

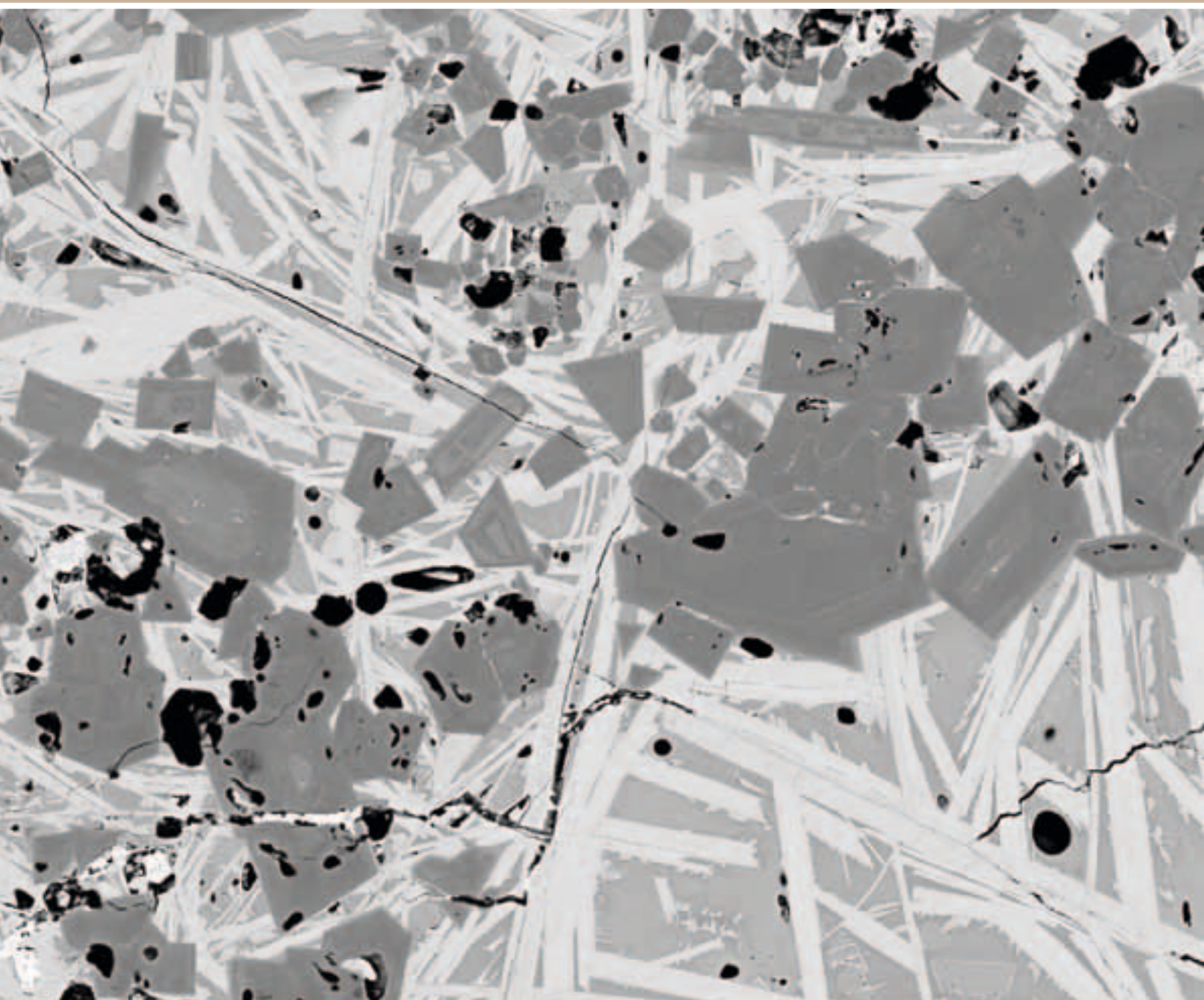
RESEARCH REPORT SERIES no. 15-2012

# ST ALGAR'S FARM, FROME, SOMERSET

## THE ANALYSIS OF LEAD-WORKING WASTE

TECHNOLOGY REPORT

Joanna Dunster and David Dungworth



INTERVENTION  
AND ANALYSIS



ENGLISH HERITAGE

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## **SUMMARY**

Metal-working waste from a Roman-period settlement is examined to determine the processes responsible for its formation. SEM-EDS analysis confirms that most of this material is litharge, a waste material formed during the extraction of silver from lead.

## **ACKNOWLEDGEMENTS**

Material for this research was provided from the excavations at St Algar's Farm, courtesy of Ceri Lambdin. Thanks are owed to the land-owner for permitting the study of this important early industrial site. This report would not have been possible without the guidance and support of Sarah Paynter and David Dungworth. I am most grateful to Roger Wilkes for his continued generosity with technical expertise, this work is dedicated to his happy retirement from English Heritage.

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## INTRODUCTION

The archaeological excavations undertaken on the site of the Roman settlement at St Algar's Farm, Frome in 2010 and 2011 recovered quantities lead and related materials. The related materials have generally comprised small lumps with a sufficiently high density for the suggestion that they are associated with lead working. During excavation in 2011 examination of several lumps of this material in the field led to the suggestion this it was litharge.

## METHODS

All of the material was examined visually to assess colour, size and shape (Bayley *et al* 2001). Most samples had a surface comprising cream-coloured powdery corrosion products (probably cerussite,  $\text{PbCO}_3$ ). In addition, the density of all samples was gauged by hand. Malleability was also assessed by applying a light strain (by hand) to each sample — samples with appreciable malleability were clearly metallic lead while those with no malleability were litharge, slag or some other material. Once assigned to a category, all material was weighed.

A small selection of samples were examined in more detail. This included an examination of microstructure using a scanning electron microscope (SEM, FEI Inspect F) and analysis using an energy dispersive spectrometer (EDS, Oxford instruments X-act) attached to the SEM. The analysed samples were mounted in epoxy resin and ground and polished to a 1-micron finish.

## RESULTS

### Visual examination

The waste from metal-working at St Algar's Farm was sorted into categories of metallic lead and other material, which includes litharge, hearth-lining, slag and some fragments of unreacted ore. The litharge has a cream-coloured corroded surface and a dark pink-red core (where fresh fracture surfaces are available), and this suggests that it contains no copper (which tends to render litharge green. This suggests that this is primary litharge produced during the extraction of silver from lead, rather than secondary litharge produced during the recovery of silver from debased silver alloys (cf Bayley *et al* 2001).

Table 1. Summary of metallic lead, litharge and related materials

| Trench       | Context | SF                 | No         | Weight       | Type  |
|--------------|---------|--------------------|------------|--------------|---|
| 1            | 10      |                    | 3          | 33           | metallic Pb   |
| 1            | 30      |                    | 4          | 150          | metallic Pb, fuel waste                             |
| 7            | spoil   |                    | 1          | 19           | metallic Pb   |
| 7            | 20      |                    | 2          | 17           | metallic Pb   |
| 7            | 50      |                    | 13         | 354          | metallic Pb, litharge, fuel waste                   |
| 7            | 60      |                    | 1          | 14           | metallic Pb   |
| 7            | 70      |                    | 13         | 239          | metallic Pb, litharge                               |
| 8            | 10      |                    | 1          | 43           | metallic Pb   |
| 8            | 30      |                    | 1          | 14           | metallic Pb   |
| 8            | 80      |                    | 1          | 26           | metallic Pb   |
| 9            | 20      |                    | 1          | 19           | metallic Pb   |
| 9            | 40      |                    | 4          | 62           | metallic Pb   |
| 9            | 50      |                    | 2          | 202          | metallic Pb, unreacted ore                          |
| 9            | 60      | 15                 | 1          | 641          | metallic Pb   |
| 10           | 10      |                    | 4          | 37           | metallic Pb   |
| 10           | 20      | 3                  | 32         | 116          | metallic Pb, litharge                               |
| 10           | 30      | 8                  | 92         | 654          | metallic Pb   |
| 100          | spoil   |                    | 5          | 104          | metallic Pb   |
| 100          | 102     |                    | 6          | 74           | metallic Pb, fuel waste                             |
| 100          | 103     |                    | 1          | 10           | metallic Pb   |
| 100          | 104     |                    | 2          | 145          | litharge, Pb-rich waste                             |
| 100          | 105     |                    | 11         | 183          | metallic Pb   |
| 100          | 106     |                    | 4          | 72           | metallic Pb, Pb-rich waste                          |
| 200          | spoil   | 229                | 1          | 36           | metallic Pb   |
| 200          | 201     | 203, 204, 205, 206 | 26         | 399          | metallic Pb   |
| 200          | 202     |                    | 47         | 1936         | metallic Pb, litharge, fuel waste                   |
| 200          | 204     |                    | 3          | 440          | metallic Pb, litharge                               |
| 200          | 205     | 209, 212, 218      | 146        | 3491         | metallic Pb, litharge, hearth lining, Pb-rich waste |
| 200          | 206     |                    | 21         | 1673         | metallic Pb, litharge, hearth lining                |
| 200          | 208     |                    | 27         | 1136         | metallic Pb, litharge, Pb-rich waste                |
| 200          | 211     |                    | 3          | 63           | metallic Pb, litharge, hearth lining                |
| 300          | 307     |                    | 1          | 101          | hearth lining                                       |
| 400          | 402     |                    | 3          | 31           | litharge  |
| 400          | 406     |                    | 1          | 4            | metallic Pb   |
| 400          | 408     |                    | 1          | 37           | metallic Pb   |
| 400          | 418     |                    | 2          | 198          | litharge  |
| 400          | 422     |                    | 4          | 32           | litharge  |
| 400          | 427     |                    | 1          | 73           | litharge  |
| 200/500      | spoil   |                    | 21         | 553          | metallic Pb, hearth lining, ceramic, fuel waste     |
| 500          | spoil   |                    | 7          | 67           | metallic Pb, litharge                               |
| 500          | 502     |                    | 33         | 470          | metallic Pb, litharge, hearth lining, Pb-rich waste |
| 500          | 503     |                    | 139        | 2827         | metallic Pb, litharge                               |
| 500          | 504     |                    | 69         | 3147         | metallic Pb, litharge, hearth lining, Pb-rich waste |
| 500          | 505     |                    | 98         | 2951         | metallic Pb, litharge, hearth lining, Pb-rich waste |
| 500          | 506     |                    | 25         | 1307         | metallic Pb, litharge, hearth lining                |
| 600          | 603     |                    | 5          | 256          | metallic Pb, litharge                               |
| <b>Total</b> |         |                    | <b>889</b> | <b>24456</b> |   |

## Scientific examination

Twelve samples were selected from among the litharge and lead related materials for scientific examination and analysis. Analysis showed that two of these were ironworking slags and one was a fragment of oolitic limestone — these samples are not report here. The remaining 9 samples included 2 samples of slag and 7 of litharge.

The litharge samples all contain large proportions of lead oxide with smaller amounts of minor phases (Figs 1–2). The small size of many of these phases made their definite identification uncertain in many cases. Nevertheless, it was possible to identify several calcium lead silicates ( $\text{CaPbSiO}_4$ ,  $\text{Ca}_2\text{PbSiO}_5$  and  $\text{CaPb}_2\text{SiO}_5$ ), a lead silicate ( $\text{Pb}_3\text{SiO}_5$ ), a magnesium calcium silicate ( $\text{Mg}_{0.5}\text{Ca}_{1.5}\text{SiO}_4$ ), wollastonite ( $\text{CaSiO}_3$ ), di-calcium silicate ( $\text{Ca}_2\text{SiO}_4$ ) and an aluminium potassium silicate ( $\text{AlKSiO}_4$ ).

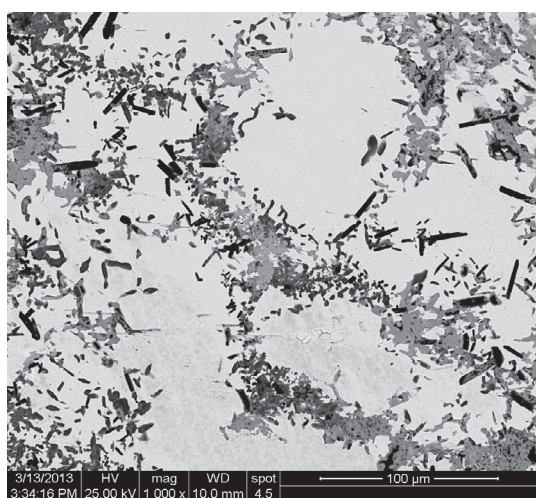


Figure 1. SEM image (backscattered detector) of litharge (sample 1) showing lead oxide matrix (light grey), calcium lead silicates (mid-dark grey) and calcium silicates (dark grey or black)

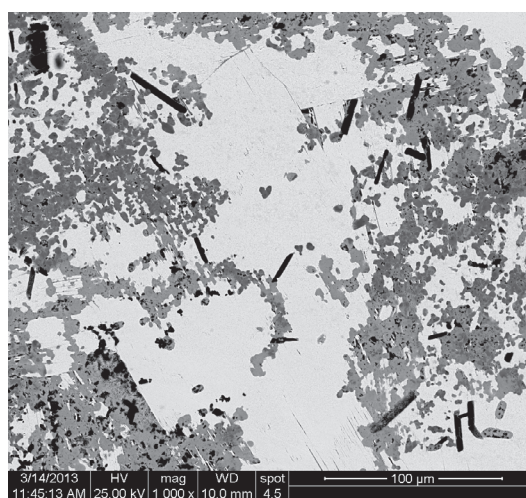


Figure 2. SEM image (backscattered detector) of litharge (sample 9) showing lead oxide matrix (light grey), calcium lead silicates (mid-dark grey) and magnesium calcium silicates (dark grey or black)

Table 2. Composition (wt%) of St Algar's Farm litharge

|    | Na <sub>2</sub> O | MgO | Al <sub>2</sub> O <sub>3</sub> | SiO <sub>2</sub> | P <sub>2</sub> O <sub>5</sub> | SO <sub>3</sub> | K <sub>2</sub> O | CaO | MnO  | Fe <sub>2</sub> O <sub>3</sub> | CuO  | PbO  |
|----|-------------------|-----|--------------------------------|------------------|-------------------------------|-----------------|------------------|-----|------|--------------------------------|------|------|
| 1  | <0.1              | 0.6 | 0.4                            | 6.5              | <0.2                          | <0.2            | 0.2              | 8.1 | <0.1 | <0.1                           | <0.1 | 84.2 |
| 3  | <0.1              | 0.3 | 0.8                            | 6.5              | <0.2                          | <0.2            | <0.1             | 6.9 | <0.1 | 0.2                            | <0.1 | 85.1 |
| 4  | <0.1              | 0.5 | 0.8                            | 7.7              | <0.2                          | <0.2            | 0.6              | 5.8 | <0.1 | <0.1                           | <0.1 | 84.7 |
| 6  | <0.1              | 0.3 | 0.3                            | 6.5              | <0.2                          | <0.2            | 0.2              | 3.1 | <0.1 | 0.2                            | <0.1 | 89.5 |
| 9  | <0.1              | 0.5 | 0.3                            | 7.0              | <0.2                          | 0.2             | <0.1             | 8.2 | <0.1 | <0.1                           | <0.1 | 83.9 |
| 10 | <0.1              | 0.3 | 1.0                            | 12.3             | <0.2                          | 0.5             | 0.3              | 4.9 | <0.1 | 0.5                            | <0.1 | 80.0 |
| 12 | <0.1              | 0.6 | 0.6                            | 7.1              | <0.2                          | <0.2            | <0.1             | 7.5 | <0.1 | <0.1                           | 1.0  | 83.3 |

The litharge is all lead rich with minor amounts of calcium and silicon (Table 2). Phosphorus was not detected in any of the samples of litharge. This suggests that bone

ash was not used to line the cupellation hearth. Copper was detected in one sample of litharge (sample 12). The absence of copper from the other litharge samples is consistent with primary cupellation rather than secondary cupellation.

The two samples of lead slag have rather different microstructures. One sample (7) has a lead-rich, glassy matrix with some wollastonite crystals (Fig 3) while the other (sample 11) is composed mostly of quartz and recrystallised silica polymorphs in a lead-rich, glass matrix (Fig 4). The microstructure of sample 11 is not consistent with a smelting slag but may be a waste product formed by reactions between slag and a silica-rich furnace wall.

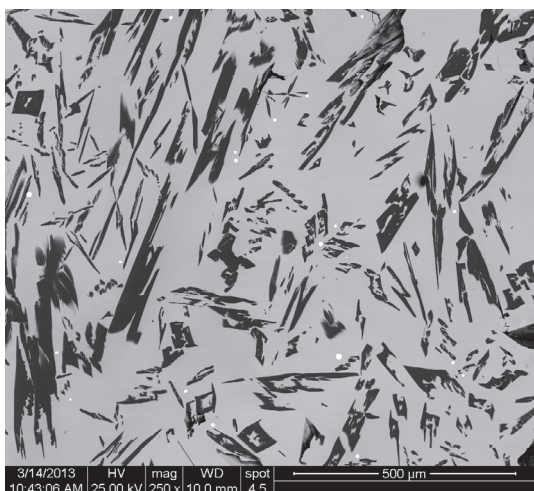


Figure 3. SEM image (backscattered detector) of lead slag (sample 7) showing lead-rich glassy matrix, wollastonite crystals (dark grey to black) and droplets of lead oxide (white)

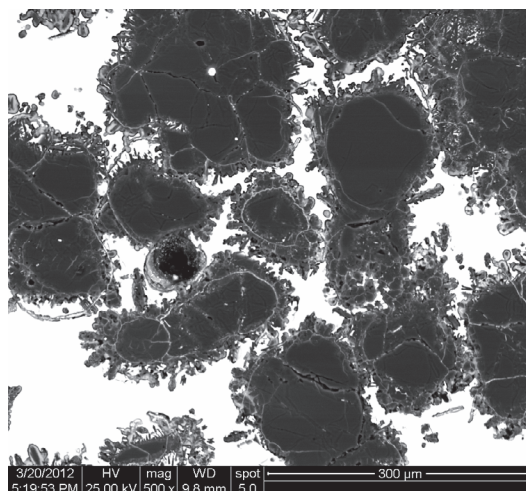


Figure 4. SEM image (backscattered detector) of lead slag (sample 11) showing lead oxide matrix (white) and silica (some relict silica surrounded by recrystallised silica polymorphs)

Table 3. Composition (wt%) of lead slags (NB the data for sample 11 represents the lead-rich matrix and excludes the relict and recrystallised silica)

|    | Na <sub>2</sub> O | MgO | Al <sub>2</sub> O <sub>3</sub> | SiO <sub>2</sub> | P <sub>2</sub> O <sub>5</sub> | SO <sub>3</sub> | K <sub>2</sub> O | CaO  | MnO | Fe <sub>2</sub> O <sub>3</sub> | CuO  | PbO  |
|----|-------------------|-----|--------------------------------|------------------|-------------------------------|-----------------|------------------|------|-----|--------------------------------|------|------|
| 7  | 0.1               | 0.8 | 2.8                            | 34.9             | 0.3                           | <0.2            | 1.1              | 14.3 | 0.2 | 2.3                            | 0.1  | 42.7 |
| 11 | 0.3               | 0.8 | 4.2                            | 48.7             | 0.8                           | <0.2            | 2.0              | 8.7  | 0.2 | 2.6                            | <0.1 | 31.4 |

The lead slags contain relatively high levels of silica and lead with a wide range of minor elements (calcium, aluminium, iron, potassium, etc, see Table 3).



## DISCUSSION

The examination of the lead-rich materials from St Algar's Farm has identified two types of waste: smelting slag and litharge. It is notable that no samples of slag were positively identified during the visual examination of the material. The scientific analysis showed that two samples were produced during the smelting of lead ores; however, these samples were very small. It is likely that lead smelting took place in the vicinity of the excavated trenches; however, it is possible that the smelting took place several hundred metres away. The assemblage of lead-working materials from St Algar's Farm, includes much more litharge and it is likely that cupellation took place either within the excavated areas or close by.

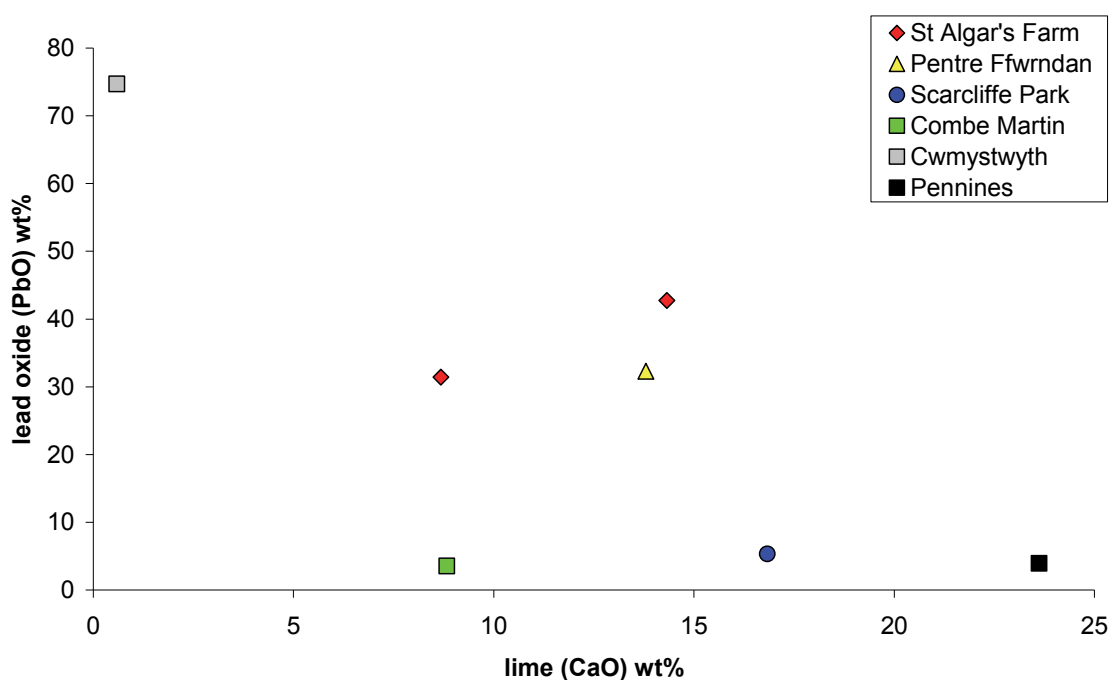


Figure 5. Lime and lead oxide content of the St Algar's Farm lead slags compared to other lead smelting slags

There are few analysed samples of Roman lead smelting slag that the St Algar's Farm can be compared with. Tylecote reports the analysis of lead smelting slag from Pentre Ffwrndan, Flintshire (Tylecote 1986, 56), Hetherington (1979) analysed slags from Scarcliffe Park, Nottinghamshire although some confusion existed over whether these are Roman (Anon 1971) or medieval (Tylecote 1986, 56), Anguilano has analysed slags of medieval date from Cwmystwyth (Anguilano *et al*/2010), Paynter has analysed early post-medieval slags from Combe Martin (Paynter *et al*/2010) and Gill analysed 18th- and 19th-century samples from various sites in the Pennines (Gill 1986).

It is clear that the St Algar's Farm lead slags have compositions which fall within the range indicated by the analysis of other lead smelting slags (Figures 5 and 6); however, the range

of compositions noted by other researchers is extremely broad (cf Smith 2006). The St Algar's Farm lead slags contain relatively high levels of lead which would suggest that the smelting process gave a relatively low yield of metallic lead. Nevertheless, the lead content of the St Algar's Farm slags is still lower than the Cwmystwyth samples, although the latter seem as high as many ore sources and leave some doubt about the implied yield of metallic lead.

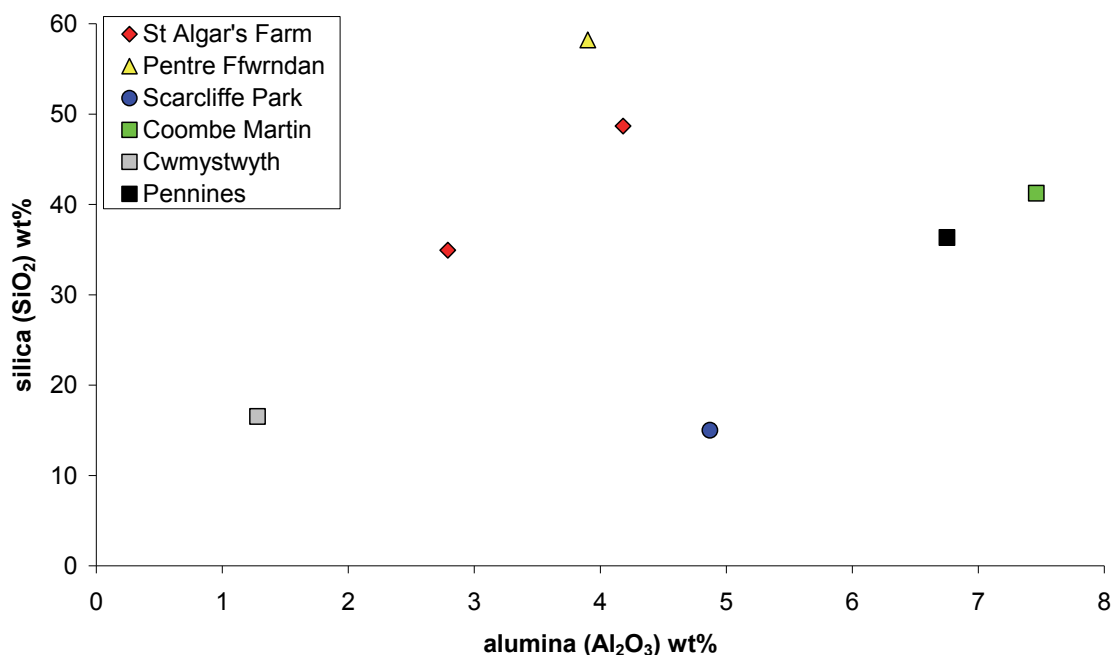


Figure 5. Alumina and silica content of the St Algar's Farm lead slags compared to other lead smelting slags

The analysed litharge samples all contain high levels of lead oxide with smaller proportions of calcium, silicon and aluminium. The absence of phosphorus from the analysed litharge suggests that the cupellation hearth was not lined with bone ash — a calcareous marl was the most likely lining. While bone ash and calcareous marls were both used to line hearths for secondary cupellation (*ie* recovery of silver from silver alloys, cf Bayley and Eckstein 1997; 2004; Girbal 2011), much less data is available for primary cupellation. To date only four UK sites with evidence for primary cupellation in the Roman period have been proposed: St Algar's Farm, Somerset (this report), Chew Valley, Somerset (Rahtz and Greenfield 1977), Green Ore, Somerset (Ashworth and Palmer 1957–1958) and Pentrehyling, Brompton, Shropshire (Bayley and Eckstein 1997). The chemical analysis of 7 samples from St Algar's Farm (Table 2) and 4 from Pentrehyling (Table 4) indicates that these all contain very low levels of phosphorus. While some phosphorus was detected in samples of litharge from Green Ore, this was described as 'of doubtful significance' and 'not above normal' (Ashworth and Palmer 1957–58, 14). The chemical composition of analysed Roman primary litharge thus conforms to later descriptions which mention only plant ashes, and especially washed wood ash (Hoover and Hoover 1950, 496), which

would be rich in calcium. The aluminium and silicon content of the primary litharge is consistent with Agricola's suggested use of washed wood ash and lute, or clay (Hoover and Hoover 1950, 496).

*Table 4. Composition (wt%) of Pentrehyling, Brompton litharge (1 = Bayley and Eckstein 1998 [nr = not reported], 2–4 the authors' unpublished data)*

|   | Na <sub>2</sub> O | MgO | Al <sub>2</sub> O <sub>3</sub> | SiO <sub>2</sub> | P <sub>2</sub> O <sub>5</sub> | K <sub>2</sub> O | CaO | Fe <sub>2</sub> O <sub>3</sub> | CuO   | PbO  |
|---|-------------------|-----|--------------------------------|------------------|-------------------------------|------------------|-----|--------------------------------|-------|------|
| 1 | nr                | 0.4 | 2.2                            | 6.9              | 0.3                           | 0.4              | 7.7 | nr                             | nr    | 82.1 |
| 2 | 0.1               | 0.5 | 2.5                            | 7.5              | <0.2                          | 1.1              | 9.3 | 0.08                           | <0.05 | 78.9 |
| 3 | 0.1               | 0.6 | 2.6                            | 6.9              | <0.2                          | 0.5              | 8.8 | 0.10                           | <0.05 | 80.3 |
| 4 | 0.1               | 0.6 | 2.3                            | 8.3              | <0.2                          | 0.4              | 7.5 | 0.24                           | <0.05 | 80.6 |

The nature of the cupellation hearth lining may be assessed by assuming that the lead content of the litharge derives from the cupelled lead, and that the balance derives from the hearth lining (clay and ash). Table 5 shows the average values for the hearth lining with lead removed and the results normalised to 100wt%. In both cases the results are consistent with the use of clay and washed wood ash. The differences in the Al:Si between St Algar's Farm and Pentrehyling probably reflect inherent differences in the available clays.

*Table 5. Estimated composition (wt%) of cupellation hearth linings*

|                 | MgO | Al <sub>2</sub> O <sub>3</sub> | SiO <sub>2</sub> | P <sub>2</sub> O <sub>5</sub> | K <sub>2</sub> O | CaO  | Fe <sub>2</sub> O <sub>3</sub> |
|-----------------|-----|--------------------------------|------------------|-------------------------------|------------------|------|--------------------------------|
| St Algar's Farm | 2.9 | 3.8                            | 49.3             | <1                            | 1.3              | 40.6 | 0.8                            |
| Pentrehyling    | 2.8 | 12.6                           | 38.8             | <1                            | 3.1              | 43.6 | 0.5                            |

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