition of the samples from Hounslow (measured by EDS, wt% normalised, silver below detection limit (0.5wt%)).

Sample		Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	Fe_2O_3	CuO	PbO
1	Cupel	<0.5	0.3	0.3	0.2	21.3	<0.1	0.5	<0.1	36.9	<0.1	<0.1	0.4	40.1
		<0.5	0.4	0.3	0.2	19.6	<0.1	0.6	<0.1	34.3	<0.1	<0.1	0.7	43.6
		<0.5	0.4	0.3	0.1	24.2	<0.1	0.3	<0.1	37.9	0.1	<0.1	0.2	36.3
2	Cupel	<0.5	0.3	0.1	0.2	20.2	0.1	0.3	<0.1	34.7	<0.1	<0.1	0.4	43.4
		<0.5	0.4	0.6	0.4	19.0	<0.1	0.7	<0.1	33.8	<0.1	0.1	0.3	44.6
		<0.5	0.3	0.3	0.2	18.5	0.1	0.6	<0.1	34.2	0.1	<0.1	0.4	45.3
3	Porcelain	0.6	0.7	10.1	36.7	17.2	2.9	0.1	1.8	25.3	0.4	0.5	<0.1	3.5
		0.8	0.7	9.6	36.5	17.5	2.7	0.2	1.8	25.7	0.3	0.6	<0.1	3.6
		0.7	0.6	9.9	38.5	16.6	2.6	0.2	1.9	24.7	0.4	0.5	<0.1	3.5
4	Porcelain	0.7	0.7	7.8	36.8	16.8	2.9	0.1	1.6	28.8	0.3	0.4	0.1	3.0
		0.9	0.7	8.0	39.4	15.3	2.7	0.2	1.7	27.2	0.3	0.5	<0.1	3.1
		1.0	0.7	8.0	39.6	15.0	2.6	0.2	1.7	27.3	0.3	0.5	0.1	3.2
5	Porcelain	1.2	0.8	8.6	38.9	15.6	2.3	0.3	1.7	26.9	0.3	0.5	<0.1	3.0
		0.9	0.8	8.8	38.4	15.3	2.5	0.2	1.6	27.6	0.3	0.3	0.1	3.1
		0.7	0.7	8.6	39.1	15.6	2.5	0.2	1.6	27.2	0.3	0.4	<0.1	3.1
6	Porcelain	1.0	0.8	8.6	36.3	16.2	2.7	0.1	1.7	28.4	0.3	0.5	<0.1	3.3
		1.2	0.8	9.3	37.5	17.3	2.9	0.2	1.8	24.9	0.4	0.5	<0.1	3.2
		1.0	0.8	9.4	37.2	17.1	2.8	0.2	2.0	25.3	0.3	0.5	<0.1	3.5
7	Porcelain	0.6	0.4	6.5	43.9	19.9	0.5	0.1	1.5	25.8	0.2	0.6	<0.1	0.2
		0.6	0.5	6.6	45.8	18.8	0.7	<0.1	1.5	24.6	0.3	0.4	<0.1	0.3
		0.6	0.4	6.4	49.3	17.5	0.4	<0.1	1.5	22.9	0.3	0.5	<0.1	0.2
8	Porcelain	0.9	0.1	10.2	69.6	0.3	0.1	0.4	4.0	4.8	0.6	0.6	<0.1	8.2
		0.8	0.1	10.4	69.5	0.4	0.3	0.3	4.1	4.8	0.5	0.5	<0.1	8.2
		0.6	0.1	10.7	67.8	0.4	0.4	0.3	4.3	5.2	0.6	0.6	<0.1	8.9
9	Porcelain	0.8	0.8	8.5	38.7	17.7	2.6	0.2	1.6	25.5	0.3	0.4	<0.1	2.8
		0.9	0.7	8.4	38.7	17.6	2.7	0.2	1.7	25.2	0.3	0.4	<0.1	3.1
		0.8	0.7	8.3	40.0	17.3	2.5	0.1	1.7	24.8	0.3	0.5	<0.1	3.0

Table 6: Corning Glass Standards (measured by EDS, normalised, average of three).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3	CuO	ZnO	SnO_2	SbO	BaO	PbO
Standard A	15.3	2.7	1.0	66.0	0.1	0.1	2.8	5.0	0.8	1.2	1.0	1.2	0.10	0.17	1.7	0.4	0.1
SEM Analysed A	14.5	2.8	1.0	66.6	0.1	0.2	2.9	5.3	0.8	1.2	1.1	1.2	<0.1	<0.3	1.7	0.5	0.1
Standard B	18.1	1.0	4.3	60.7	0.9	0.6	1.0	8.7	0.1	0.3	0.3	2.6	0.20	0.02	0.3	0.1	0.5
SEM Analysed B	17.3	1.2	4.2	61.6	0.8	0.5	1.1	8.7	0.1	0.3	0.4	2.7	0.20	<0.3	0.5	0.1	0.4
Standard C	0.8	2.8	1.0	34.2	0.1	0.4	2.8	5.2	0.8	<0.1	0.3	1.2	0.04	0.16	<0.1	11.6	38.0
SEM Analysed C	1.2	2.8	0.9	34.9	0.1	0.2	2.7	5.1	0.8	<0.1	0.3	1.2	0.04	<0.3	<0.1	12.1	36.9
Standard D	1.6	4.1	5.4	55.0	4.1	0.3	11.1	14.8	0.4	0.7	0.5	0.4	0.10	0.00	0.7	0.3	0.3
SEM Analysed D	1.3	4.1	5.4	55.2	4.0	0.3	11.5	15.1	0.4	0.6	0.5	0.4	0.10	<0.3	1.0	0.3	0.3

Appendix 2

Assemblage

The slag assemblage was waste from iron working and consisted of mostly undiagnostic slag, fragments of smithing hearth bottom and one piece of possible tap slag. The undiagnostic slag did not have sufficient characteristics to be categorised. The smithing hearth bottom has a characteristic shape with an almost flat top with a curved base and is a waste product from smithing. The piece of tap slag had a surface that resembled the flow of lava and is a waste product from iron smelting (Bayley *et al.* 2001, 19-20). There was also a fragment of possible crucible in the assemblage.

Methods

The ironworking waste and the large crucible found in the quarry pit located in trench 3 were analysed using energy dispersive X-ray fluorescence (EDXRF), providing a non-destructive qualitative method of identifying the elements present. Prior to analysis the objects were not cleaned as this would have removed the areas of interest.

Analytical Results

During XRF analysis of the big crucible, traces of copper, tin and zinc were detected on the surface, therefore this crucible was probably used in copper alloy casting. XRF analysis of the ironworking waste revealed that this was predominately iron silicate and some sulphur was also detected. This is typical of 18th century ironworking waste where coke which contained some sulphur was used as fuel instead of charcoal. Iron working slag is often removed from the site where it was produced and either reused (eg, as hardcore) or dumped elsewhere. Its presence does not always indicate that ironworking took place in the immediate vicinity. The lack of large amounts of metalworking debris and associated structures suggests that metalworking was not taking place in, or near, the excavation area.