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Examination of Historic Window Lead

David Dungworth and Roger Wilkes

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Summary

The lead (cames) used in some historic windows to fix panels of glass (e.g. stained glass windows) needs periodic replacement. While the lead used in windows before the 19th century may last for many hundreds of years, there are doubts about the durability of lead used since the 19th century. Fifty-one samples of lead cames were analysed using EDXRF. Tin was detected in approximately half of the samples (probably accidentally introduced during the recycling of old cames along with soldered joints) and older lead tends to contain higher tin levels. The microstructures were examined but these provided little information.

Keywords

Lead

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Introduction

Historic windows often consist of numerous panes of glass joined together with lead strips (I or H shaped in section) called cames. Lead cames are usually stable and can remain in place for several centuries, however, there are now some doubts about the durability of lead used since the late 19th century (Naylor 1996, Mason 1998). English Heritage commitments to grant-aiding major re-leading and repairs programmes led to the Architectural Conservation Team setting up a research project to investigate the nature of historic lead cames.

Aims

As part of the wider Architectural Conservation Team research into lead cames and their failures, this report aims to investigate the metallurgy of lead cames of the post medieval period. This report describes the techniques used at the Centre for Archaeology to investigate samples of lead cames provided by David Mason.
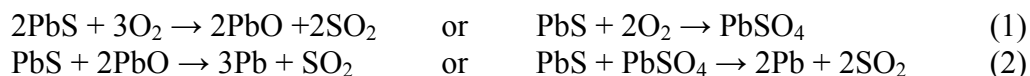
Objectives

The two objectives of this research were

- to determine the range of chemical compositions of post-medieval lead cames
- to determine if samples could be prepared in such a way as to reveal their microstructure

Production of Lead

The smelting of lead is a relatively straight-forward process which can be accomplished at fairly low temperatures. The commonest lead ore (the sulphide, galena) can be transformed to metallic lead simply by heating in air. The reason for this is that simple heating produces lead sulphates or oxides (1), which then reduce the remaining sulphide to produce metallic lead (2).

$$2PbS + 3O_2 \rightarrow 2PbO + 2SO_2 \quad \text{or} \quad PbS + 2O_2 \rightarrow PbSO_4 \qquad (1)$$
$$PbS + 2PbO \rightarrow 3Pb + SO_2 \quad \text{or} \quad PbS + PbSO_4 \rightarrow 2Pb + 2SO_2 \qquad (2)$$

Most lead ores contain small amounts of impurities (e.g. silver, copper and antimony) that may be incorporated into the smelted metal (Tylecote 1986: 71). The smelting and refining techniques used prior to the Industrial Revolution would have left many of these impurities in the lead (e.g. 0.001–0.04% Ag; 0.01–0.1% Cu; 0.005–0.2% Sb). Silver, however, would have often been removed by cupellation: the lead would be melted and oxidised to litharge (PbO), leaving a small pool of silver (Tylecote 1986: 58–61). During the early 19th century there were improvements to the refining of lead (the Pattinson and Parkes processes) that removed most of the impurities (Tylecote 1992: 156–159).

Production of Lead Came

Lead cames have been produced using three different techniques (Brown 1928):
- casting (common in medieval times but not used now)
- milling (used from the 16th century)
- extruding (20th century)

These different forming techniques are likely to produce different microstructures which may affect the physical properties of the lead cames (see below).

Properties of Lead and Lead-rich Alloys

Lead is unique among common metals. It has little mechanical strength, virtually no elasticity and is extremely soft. If stresses are applied to pure lead, it recrystalises (even at ambient temperatures), and so cannot be work hardened in the way that many other metals can be. Even relatively small stresses are continuously relieved within the metal and this gives rise to creep. In practice this may not be of great importance in short term applications, but if the situation is permitted indefinitely, the lead will fracture due to fatigue or creep. Fatigue is failure under fluctuating stress while creep is failure under low stress but over a long time. However, the softness and lack of elasticity of pure lead make it difficult to determine such physical properties,

> 'The mechanical properties of lead and its alloys, particularly the more dilute alloys, are extremely sensitive to variations in composition, grain size, metallurgical history and temperature and the rate of testing. They are therefore rarely reproducible with any degree of accuracy, except on the same sample under identical test conditions and, even then, a delay of a few hours between tests may affect the results obtained.'
> Brandes 1983: 22-49

Lead is often alloyed with very small amounts of other elements (e.g. silver, copper, antimony, tin, cadmium, arsenic and tellurium) which may have a marked effect on its physical properties (see Table 1).

Table 1: (data from Brandes 1983, table 22.16)

	Pb%	Cu%	Ag%	Te%	HV	Fatigue Strength (Nmm^{-2})		
Pure lead	99.99+				4	2.24	to	3.17
Copper lead		0.05–0.07			4.5	4.69	to	4.96
Silver copper lead		0.003–0.005	0.003–0.005		5	3.45	to	4.48
Tellurium copper lead		0.05–0.07		0.02–0.05	6	6.55	to	7.24

That alloyed lead has better fatigue strength than pure lead has been recognised as being important by those who work with lead cames (e.g. Sloan 1988) and similar products. Telephone cable sheaths were often manufactured of lead-rich alloys and, in service, were subjected to 'the action of the wind and to repeated bending caused by daily and seasonal changes in temperature' (Townsend & Greenall 1930: 395). Extensive testing of pure lead and lead-rich alloys (e.g. Beckinsale & Waterhouse 1928) established that alloys containing several percent of tin had far greater fatigue strengths than pure lead. Some additions, however, may raise the recrystallisation temperature above ambient temperatures and so allow work hardening, e.g. 0.05% tellurium (Singleton & Jones 1933).

Lead produced before the early 19th century routinely contained impurities that would naturally confer on the metal improved fatigue strength, while that produced since is likely to be very pure and so weaker (Sloan 1988).

Lead Came Samples

In partnership with the British Society of Master Glass Painters, English Heritage has amassed over 150 samples of lead cames from buildings throughout England, dating from the 16th to the 20th centuries. Representative samples were selected giving a broad date range. Small slices from each of these were cut from the came and mounted in acrylic resin to show cross-sections. Samples were chemically analysed and polished to reveal their microstructure.

Chemical analysis (SEM-EDS)

The analytical procedure employed was energy dispersive X-ray fluorescence. The spectrometer is an EDAX Eagle II fitted with a 300micron glass capillary for focussing the primary X-ray beam and a lithium drifted silicon detector for counting and measuring the fluorescent X-rays. Each spectrum was collected at 35kV and 100mA for 200 seconds (livetime). Two lead standards (MBH Analytical Ltd CRM, Lead with impurities, PR7 and PR8) were also analysed to ensure that the results were accurate. The analytical technique used proved to have relatively high detection limits for many elements (e.g. 0.5wt% for silver, cadmium, antimony and bismuth). Indeed no silver, copper, cadmium, bismuth, tellurium, antimony or indium were detected in any of the samples. This does not mean that the samples did not contain these elements, but if these elements were present they were below the detection limits. This is regrettable as very small additions of some elements (discussed above) may dramatically alter the physical properties of lead and have been detected by other researchers using different analytical techniques (e.g. Cuzange & Texier 2000).

The only element (other than lead) that was routinely detected in the lead cames was tin (see appendix). Tin was detected (i.e. 0.5% tin or more) in over half of the cames, and the maximum tin content is 4.6% (cf. Cuzange & Texier 2000: 47). Older lead cames tend to contain more tin than recent came (see table 2).

Table 2: Tin content of lead cames (for the calculation of the mean values, samples with no detected tin were treated as if tin=0%)

Date	Mean	Standard Deviation
17th century	1.33	±0.53
18th century	1.20	±0.94
19th century	0.49	±1.02
20th century	0.25	±0.34

It seems likely that the tin was not deliberately added to the lead in order to make an alloy (*contra* Brown 1928) but was accidentally introduced when old lead came, complete with solder which would contain tin, was melted down (Sloan 1988: 24). The decline in the tin content of lead cames from the 17th to the 20th century appears to show that the practice of using old lead cames as a source of lead has also declined.

In some cases the leads from the same building, the same window, apparently installed at the same date, varied considerably in tin content. For instance, out of nine samples (all believed to be late 19th century in date) taken from St. Mary & St. Hugh, Old Harlow, six contained no

detectable lead (i.e. <0.5%) while the other three contained 0.7, 1.5 and 4.6% tin. Not all of the lead cames from this building, however, are of the same size and shape: the three examples with some tin detected have heart thicknesses of 0.6–1.0mm, while those with no tin detected have heart thicknesses of 1.3–2.1mm. In general, the 17th and 18th century lead cames analysed have smaller heart thicknesses (~0.9mm) than more recent lead cames (~1.3mm). It is possible that not all of these lead cames were installed at the same time, although modern glaziers may use cames of different profiles in the same window to accommodate glass of different thicknesses or to emphasise the design.

Microstructure

The microstructure of metals can provide a range of information about the ways in which it has been formed and used. Samples are usually embedded in a resin and ground and polished to a mirror finish and then etched to reveal the microstructure. Typical features of microstructure that can be examined in this way are the boundaries between individual grains of metal, the different metallic phases that might be present, cored dendrites (indicative of casting), strain lines (indicative of cold working) and annealing twins (indicative of annealing). Not all metals and alloys will contain all of these microstructural features and lead is likely to show very few (indeed the usefulness of the metallographic examination of lead has been questioned, Vander Voort 1993: 134). The only features that may be present are the boundaries between grains and annealing twins. Lead does not form significant solid solutions with other elements and so cored dendrites are not formed, while the low recrystallisation temperature of lead means that strain lines do not survive beyond a few hours or days.

Cuzange & Texier (2000: 48) found that medieval cast lead cames had large columnar grains (perpendicular to the length of the cames, showing that it had not been mechanically worked), while 17th and 18th century milled cames had smaller recrystallised grains. Jones (1937) found that grain size was generally small in rolled lead and large in extruded lead. On the whole a smaller grain size confers greater strength to the lead (Jones 1937: 188). Jones also investigated the effects of strain (including simply rough handling) on extruded and rolled lead. Small stresses on rolled lead (i.e. 1–2% elongation) gave rise to grain growth (i.e. large grains increase in size at the expense of the small grains), while larger stresses (i.e. 5% elongation) gave rise to recrystallisation that produced a microstructure similar to that seen in the original rolled lead. Any stresses on extruded lead gave rise to recrystallisation which could (5% elongation or more) produce a microstructure similar to that seen in rolled lead. The strength of a metal is inversely proportional to grain size and where excessive grain growth is allowed to occur the metal may fail due to intercrystalline cracking (Jones 1937: 194).

The preparation of lead samples by conventional methods (grinding and polishing using silicon carbide abrasive papers and diamond suspensions) has limited success because of the softness of the metal. During grinding and polishing abrasive particles become embedded in the surface and may obscure the microstructure. More importantly, however, the polishing itself will disturb the surface of the metal and produce a surface with fine recrystallised grains that may bear little relationship to the true microstructure. A variety of sample preparation techniques were tried, including conventional polishing and chemical polishing (Jones 1933; Slepian & Blann 1979; Vander Voort 1999; Vilella & Beregekoff 1927). Different chemical polishing solutions were experimented with (e.g. ammonium molybdate, glacial acetic acid, nitric acid, hydrogen peroxide). Chemical polishing using ammonium molybdate solution

followed by etching with glacial acetic and nitric solutions were found to be successful (figure 1).
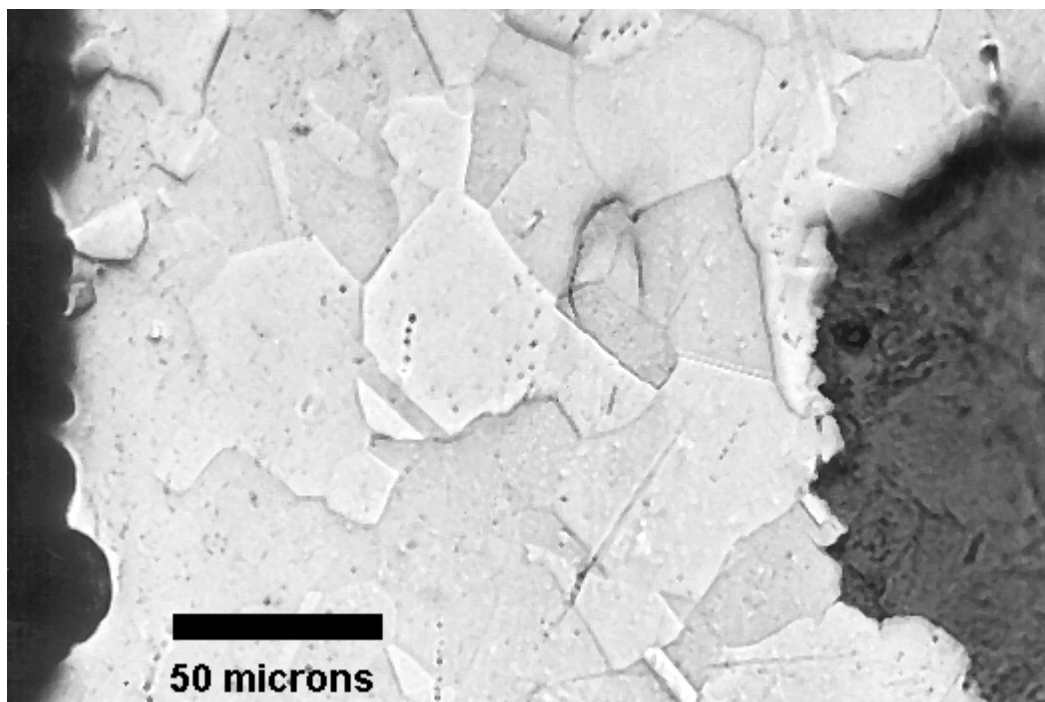

Figure 1. Photomicrograph of late 19th century lead came (sample ID number 14)

The revealed the grain structure and showed that most had fairly large grain sizes (ASTM[G] = 4–5) and occasional annealing twins. The large grain size could be the result of production by extruding (only possible for 20th century examples) or the result of grain growth arising from small amounts of stress (during transport, fitting, service or removal). This microstructure was seen in examples of 20th century lead came that may have been produced by extruding as well as pre-20th century examples that were milled. It was also seen in samples that were known to have failed in service as well as those that had not. It is possible that all of the samples had been subjected to small amounts of stress during service and that this had produced grain growth. Cames are usually straightened before use by clamping one end and tugging on the other. This straightening is unlikely to have caused significant recrystallisation and certainly will not have produced any grain growth.

Discussion

The 51 samples of lead cames investigated have been analysed using EDXRF to determine their chemical composition and polished and etched to reveal their microstructures.

EDXRF proved unable to detect small amounts of many elements of particular interest (e.g. copper and silver). Previous work has established that pre-19th century lead cames frequently contained small amounts (i.e. 0.01–0.1%) of copper and silver but that from the 19th century these elements were largely removed from lead. These elements may have conferred beneficial properties on the lead, such as improved fatigue strength. Alternative analytical techniques, such as Inductively Coupled Plasma Spectroscopy, would be able to detect the small amounts of these elements that may be present.

The technique used to analyse the lead came (EDEXRF) was able to detect tin in many of the samples and it has been shown that the tin content has declined from the 17th to the 20th

century. The tin was probably an unconscious addition in scrap lead cames and presumably the practice of melting down old lead has declined. Work in the early 20th century on lead aerial sheaths showed that lead containing a few percent of tin had greater fatigue strength than pure lead. It is possible that the perceived decline in the quality of lead cames (in particular the fatigue strength) has been caused by the increased use of pure lead in the 19th and 20th centuries.

Despite the significant technical difficulties it proved possible to obtain photomicrographs of the microstructure of lead cames. The results showed no significant differences in grain size between samples.

Recommendations

The chemical analysis of the lead cames has shown that tin is frequently present at levels that would have a marked effect on the physical properties of the lead. The technique used (EDXRF) was not able to detect small amounts of tin or other metals (e.g. silver and copper at 0.001–0.1%) that may also have affected the physical properties. It is recommended that the samples be analysed using ICPS (approximately £10 per sample) which would detect these elements down to 0.0001%.

Conclusion

The chemical composition of lead used in the manufacture of lead came is likely to be a significant factor in determining its fatigue strength and so the length of time that it is likely to remain in service before failing. However, even those who devoted a great deal of time to the problem were not always able to offer definitive answers on the subject,

> 'Even with our increased metallurgical knowledge, failures in lead . . . are by no means infrequent . . . [and] a very large proportion go unsolved'
> W. Singleton, in Jones (1937: 199)

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Appendix
EDXRF analysis of lead came

ID	Building	Period of Lead	Ni	Cu	Zn	As	Ag	Cd	In	Sn	Sb	Bi
1	St. Leonard's Hythe, Kent	1918-2000	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.6	<0.5	<0.5
2	St. Leonard's Hythe, Kent	1918-2000	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.4	<0.5	<0.5
11	St. Mary & St. Hugh, Old Harlow	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
12	St. Mary & St. Hugh, Old Harlow	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.5	<0.5	<0.5
13	St. Mary & St. Hugh, Old Harlow	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	4.6	<0.5	<0.5
14	St. Mary & St. Hugh, Old Harlow	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
15	St. Mary & St. Hugh, Old Harlow	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.7	<0.5	<0.5
16	St. Mary & St. Hugh, Old Harlow	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
17	St. Mary & St. Hugh, Old Harlow	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
18	St. Mary & St. Hugh, Old Harlow	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
19	St. Mary & St. Hugh, Old Harlow	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
20	Ante Chapel, New College, Oxford	1918-2000	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.7	<0.5	<0.5
21	Ante Chapel, New College, Oxford	1918-2000	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
22	Ante Chapel, New College, Oxford	1918-2000	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
23	St. Andrew's, Keighley, West Yorkshire	1800-1850	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
24	St. Anne, Catterick, North Yorkshire	1800-1850	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.2	<0.5	<0.5
25	Unknown, Thornhill, West Yorkshire	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
26	Sts. Peter & Felix, Kirby Ravensworth, North Yorkshire	1800-1850	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
27	Sts. Peter & Felix, Kirby Ravensworth, North Yorkshire	1800-1850	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.6	<0.5	<0.5
28	Sts. Peter & Felix, Kirby Ravensworth, North Yorkshire	1800-1850	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.8	<0.5	<0.5
38	Unknown	1918-2000	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
39	Unitarian Church, Portsmouth	1918-2000	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.8	<0.5	<0.5
45	Wadham College, Oxford	1600-1700	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.0	<0.5	<0.5
46	Wadham College, Oxford	1600-1700	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.1	<0.5	<0.5
47	Wadham College, Oxford	1600-1700	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.6	<0.5	<0.5
50	Norton Court, Faversham, Kent	1600-1700	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.4	<0.5	<0.5
51	Norton Court, Faversham, Kent	1600-1700	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.2	<0.5	<0.5
54	The Court Lodge, Yalding, Kent	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.8	<0.5	<0.5
55	The Court Lodge, Yalding, Kent	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.9	<0.5	<0.5
56a	The Court Lodge, Yalding, Kent	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.2	<0.5	<0.5

ID	Building	Period of Lead	Ni	Cu	Zn	As	Ag	Cd	In	Sn	Sb	Bi
56b	The Court Lodge, Yalding, Kent	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	3.6	<0.5	<0.5
63	Wroxton Abbey	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
67	York Minster	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
68	York Minster	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
69	Lincoln Cathedral	1900-1918	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
71	St Mary's, Chailey	1918-2000	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
72	St John, North Holmwood	1918-2000	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
78	Lincoln Cathedral	1800-1850	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
79	Lincoln Cathedral	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
80	Lincoln cathderal	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.8	<0.5	<0.5
85	Balliol College, Oxford	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.6	<0.5	<0.5
89	New College, Oxford	1850-1900	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
113	Battle Abbey	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.1	<0.5	<0.5
119	St. Peter and Paul's Church, Trottiscliffe, Kent	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	0.9	<0.5	<0.5
120	Norton Court, Faversham, Kent	1600-1700	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	2.2	<0.5	<0.5
122	Longfield Parish Church, Kent	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.6	<0.5	<0.5
123	Norton Court, Faversham, Kent	1600-1700	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.8	<0.5	<0.5
124	Kirby Hall	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.4	<0.5	<0.5
125	The Court Lodge, Yalding, Kent	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
126	Cranbrook Museum, Kent	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.0	<0.5	<0.5
127	Cranbrook Museum, Kent	1700-1800	<0.1	<0.1	<0.1	<0.2	<0.5	<0.5	<0.5	1.9	<0.5	<0.5