Report on the scientific analysis of glass and glass-bearing crucibles from Sigtuna, Sweden

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Introduction

The chemical and microstructural analysis of the glass and glass-bearing artefacts excavated from Sigtuna in Sweden revealed that, as suspected, lead-silica glass was certainly worked on the site and quite possibly made there. Table 1 provides compositional results for the glass fragments, ceramic bodies and interaction layers (buffer)

102012

A fragment of glass linen smoother. The light area of glass inclusions and matrix in Figure 1 (appearing pale against the black background of the resin mount) is of a high potassium, high calcium oxide composition with elevated phosphorus pentoxide and magnesia.



Figure 1: Backscattered image of glass in 102012.

106224

This vitrified lump possibly originally deriving from a furnace and certainly from a high temperature environment contains minimal detectable lead oxide. The analysis of the body and the buffering layers shows especially elevated iron and aluminium levels, as would be predicted both in heated clay and the interaction between clay and the glassy layer covering it (see Figure 2).



Figure 2: **106224** Backscattered image of buffering layer showing the vitreous phase between crystals.

106706

This is a possible crucible fragment. No lead can be detected; the image (Figure 3) taken and chemical analysis of the buffering layer indicate that there is no evidence for the development of a vitreous layer.



Figure 3: 106706 Backscattered image of the ceramic material showing an agglomerated surface layer but no development of a glassy phase

106716

This is a crucible rim with a glassy layer on the surface. Table 1 contains the analytical results for glass, interaction layer and body. The dark greenish glass is of a lead oxide-silica composition with low alumina and calcium oxide. The interaction layer predictably contains a lower lead level and the body a minimal level.



Figure 4: Backscattered image of glassy layer over interaction layer and body of the rim of the crucible with high temperature formation of needle-like silica crystals.

106728

An amorphous lump which is partially vitrified and may be furnace lining. The glassy material in this case is a fuel ash slag of mixed-alkali (soda and potassium oxide) composition with levels of soda of up to 5.3% and potassium oxide of up to 6.51%. High alumina levels in the glass of up to 19.6% clearly indicates that this is a fuel ash slag (see Figure 5).



Figure 5: 106728 Dendrites in glassy surface of fuel-ash slag.

107528

A crucible fragment with a pale buff layer on one face and a partially-vitrified layer on the other (outer) surface. However, Figure 5 shows little, if any, development of a glassy layer and its chemical analysis reveals minimal levels of alkali in the surface layer.



Figure 5: **107528** Backscattered image of body, interaction layer and surface.

The two analyses in Table 1 are of the body and phosphorus-rich phase in the artefact. The object is probably not furnace lining because there is no detectable vitrification (Figure 7). The high phosphorus and manganese levels suggest that a degree of interaction with the soil has occurred.



Figure 7: **107697** Body and phosphorus/ manganese rich phase.

A fragment of glass crucible which has a pale (vitrified) surface layer (Figure 8). The four analyses in Table 1 are of glass, interaction layer (= buffer) and body. The bubbly glass is of a lead-silica composition; its low total is due to the fact that it is bubbly and the defocused 50 micron diameter electron beam used to analyse it is larger than the largest area of glass. Much of the glass is badly cracked. The interaction layer contains a lower lead level and none was detected in the body. The latter has the same compositional characteristics as other crucible fragments.





107963

The fragment of crucible has a yellow layer adhering to what is probably its inner face. It is clear from Table 1 that the yellow layer is a weathered lead oxide-silica glass (see Figure 9) that is badly cracked throughout.



Figure 9: **107963** Backscattered image of a thick section through 107963, a fragment of crucible

107987

This fragment of ceramic disc consists of glassy, interaction and body components. The chemical analysis of each is given in Table 1 and visible in Figure 10. The glassy phase has a lead oxide-silica composition and relatively high iron oxide; the interaction layer a lower lead oxide level and the body contains negligible levels.



Figure 10: **107987** Backscattered image of body, interaction layer, glassy surface and weathered top surface.

This a vitreous lump of bubbly fuel-ash slag with relatively high alumina levels, low alkali levels and high levels of iron oxide. It probably results from an interaction of the ash inside the furnace with the furnace wall (see Figure 11).





108034b

A buff coloured, somewhat weathered sample of glass found in the furnace. The 2 analyses in Table 1 show, again that the material has a lead oxide-silica composition. Figure 12 shows the presence of trapped gas bubbles and the development of needle-like crystals; the latter are shown at greater magnification in Figure 13.



Fugure 12: **108034b** sample of lead oxide-silica glass with weathered surfaces and many trapped gas bubbles.



Figure 13: 108034b The needle shaped crystals present in the glass.

108034a

A lump of terra cotta coloured furnace lining with a layer of translucent glass. The glass is again of a lead oxide-silica composition; 0.85% iron oxide has produced the green colour. The body has the anticipated composition for a clay. Figure 14 gives an image of both components.



Figure 14: **108034a** Backscattered image of 10803a showing ceramic and glass phases. The glass is extensively cracked due to it cooling faster than the furnace brick

108037

A crucible fragment, including rim, with a layer of dark green glass covering both inner and outer surfaces (Figure 15). Again the glass phase is of a lead oxide-silica composition, the interaction layer a lower lead oxide level of lead and the body lower still. The 'darker' area of glaze (as viewed in the backscattered detector) contains lower lead, higher iron and higher alkali levels.



Figure 15: 108037 Crucible body and glazed layer

A lump of what is described as furnace lining has the composition of clay and does not contain a vitreous phase.

108081

A glass crucible fragment with an optically dark vitreous surface layer. It has the same compositional characteristics as the other crucible fragments analysed, with a lead oxide-silica glass layer (Figure 16).



Figure 16: **108081** Body and glass layer on the crucible fragment 108081.

108128

A fragment of glass with a weathered surface. The weathered surface layer contains lower lead and silica oxides (see Figure 17). Pale grey streaks of high lead glass are intermixed with lower lead oxide matrix glass showing that the glass was a discarded waste piece (see Figure 18).



Weathered surface

Figure 17: **108128** a lump of glass with a weathered surface.



Figure 18: **108128** Heterogeneous bands of higher lead oxide glass (pale grey) running through a glass matrix containing lower lead oxide levels (darker grey).

Chemical analyses and imaging clearly show the furnace detail does not have a development of a surface glassy layer (Figure 19).



Figure 19: **109541: b**ackscattered image of fragment of furnace detail.

109544

In section this fragment of ceramic disc has body, interaction and glass phases. Many crystals have developed from solution during the heat regime employed as is clearly visible in Figure 20. The glass composition indicates that it is weathered and does not contain lead oxide, but mainly consists of silica and low calcium but negligible alkalis: it is therefore likely to be a fuel ash slag.



Figure 20: **109544** Backscattered image of a section through ceramic disc

This is the only pale green lump of raw (furnace) glass analysed. It is quite unlike the composition of the glasses on the crucible fragments. It is a potassium-lime-silica glass with impurities of phosphorus and magnesia consistent with the use of a wood ash source of alkali.

Conclusions

A number of conclusions can based on the chemical analyses and backscattered imaging of the samples provided can be reached. A number of materials which are commonly found on industrial sites, such as furnace lining and fuel-ash slags have been identified. These are formed in relatively high temperature environments. The greenish colour of the glaze found on crucible fragments is due to the presence of iron oxide, probably present in mixed reduced (ferrous) and oxidised (ferric) ionic states. Four pieces of glass were analysed. Two of these (108034b and 108128) result from working lead oxide-silica glass. The third piece of glass is what would be described as a piece of raw furnace glass of a potassium-lime-silica composition. This was therefore manufactured using very different raw materials, including wood ash, the source of the potassium oxide. It could not have been used in the manufacture of the lead oxide-silica glass. The fourth glass artefact (102102) is a glass spindle whorl. Its chemical composition is of the same type as the raw glass, but it contains c. 50% less calcium oxide so the raw glass could not have been used to make it.

It is clear that lead oxide-silica glass was worked on the site. From scientific evidence described here, and the range of artefacts supplied for examination, there is no *a priori* evidence that the glass was fused from raw materials on the site. There is nevertheless a possibility that silica and lead oxide were melted together in crucibles at a relatively low temperature of c. 700-800°C (Henderson 2000, 51). It is clear that wood ash or wood ash glass was not mixed with lead.

Lead oxide-silica glasses were first manufactured by the Muslims c. 800 AD (Sayre and Smith 1961; Henderson 2000, 50-51). The glass was used for the manufacture of glass vessels (Wedepohl et al 1995) and beads (Henderson and Warren 1986, Bayley 1990) in the west and for 'Linen smoothers' as early as the 8th century. Furthermore medieval pottery glazes are of a lead oxide-silica composition (Henderson 2000,125-126).

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