


Object Number	K359	Description	Pommel in gold, of cocked-hat form, with filigree decoration. Catalogue no. 10.
		Sample Description and location.	
		Sample K359-1 collected from white material inside pommel cap.	



Figure 1. Image showing sample collection point for K358-1. Sample collected from white material inside pommel cap.

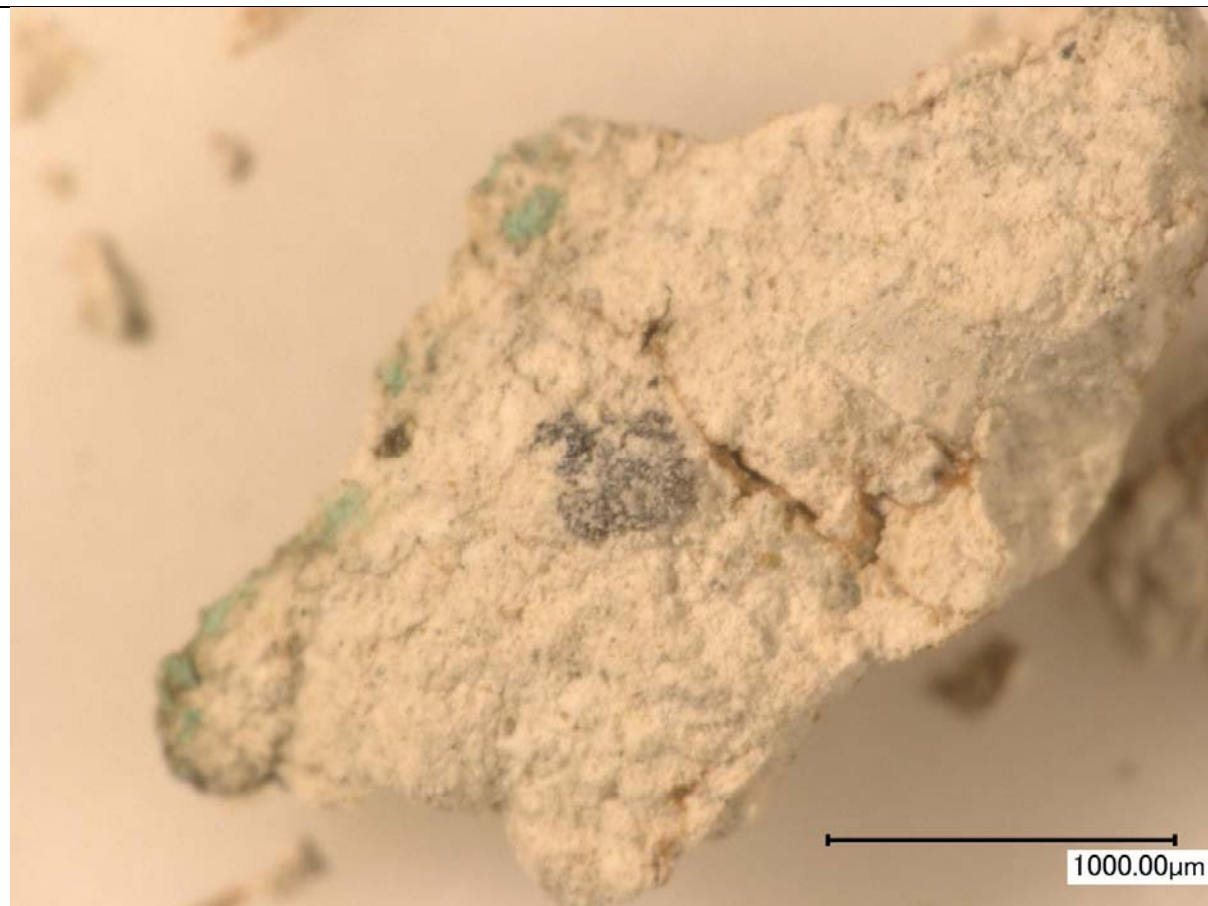


Figure 2. Detail of sample K359-1.

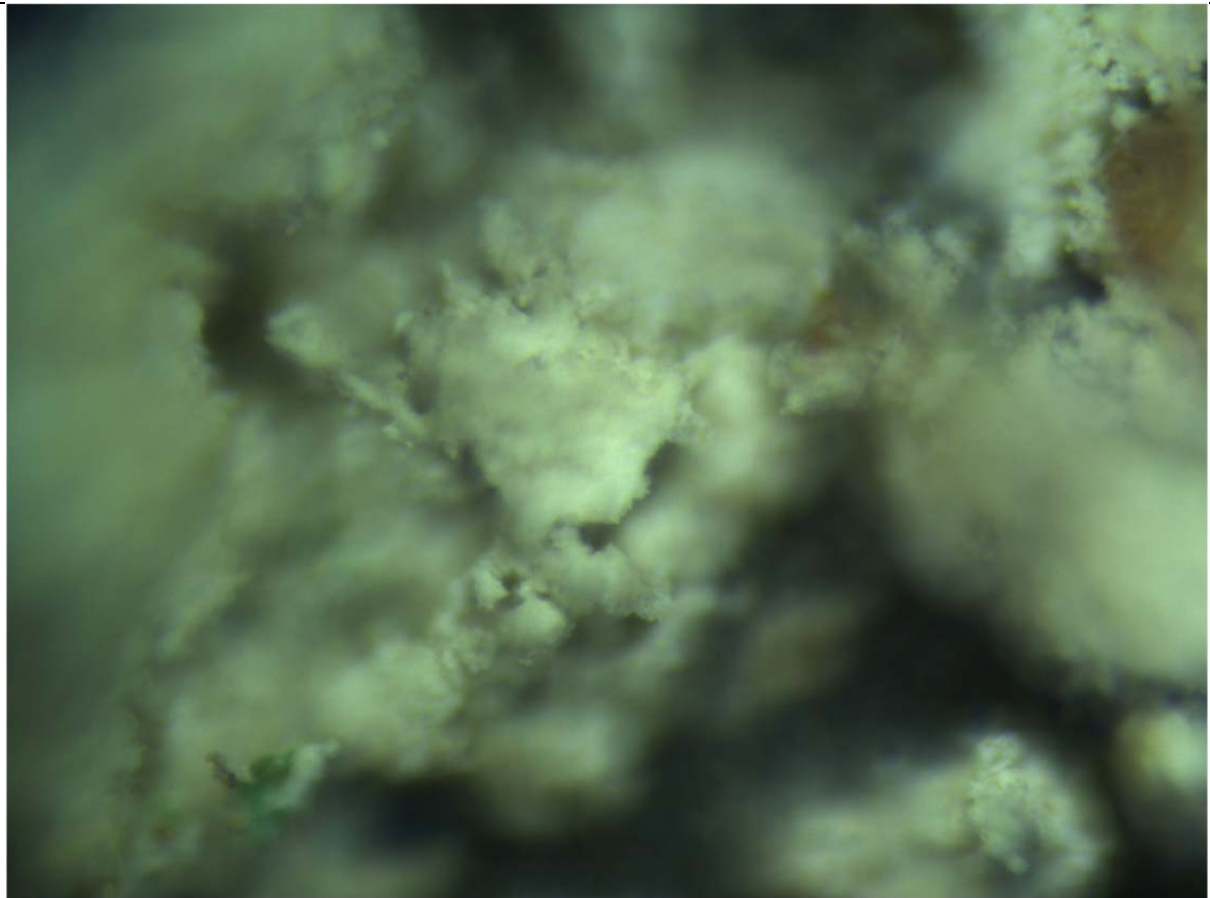


Figure 3. Detail of sample K359-1-2.

## FTIR Analysis

Comments: Spectrum K359-1-2 (top, blue) is a close spectra match for calcium carbonate based lime plaster (bottom red reference spectrum). Calcium carbonate is a complex inorganic compound that contains a calcium ( $\text{Ca}^{2+}$ ) cation, and a carbonate ( $\text{CO}_3^{-2}$ ) anion. Whereas most simple anionic compounds do not produce any vibrations in the mid-IR range, the carbonate ion is a covalently bonded functional group in itself, and undergoes internal molecular vibrations that occur within the mid-IR range. The attached cation has a slight impact on the position of the absorption bands for the complex anion, with heavier cations shifting the bands to a lower frequency, particularly at lower wavenumbers (Derrick et al 1999). Peak shifts to slightly higher wavenumbers than those known for calcium carbonate may indicate therefore the presence of magnesium carbonate within a sample.

Calcium carbonate shows a smooth symmetrical and broad absorption band from C-O stretching at about  $1414\text{cm}^{-1}$ . Additional peaks at about  $872\text{cm}^{-1}$  and  $712\text{cm}^{-1}$  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.

## Representative Spectrum

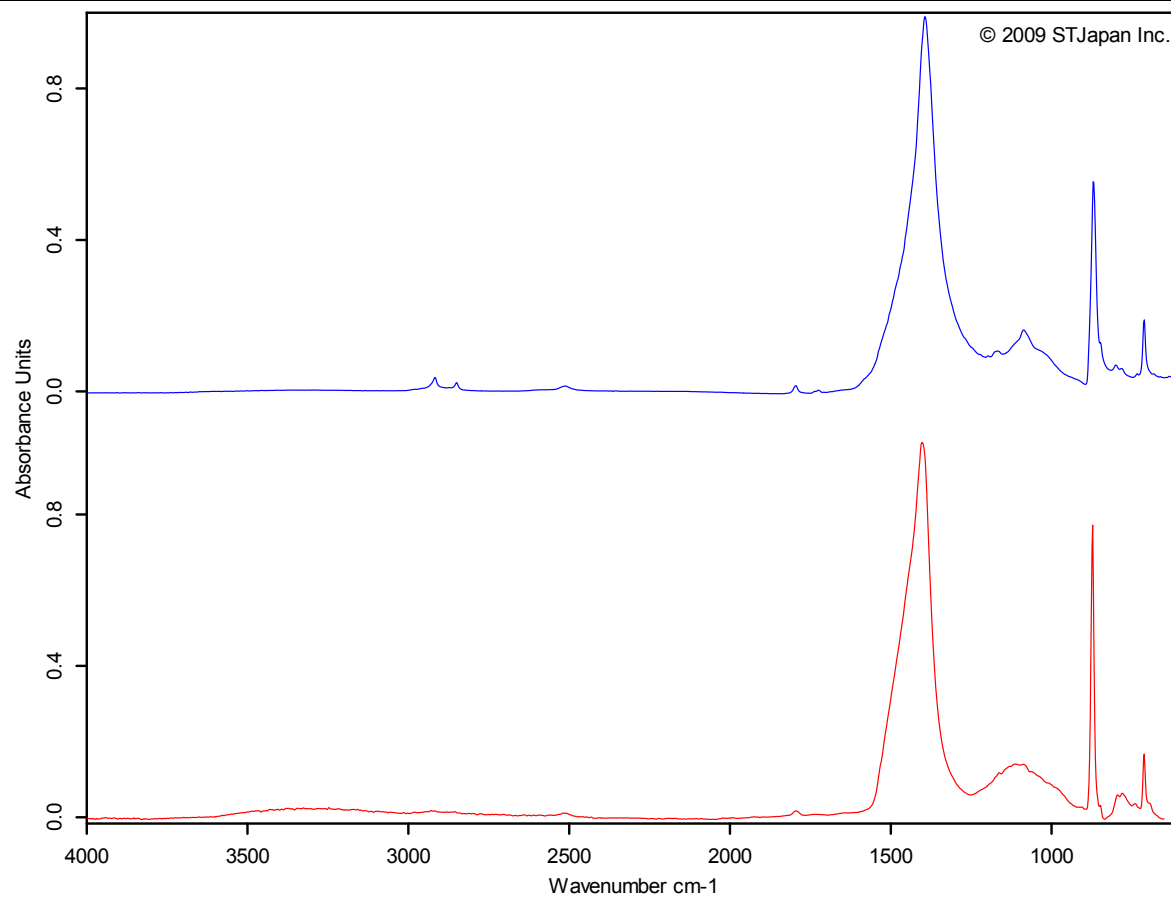


Figure 4: Top (blue) K359-1-2. Bottom (red) Lime Plaster MVJ-1 Extra reference spectrum, ST Japan 2009.



## SEM Analysis

SEM EDS analysis was carried out to determine the elemental composition of sample K359-1. Figures 5 and 6 indicate a regularly sized particulate consistent with calcium carbonate that has undergone lime processing, with no micro fossils obvious across the sample. EDS analysis indicates that this is a very pure form of calcium carbonate with calcium and oxygen distributed throughout the sample, and only a small localised distribution of silicon evident within the site of interest as shown in the elemental maps below.

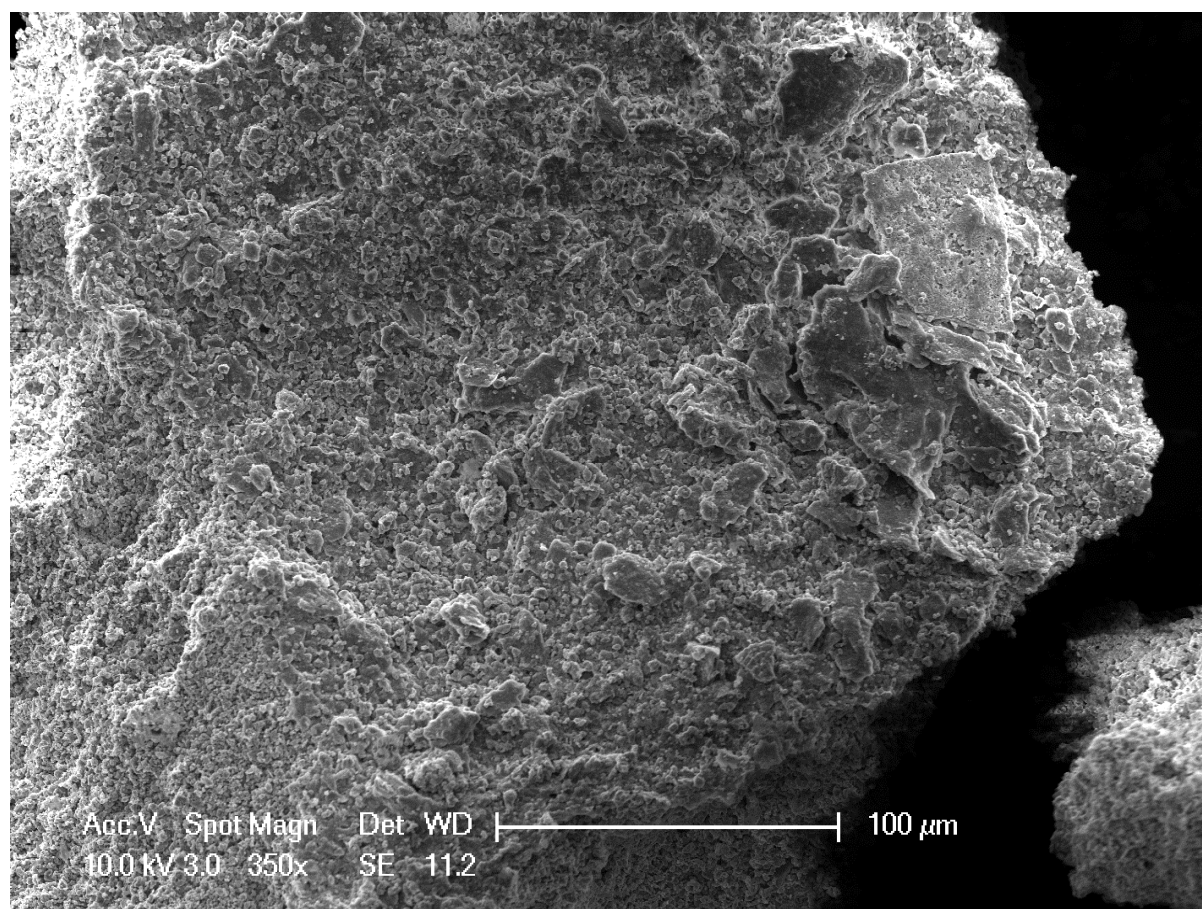


Figure 5. Secondary electron detail showing particulate texture of K359-1.

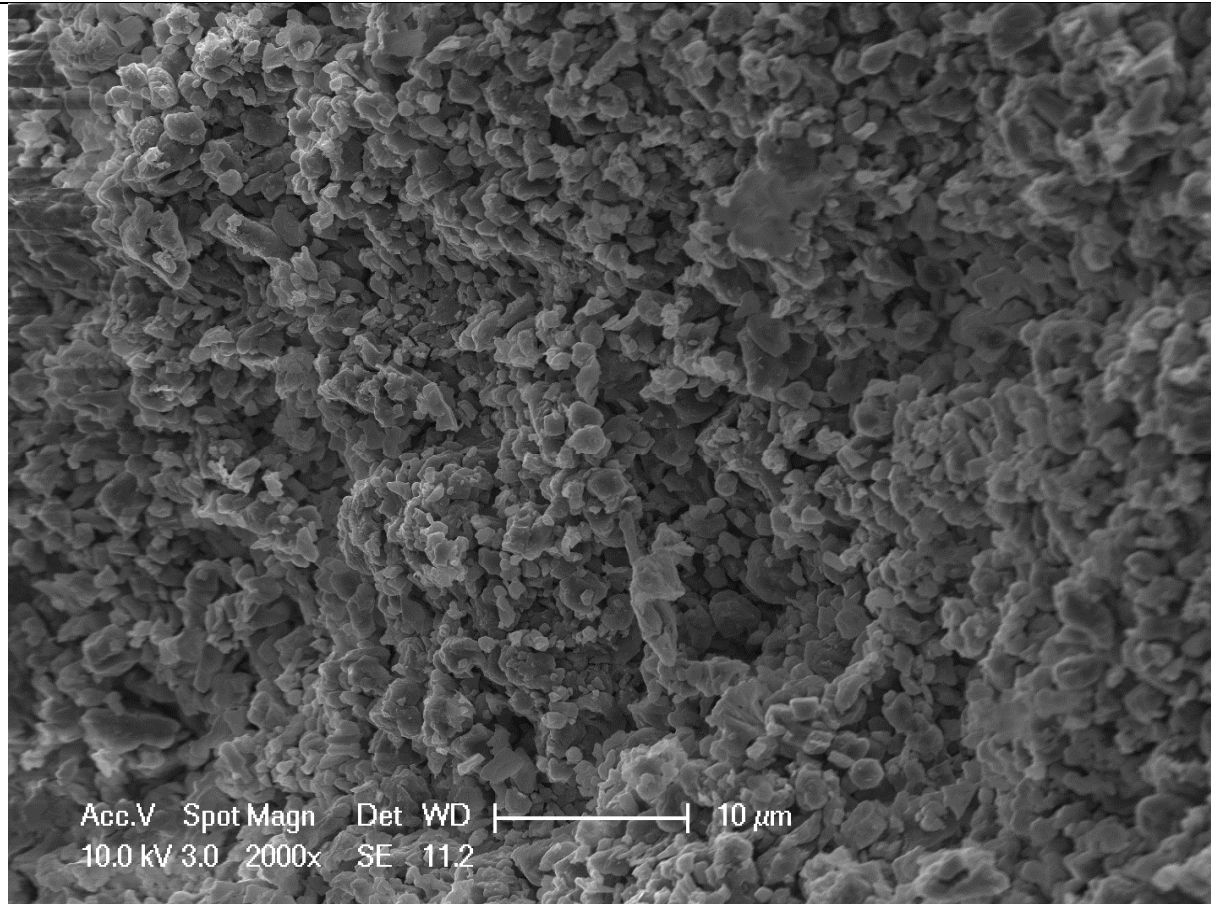


Figure 6. High magnification secondary electron image showing regular particle size of material.

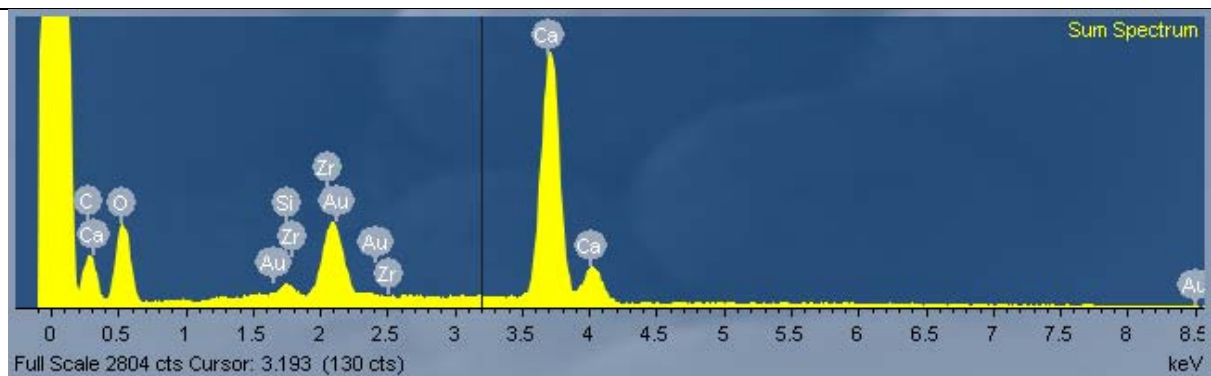
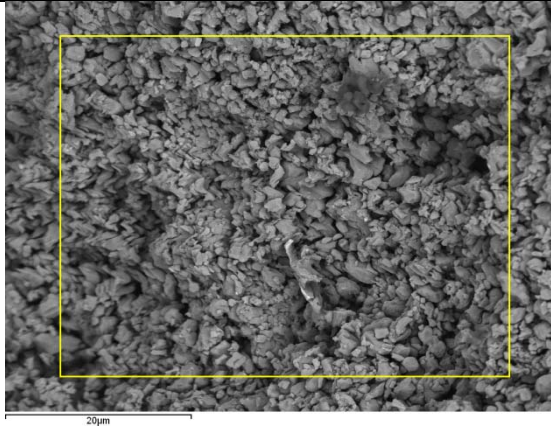
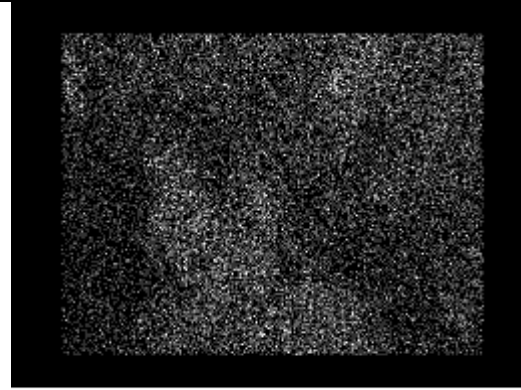


Figure 7. Sum spectrum for K359-1 SOI 2.

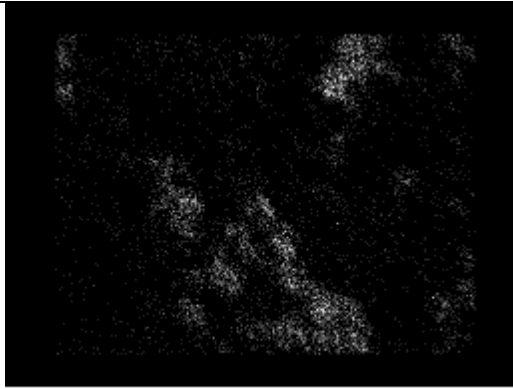




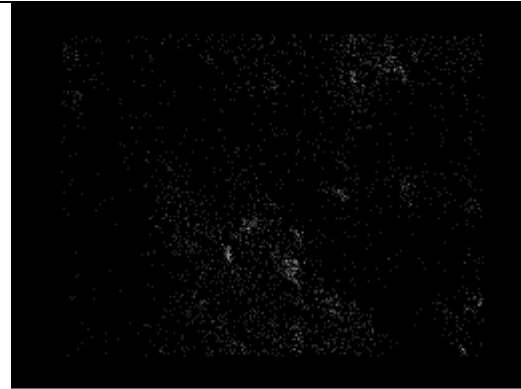
K359-1 SOI 2 Electron Image



Ca Ka1



O Ka1



Si Ka1