

| | | | |
|---------------|------|-------------|--|
| Object Number | K546 | Description | Helmet-crest channel, cast in silver and gilded, with animal ornament and animal-head terminal. Catalogue no. 589. |
|---------------|------|-------------|--|


| | |
|---|---|
|  | Sample Description and location. |
| | Sample K546-3 collected from white material indicated in images below. See Sonia O'Connor report for preliminary microscopic analysis of this material. |



Figure 1. Sample K546-3 collected from white material inside ridge of object K546.

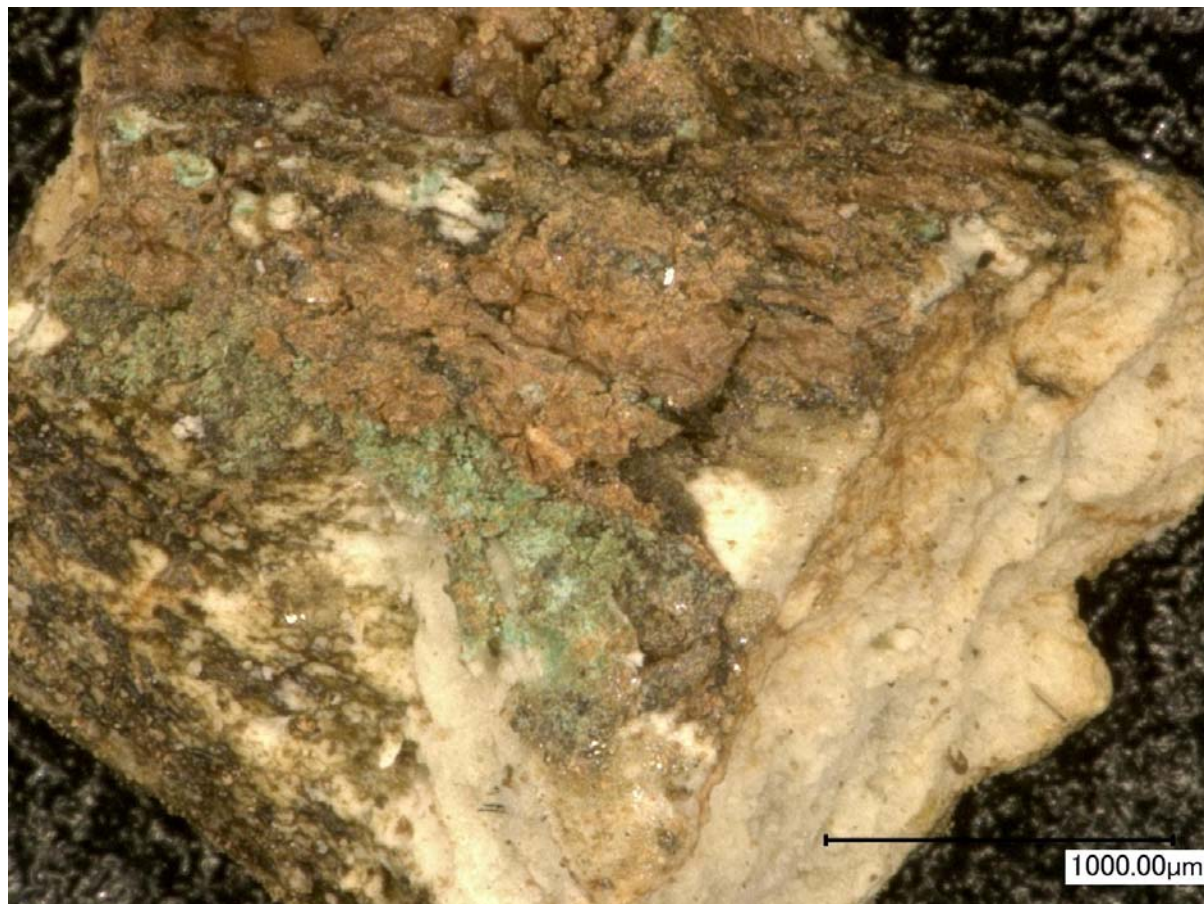


Figure 2. Detail of sample K546-3. Note trace of green coloured material on surface.

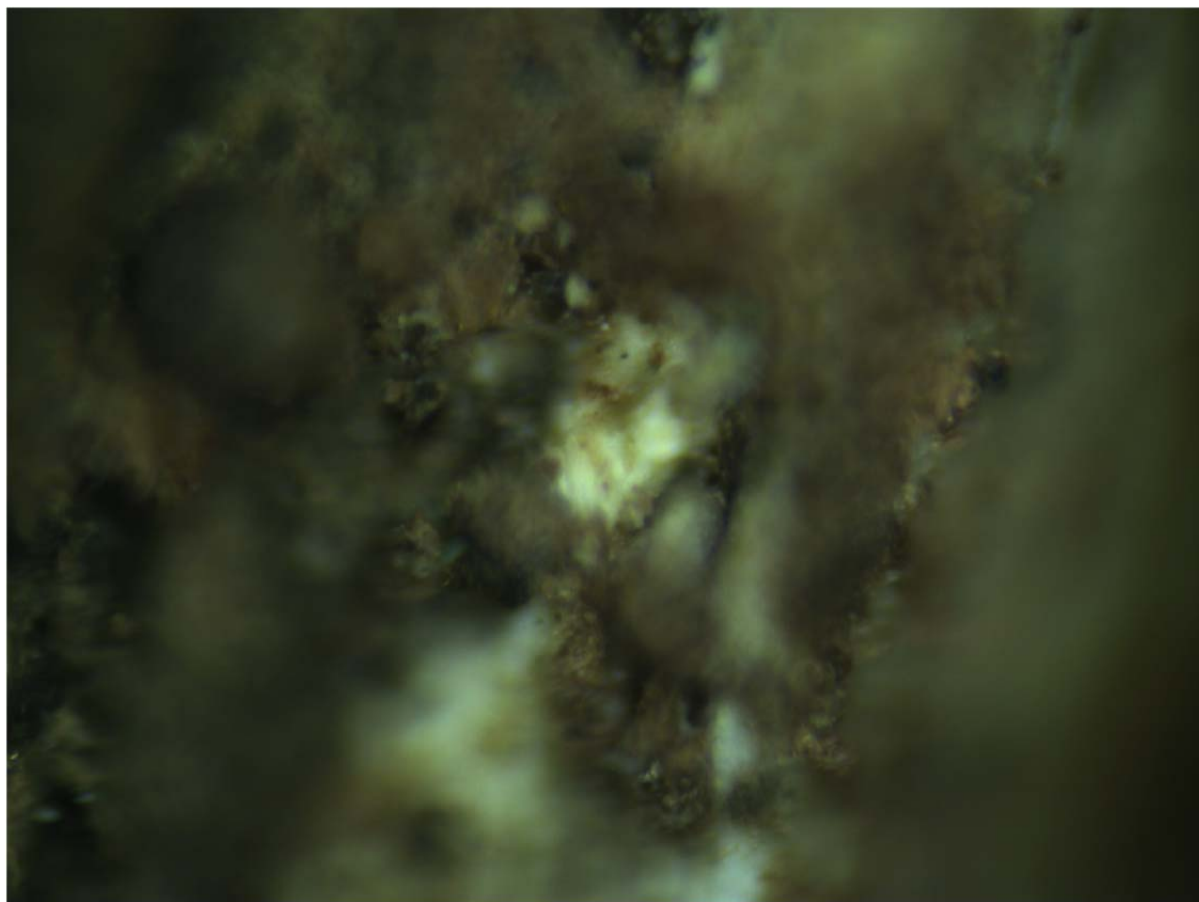


Figure 3. Detail of FTIR analysis point for sub-sample K546-3-1.

FTIR Analysis

Comments: Spectrum K546-1-3 appears to be a complex mixture of at least three raw materials-calcium carbonate, beeswax, and silicate material. Calcium carbonate shows a smooth symmetrical and broad absorption band from C-O stretching at about 1414cm^{-1} . Additional peaks at about 872cm^{-1} and 712cm^{-1} relate to C-O out of plane and in-plane bending within the carbonate ion respectively. Because few organic compounds have strong absorptions in this region, these sharp bands are very useful for confirmation and identification of carbonates in a spectrum (Derrick et al. 1999, Clark 1999, Trinkūnaitė-Felsen et al. 2012, Adler & Kerr, 1963). Splitting of the two lower wavenumber peaks indicates the presence of the aragonite crystalline phase in the calcium carbonate sample, whereas non-splitting indicates the calcite phase (Trinkūnaitė-Felsen et al. 2012). As aragonite transitions to the calcite crystalline phase at elevated temperatures (Balmain et al. 1999, Oates 1998) or over time in the presence of water (Oates 1998), the aragonite crystalline phase is not normally associated with heat processed calcium carbonate materials such as quick lime, lime putty or milk of lime.

The FTIR spectrum for beeswax is characterized by dominant absorption bands around 2950cm^{-1} and 2850cm^{-1} that relate to C-H stretching of the methylene (CH_2) groups, a band at 1740cm^{-1} that relates to C=O bonding characteristic of the ester groups, a band around 1460cm^{-1} relates to C-H bending, and a doublet between 720 and 730cm^{-1} relating to non planar skeletal deformation vibrations of long chain hydrocarbons (Derrick *et al* 1999, Birshtein and Tul'chinskii 1977). The bands in the region $1350 - 1180 \text{cm}^{-1}$ may be assigned to a phenomenon known as a 'band progression' present in fatty acids and fatty acid esters which result in a series of evenly spaced bands in this region. These are due to wagging and twisting vibrations of successive carboxyl coupled methylene groups (Baeten et al. 2010).

Generally speaking, silicates are characterized by a region of strong absorption near 1000cm^{-1} due to Si-O stretching, and a second region of absorption near 500cm^{-1} arising from Si-O bending, often in combination with other cation-oxygen vibrations (Djomgoue & Njopwouo 2013). The silicates in this case may be from the burial environment, or may have been deliberately added to what may be a lime plaster mixture. This sample may be a lime plaster and beeswax mixture.

Representative Spectrum

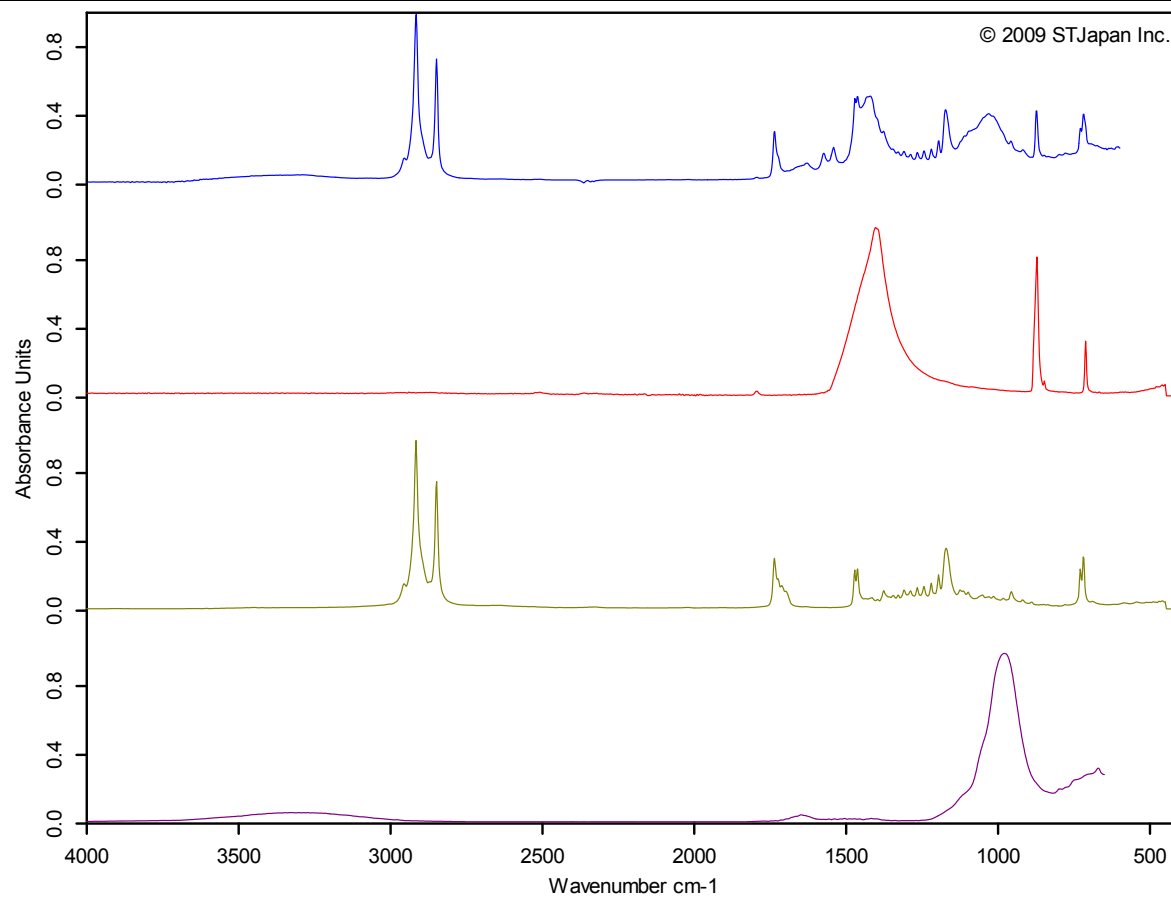


Figure 4. Top (blue) K546-3-1. Upper middle (red) Calcium Carbonate reference spectrum, ST Japan 2009. Lower middle (green) Beeswax reference sample, ST Japan 2009. Bottom (purple) Reference sample Aluminium Silicate, ST Japan 2009.

SEM Analysis

Sample K546-3 was analysed using SEM to examine the surface structure and elementally characterize the material. The surface texture appears consistent with the FTIR findings for this material with particulate distributed through the beeswax.

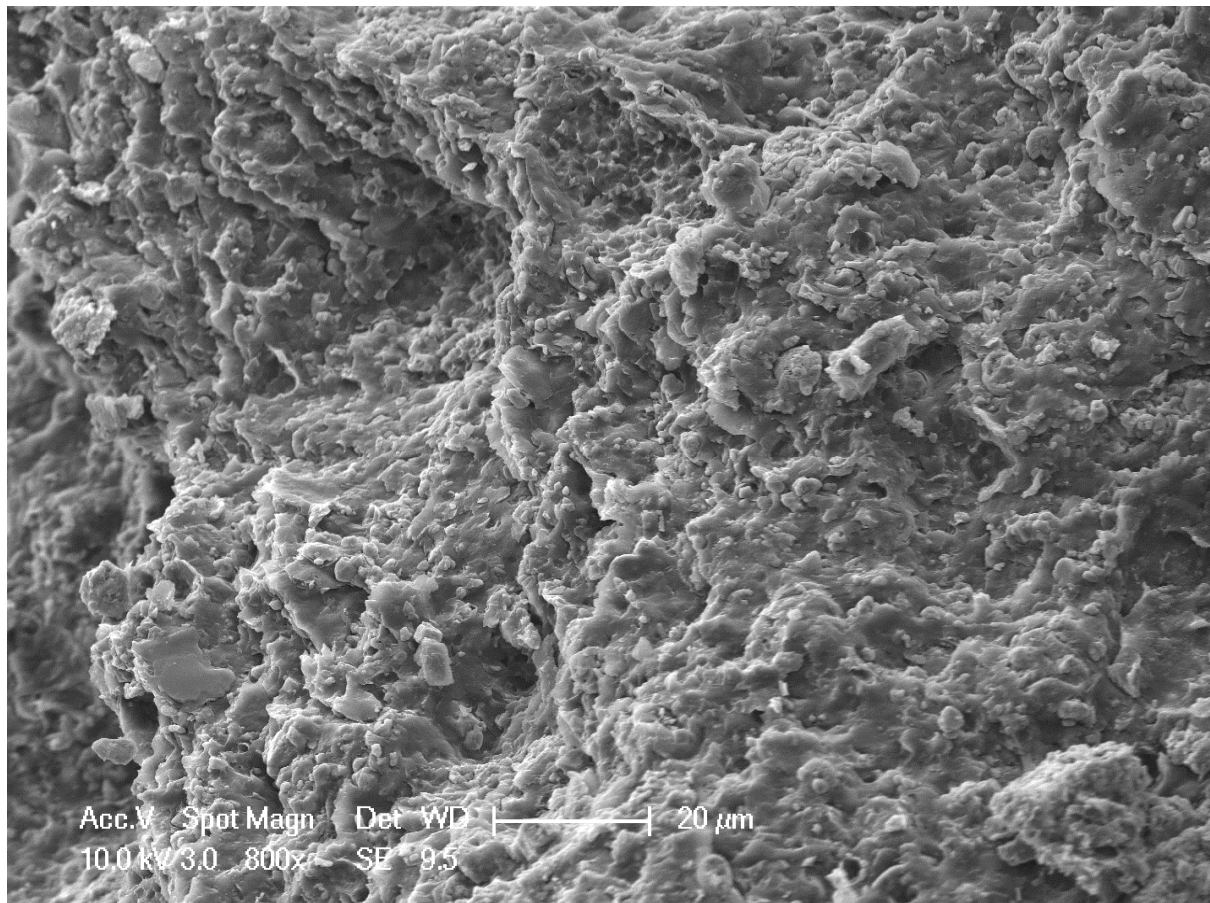


Figure 6. Secondary electron image of K546-3 showing texture of white material collected from helmet crest channel. The sample appears waxy with particulate distributed across the surface.

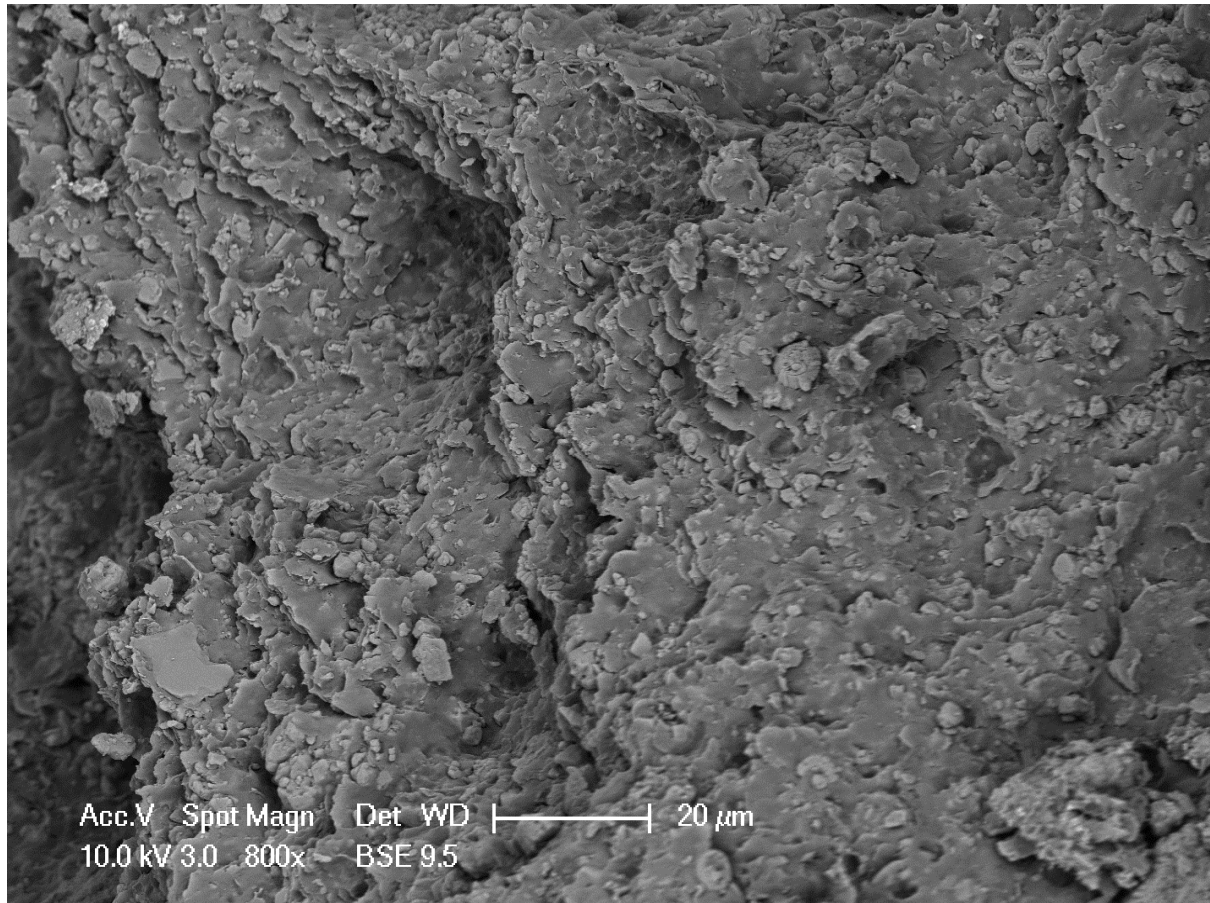


Figure 6. Back scatter electron image of K546-3 showing texture of white material collected from helmet crest channel. The sample appears elementally homogeneous.