6 The Technical Characterization of Cramp
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6.1 Introduction

The purpose of this analysis was to elucidate the nature and composition of the vitrified fuel ash waste (VFAW) or cramp recovered from the site, if possible, by: a) identifying the key elements that characterize cramp; b) identifying the key ingredients of cramp, such as soil, bone, fuel and fuel ash, and in what combination they occur; c) identifying any key elements that characterise each ingredient; d) identifying how the ingredients contribute to the formation of cramp; e) identifying the conditions (eg temperature, duration of heating) which prevail in the course of cramp formation and what parameters contribute to its vitreous state and extensive porosity; f) comparing the cramp's chemical and mineralogical composition to cramp examined from other sites.

6.2 Weight, size and texture

A total amount of *c* 1348g of cramp has been collected from all contexts (including 900.5g from the topsoil over Structure 1). From the area of Structure 2, the combined contents of Cist 006 produced the largest quantity (*c* 356g), with the second largest amount retrieved from the topsoil (*c* 65g). The amount of cramp recovered from all the remaining features is a fraction of the total (Table 1).

The Loth Road cramp is predominantly dark grey in colour and consists of a light, frothy, highly siliceous vesicular material comprised of small spherules with a glassy skin. The average particle size is less than 1cm. Fill 030 in Cist 006 produced cramp of a larger size (3–4cm along the long axis). Larger cramp particles are probably formed as the result of many smaller particles having been scooped together and deposited while still hot. The cramp retrieved does not always reflect the original shape, volume or weight of the material.

All pieces, large or small, have a glassy skin covering a porous and particulate under-surface, which is in places glassy and in others only partially heated and therefore very friable. As such the contents of some bags appeared to be filled with soil, but in fact contained cramp which has never reached vitrification, in other words, soil and fuel ash. Evidence from other sites suggests that cramp will form as a multitude of spherules fused together (Photos-Jones 1999; Photos-Jones 2001; Photos-Jones 2003a; Photos-Jones 2003b). Vesicularity arises from gas evolution, either of organic matter or CO_2. Bone appears to be engulfed by the fused glass either in small fragments visible to the naked eye or micro-particles visible only with the microscope.

6.3 Analytical methods

6.3.1 ICP-OES analysis

Inductively coupled plasma optical emission spectroscopy (ICP-OES) analysis of cramp provides bulk chemical composition as well as providing elemental composition of major, minor and trace elements, pinpointing the potential fingerprint elements associated with each ingredient, such as soil and bone.

6.3.2 SEM-EDAX analysis

Scanning electron microscope analyses with energy dispersive analyser (SEM-EDAX) is based on both area analysis equivalent to bulk chemical composition and spot (single-phase) analysis. SEM-EDAX analysis was undertaken to establish the cramp composition and is particularly useful in the analysis of micro-phases, which can provide information on the mechanism of cramp formation.

6.4 Results

6.4.1 ICP-OES analysis

Four cramp samples were analysed from Structure 2, all of them retrieved as finds. One was from the topsoil whilst the remainder were from the fills of Cist 006. The soils analysed were subsamples of nine bulk samples. Five were pit fills, one a box fill, three were from the fills of Cist 069 and one from Cist 006. This was the only context – 030, the secondary fill of the cist – from which both soil and cramp samples were analysed.

Analysis shows that the cramp is an iron aluminosilicate with small amounts of calcium, magnesium, potassium and phosphorus. This is consistent with cramp from other sites, such as the Knowes of Trotty, Kewing and Crantit (Photos-Jones 2001; Photos-Jones 2003a; Photos-Jones 2003b). The similarity between the composition of the soil and the cramp samples is strong. Elements like silica, alumina and iron are instrumental in cramp formation, as well as trace elements like barium. Given these similarities, it is clear that the major component in cramp formation is the quartz-rich soil found within the pits and cists. The assimilation of bone, a calcium phosphate (hydroxyl-apatite), within the glassy matrix can contribute to cramp formation and this would be reflected in the relative concentrations of calcium, phosphorus and strontium. However, calcium and phosphorus could come from other sources, such as soil or fuel.

6.4.2 SEM-EDAX analysis

Area analyses for 'bulk' chemical compositions were carried out on the four cramp samples, confirming the results of the ICP-OES analyses. The SEM-SE images of the sample from Fill 026 in Cist 006 show a glassy matrix enveloping un-reacted or partly assimilated quartz grains; porosity is extensive and the glass is not uniform. Two of the micro-phases illustrate the behaviour of the cramp glass during cooling, one reflecting a liquid high in silica and alumina (glass), and the other high in phosphorus and iron (bone). This liquid immiscibility clearly points to the cramp having been molten as the two phases separate as the liquid cools.

The study of micro-phases can resolve a number of questions, including to what extent the phosphorus derives from the break-up of the bone or from another source or both. Plotting of molecular weights in a ternary diagram consisting of $CaO–SiO_2–P_2O_5$ (Photos-Jones *et al*, in prep) shows that the ratio of calcium to phosphorus is consistent with that of bone. However, at times there is either excess calcium or excess phosphorus, indicating that both calcium and phosphorus can be procured by different sources in addition to bone. An additional source for phosphorus could be minerals concentrating within peat and turf. SEM-SE images of a cramp sample from the Knowes of Trotty (KOT9, from context 044) point to the presence of a glassy matrix, small fragments of bone and cellular plant matter trapped in the glassy matrix, with mineral inclusions of aluminium, phosphorus and iron (Photos-Jones 2003b).

6.5 Conclusion

The results show that the key ingredient in Loth Road cramp formation is quartz-rich soil. Other materials – bone, fuel and fuel ash – are incorporated by the cramp and may also contribute to cramp formation. One component missing from the results of the analysis is fuel ash. However, we would argue that seaweed ash is the key ingredient in glass formation, because of its relatively high alkaline content, acting as a flux to lower the temperature at which the silica in the quartz or sand grains fuses or melts to form the cramp (Photos-Jones *et al*, in prep).