# OXBURGH HALL, OXBOROUGH, NORFOLK PORTABLE XRF ANALYSIS OF SOME WINDOW GLASS FROM THE GATEHOUSE

# **TECHNOLOGY REPORT**

David Dungworth





INTERVENTION AND ANALYSIS

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#### SUMMARY

Ninety-one panes of window glass at Oxburgh Hall were analysed *in situ* and nondestructively using portable X-Ray Fluorescence. This technique successfully identified individual panes with compositions that can be assigned to types that are known to have been produced during specific periods in the past. Slightly more than half of the analysed glass belongs to a type (HLLA) which was originally produced before the 18th century but most of the remaining glass would have been made in the 19th or 20th centuries. A consideration of the arrangement and distribution of the glass suggests that most of the windows are 19th-century creations.

#### ACKNOWLEDGEMENTS

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#### DATE OF RESEARCH

July 2014

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## INTRODUCTION

The preservation of the fabric of historic buildings must include an appreciation of a wide range of architectural materials. While stone and brick features are prominent components of a historic building, some other materials are less highly regarded. The transparency of window glass can occasionally mean that it is less noticed; in extreme cases it may even be replaced with modern glass. This is regrettable as the surface texture and tint of much historic window glass lend character to buildings. An essential requirement for making a historic window glass conservation decision is a clear understanding of the age of surviving glass. If glass is original then it will usually have greater value. This report describes the application of portable X-Ray Fluorescence (pXRF) to determine approximate date of manufacture of *in situ* window glass to enhance conservation decisions.

## AIMS AND OBJECTIVES

English Heritage has undertaken significant research into historic window glass (Dungworth 2011; 2012a; 2012b). This research has demonstrated that chronological changes in window glass composition (due to the adoption of new technologies and raw materials) can be used as the basis for determining the approximate age of manufacture of glass. The development of pXRF (eg Dungworth and Girbal 2011; Girbal and Dungworth 2011) has enabled data to be gathered non-destructively on *in situ* windows. This report presents the pXRF analysis of a small sample of the windows from Oxburgh Hall in order to provide baseline information on the date of manufacture of the glass and to enhance conservation decisions (cf Clark 2001).

## HISTORIC WINDOW GLASS: CHEMICAL COMPOSITON AS A GUIDE TO DATE OF MANUFACTURE

Traditionally, window glass was made using sand and plant ashes. For most of the medieval period the glass has a high potassium content and is believed to have been made using bracken ash (Dungworth and Clark 2004). Towards the end of the 16th century French glassmakers introduced the manufacture of a high-lime low-alkali (HLLA) glass which was probably made using the ash of hardwoods (such as oak). This HLLA glass remained in use until the end of the 17th century when it was superseded by a glass made using seaweed (kelp) ash (Dungworth 2013). Kelp glass dominated the window glass industry until the early part of the 19th century when it was abandoned in favour of soda-lime-silica (SLS) glass made using synthetic soda (Cooper 1835; Ure 1844; Muspratt 1860). Continued development of window glass manufacturing technologies led to further chronologically significant changes in glass composition (Table 1).

Phase		2a	2b	3	4a	4b	5a	5b
Start		с1567	с1600	c1700	c1835	c1870	c1930	с1960
End	с1567	c1600	c1700	c1835	c1870	c1930	с1960	
Туре	Forest	HLLA	HLLA	Kelp	SLS	SLS	SLS	SLS
Na <sub>2</sub> O	2.5±0.3	1.4±0.7	2.4±1.4	7.9±0.7	12.7±0.9	2.9±2.	13.9±0.5	13.3±0.4
MgO	7.3±0.7	3.4±0.5	3.0±0.7	5.3±0.3	0.2±0.1	0.2±0.2	2.8±0.2	3.8±0.1
$Al_2O_3$	1.6±0.5	2.8±1.0	3.0±1.3	2.6±0.6	0.6±0.1	1.2±0.3	0.9±0.6	1.3±0.2
SiO <sub>2</sub>	55.8±2.5	60.4±1.8	60.9±2.0	66.5±1.4	70.8±1.2	71.9±0.4	72.2±0.7	72.2±0.5
$SO_3$	0.3±0.1	0.2±0.1	0.4±0.2	0.2±0.1	0.4±0.1	0.4±0.2	0.4±0.2	0.2±0.1
CI	0.4±0.2	0.3±0.2	0.2±0.1	0.6±0.1	0.1±0.1	<0.1	<0.1	<0.1
$P_2O_5$	3.2±0.4	2.1±0.2	2.1±0.6	1.1±0.2	<0.2	<0.2	< 0.2	<0.2
K <sub>2</sub> O	.4± .5	5.6±1.6	5.1±1.9	4.2±0.2	0.1±0.1	0.5±0.2	0.1±0.1	0.6±0.1
CaO	15.3±1.6	21.5±1.9	21.1±1.7	10.4±1.0	14.0±0.8	12.9±1.6	9.7±0.8	8.3±0.6
MnO	1.26±0.30	0.94±0.37	0.24±0.20	<0.10	<0.10	<0.10	<0.10	<0.10
$Fe_2O_3$	0.65±0.13	1.01±0.20	1.31±0.29	0.71±0.14	0.22±0.06	0.21±0.06	0.13±0.03	0.12±0.01
$As_2O_3$	< 0.20	<0.20	<0.20	< 0.20	0.22±0.16	< 0.20	<0.20	<0.20
SrO	0.07±0.01	0.09±0.02	0.07±0.01	0.45±0.10	0.03±0.01	0.02±0.01	0.01±0.01	0.01±0.01

Table 1. Average chemical composition of historic window glass at different times (Dungworth 2011)

## OXBURGH HALL: ARCHITECTURAL BACKGROUND

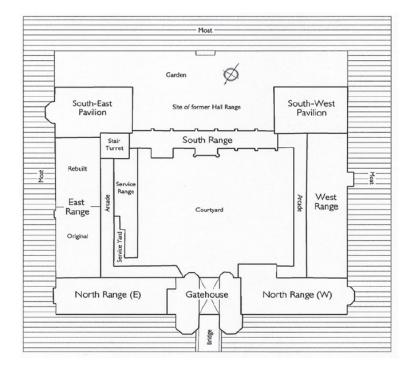


Figure I. Plan of Oxburgh Hall (after Menuge 2006)

Oxburgh Hall is a moated house near the village of Oxborough in Norfolk (Menuge 2006). The Hall as it currently survives was originally constructed in the later 15th century by Sir Edmund Bedingfield. While various parts of the moated complex have been subject

to numerous alterations, the gatehouse appears to have undergone little change (Figure I). The gatehouse was provided with substantial rooms for important visitors, possibly including Henry VII in 1487. The family fell from favour following the Reformation due to its adherence to the Catholic faith and this meant that few alterations were made to the property during the 16th, 17th and 18th centuries. The fortunes of the family improved during the 19th century and substantial repairs and other works were carried out. The Hall was gifted to the National Trust in 1952 although part is still occupied by the family.

#### METHODS

The pXRF instrument chosen to undertake the *in situ* non-destructive analysis of the historic window glass at Oxburgh Hall was a Niton XL3t (Cu/Zn Mining Mode) which allowed the simultaneous determination of the concentration of over 20 elements. The only element which was routinely determined using laboratory-based techniques but which could not be determined with the Niton XL3t was sodium. While the helium flush has been used previously in an attempt to improve the detection of light elements (especially magnesium, aluminium, silicon and phosphorus) that experience suggested that it was an unreliable method for the *in situ* analysis of historic window glass (Dungworth *et a*/2011; Girbal and Dungworth 2011). The duration of each analysis (70s) was selected to allow the collection of data from the largest possible number of panes while still obtaining reliable data. The analysis of a wide range of reference materials allowed the estimation of the likely analytical errors, precision and limits of detection (Table 2).

Oxide	Error (1 sd)	Precision (1 sd)	LoD
MgO	1.0	0.8	2.0
$AI_2O_3$	0.7	0.1	1.0
SiO <sub>2</sub>	1.0	1.0	NA
$P_2O_5$	0.1	0.1	0.2
SO3	0.2	0.1	0.2
CI	0.1	0.1	0.2
K <sub>2</sub> O	0.3	0.1	0.1
CaO	0.3	0.3	0.1
TiO <sub>2</sub>	0.02	0.01	0.05
MnO	0.02	0.01	0.02
$Fe_2O_3$	0.02	0.01	0.02
$As_2O_3$	0.02	0.01	0.02
SrO	0.005	0.002	0.005
$ZrO_2$	0.005	0.002	0.005

The pXRF analysis of weathered glass (especially where results could be directly compared with laboratory-based analyses of the same glass) has shown that it can be very difficult to obtain reliable, quantitative results from such glass (Dungworth and Girbal 2011, Table 6; Dungworth *et al* 2011). The weathered surface of glass usually has a different chemical composition to that of the uncorroded glass. In many cases this is also

accompanied by an increase in sulphur. The analysed window glass from Oxburgh Hall generally had levels of sulphur ( $< I wt\% SO_3$ ) consistent with little or no weathering. The errors and limits of detection have been increased for those panes which had UV-absorbing films (following data in Dungworth and Girbal 2011), although no reliable data could be obtained for the lightest elements (Mg, Al, S, P, S and Cl).

#### RESULTS

A total of 91 panes of glass from nine windows in the gatehouse (see Figure 1) were analysed using pXRF. This represents a small proportion of the current glazing and the selection was made following an examination of the visual characteristics of the glass with the window glass conservator Steve Clare. The visual assessment suggested that some panes were historic (ie pre-19th century) and some were more recent replacements or repairs. Some of the glass identified as being recent repairs had pronounced tint (usually green), seed (small bubbles) and, on occasion, surface irregularities. This glass was probably made specifically for use in conservation studios for the restoration of historic windows (cf Girbal and Dungworth 2011).

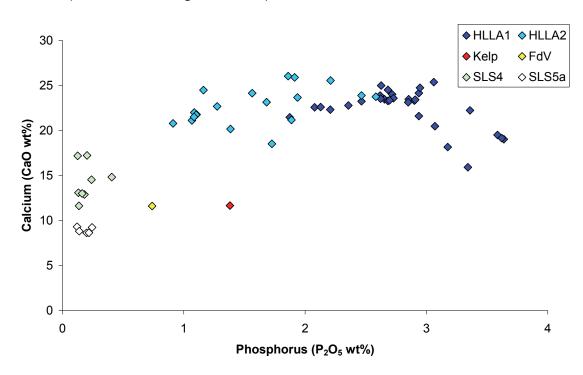


Figure 2. Phosphorus and calcium content of the window panes analysed using pXRF

Examining all of the glass results (Figures 2 and 3) allows the panes to be sorted into compositional groups. The various plant ash glasses are characterised by the presence of phosphorus (>0.5wt%  $P_2O_5$ ) while the synthetic soda glasses (soda-lime-silica glass, SLS) contain little or no phosphorus (Figure 2). The SLS glass also contains very low levels of strontium. The SLS glass can be divided into two groups based on calcium content (cf

Table 1). The higher calcium SLS glass is likely to have been manufactured between c1835 and c1930 (SLS4) while the lower calcium SLS glass is likely to have been produced after c1930 (SLS5a).

Most of the plant ash glasses are high-lime low-alkali (HLLA) glass characterised by high levels of calcium (Figure 2) The HLLA glass can be divided into two groups based on the levels of barium (Figure 3). The HLLA2 can be divided into two groups based on the strontium content: the smaller group has levels of strontium that are normal for HLLA glass (0.06–0.12wt% SrO) but the remaining HLLA2 glass has much higher levels of strontium (0.2–0.3wt% SrO). Nevertheless, high-strontium HLLA glass has been noted from Cullompton (Girbal and Ford 2010) and Apethorpe House (Dunster 2011).

The single pane of kelp glass was identified through a combination of elements but especially from the high levels of strontium (Figure 3). A single pane has also been identified as potentially façon de venise glass. This glass type was manufactured using high quality and partially purified plant ashes following the technologies developed in Venice in the medieval period (Verità 2013). It is best known from the Low Countries although production also occurred in London (Janssens *et al* 2013). Façon de venise glass is known to have been used in the manufacture of tableware but recent analysis of window glass from Apethorpe House (Dunster 2011) confirms that it was also used for windows.

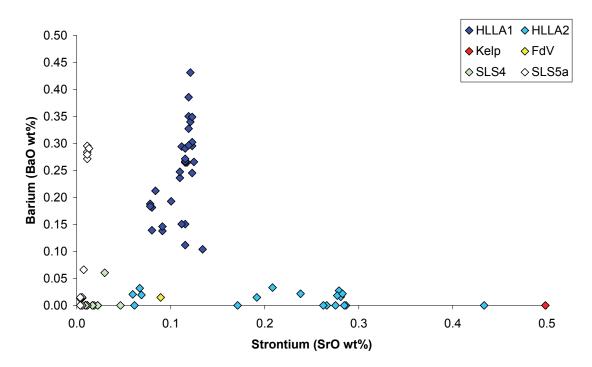


Figure 3. Strontium and barium content of the window panes analysed using pXRF

#### King's Room

The King's Room contains a large north-facing window which overlooks the bridge and approach to the Hall, and two south-facing oriel windows. The north-facing window contains eight lights in two rows with at least 700 panes of diamond-shaped glass held together with lead cames (Figure 2). The glass has varied surface finishes and tints. A sample of 18 panes from two lights (nine from each) was selected (all of these had a protective UV-absorbing film). The panes comprised the lowest two rows of whole panes in the middle right and left lights in Figure 4. These were selected to include plain (colourless) glass as well as possible studio (tinted) glass.

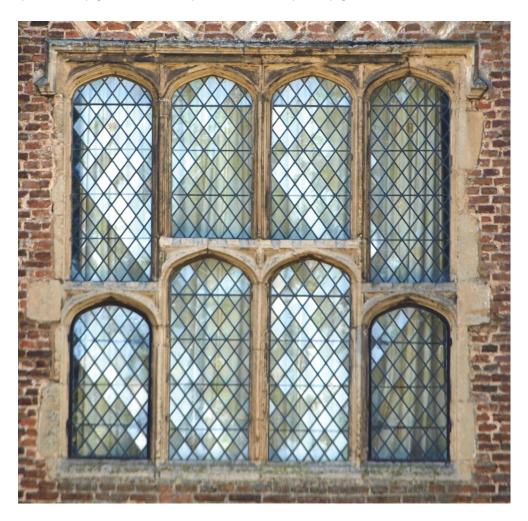


Figure 4. Oxburgh Hall, King's Room, north-facing window (exterior)

A further fifteen panes from the oriel windows on the southern side of the room (southeastern corner) were analysed. In two cases (#19 and #27) this included decorated glass which is presumed to have been Flemish in origin but added to these windows in the 19th century (Figure 5). The remaining panes were plain diamond-shaped quarries similar in quality to those of the north-facing window. The plain panes had UV-absorbing films but these were absent from the two Flemish panes.



Figure 5. Decorated 17th-century Flemish glass from the King's Room, South-eastern oriel, central window (#19)

The two Flemish panes are clearly HLLA glass. This type of glass was in use for window manufacture in Belgium from the 15th century until the 17th century (see Schalm *et al* 2007). The remaining panes (not withstanding the presence of UV-absorbing films) have compositions which closely match glass made using synthetic soda and so would have been manufactured after *c*1835. This SLS glass includes examples with high levels of calcium (SLS4, *c*1835–1930) and low levels of calcium (SLS5a, *c*1930–*c*1960). None of the SLS glass appeared to belong to the most recent category (SLS5b, 1960+). The presence of SLS5a glass suggests that repairs have been carried out since *c*1930. Most of this SLS glass contains low levels of iron (mean = 0.16wt% Fe<sub>2</sub>O<sub>3</sub>) but six panes contain 0.6-1.1wt% Fe<sub>2</sub>O<sub>3</sub> (as well as elevated levels of manganese). This is much higher than contemporary plain window glass (cf Table 1) and would appear to represent the deliberate addition of colouring agents to glass in order to manufacture a material which had some of the visual qualities of medieval glass (*ie* studio glass, cf Girbal and Dungworth 2011). The studio glass panes are distributed through the windows in order to produce an irregular effect.

King's Room – Turret

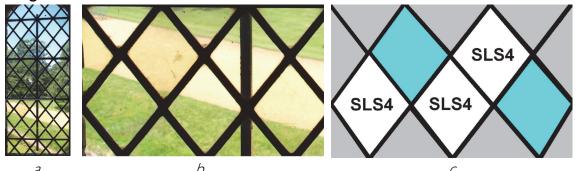


Figure 6. Window K2. a = overview; b = detail; c= diagram (light blue = HLLA2; white = SLS; grey = not analysed

The turret on the north-eastern corner of the King's Room contains five windows — all of plain diamond-shaped quarries joined with lead cames. These windows are labelled KI-5 (clockwise from the entrance from the King's Room). Approximately half of the panes in K3 were analysed but only five from K2, one from K4 and none from K1 or K5.



Figure 7. The lower portion of window K3 in the King's Room Turret a = photograph b = diagram of the same window (dark blue = HLLA I; light blue = HLLA2; red = kelp; yellow = façon de venise; white = SLS; grey = not analysed)

The five panes from K2 were selected to confirm the identification on visual aesthetic grounds of historic versus modern/studio glass (Figure 6). The pXRF demonstrated that the two panes which it was suspected were historic were HLLA glass and the other three were SLS glass. The SLS glass contains low levels of iron consistent with the use of plain (rather than studio) glass. The absence of SLS5 glass from the small number of panes analysed from this window is consistent with repair/restoration between c1835 and c1930.

The visual examination of window K3 suggested that it contained a high proportion of historic window glass and the opportunity was taken to analyse all 34 whole diamond quarries from the lower (opening) part of the window (Figure 7). The pXRF analysis shows that all but four of the panes are HLLA glass and so were probably manufactured before the end of the 17th century. This window contains one pane of kelp glass (#49) and one pane of façon de venise (FdV) glass (#63) and two panes of SLS glass (one SLS4 and one SLS5a). The SLS4 pane contains slightly elevated iron (and manganese) and may have been deliberately produced with a tint for restoration work (studio glass). The SLS5a glass suggests that some repair/restoration has occurred since c1930.

The single pane from K4 was selected to confirm that it was a studio type glass. The pXRF shows that this a SLS5a glass and reveals the high levels of iron (although not manganese) that characterises studio glass.

#### Queen's Room – Turret

The Queen's Room Turret contains five windows in a similar arrangement to the King's Room Turret on the floor below (Q1–Q5). A total of 21 panes of glass were analysed from two windows (Q1 and Q3).

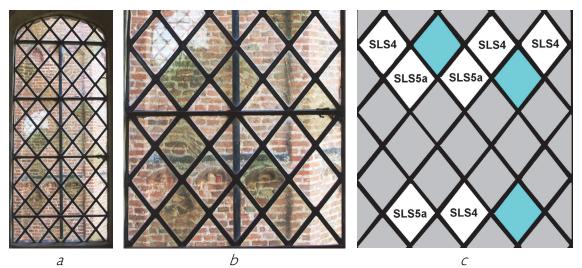


Figure 8. Window Q1. a = overview; b = detail; c= diagram (light blue = HLLA2; white = SLS; grey = not analysed

Ten panes from window QI were selected for analysis to confirm the identification on visual aesthetic grounds of historic versus modern/studio glass (Figure 8). The pXRF analysis confirmed that three panes are HLLA glass while the remaining panes are all SLS glass. The SLS4 glass panes are all plain glass while the panes of SLS5a are all studio glass. The presence of SLS5a indicates repair/restoration after c1930.

Eleven panes were selected from Q3 (overlooking the entrance to the Hall) and pXRF shows that all were HLLA glass.

## DISCUSSION

The analysis of 91 panes of glass was successfully carried out at Oxburgh Hall over a period of 3.5 hours using pXRF. The windows were analysed *in situ* and no damage was done to any of the windows. All of the panes analysed could be assigned to one of the major glass types based on an assessment of the full range of elements detected. This was less easy to achieve for those 27 panes which were covered with a UV-absorbing film; however, the data for heavy elements is less affected by UV-absorbing films than the light elements. Over half (54%) of the panes are HLLA glass which was probably manufactured between *c*1567 and *c*1700 (although any imported HLLA could have been made somewhat earlier). Slightly less than half (45%) of the panes are SLS glass which would have been made after *c*1835. The SLS glass can be divided into SLS4 (20%) made between *c*1835 and *c*1930 and SLS5a (25%) made between *c*1930 and *c*1960. The two remaining panes included one kelp glass and one façon de venise glass. The absence of any forest glass suggests that none of the original 15th-century glazing survives.

The HLLA glass shares the same overall chemical composition as HLLA glass identified in other historic buildings, such as Ightham Mote (Girbal and Dungworth 2011). The examination of the variation in minor and trace elements confirms the previous division of HLLA glass into two types: the first with high manganese and the second with low manganese. The Oxburgh Hall data also shows that there are correlations between manganese and barium. The two examples of imported Flemish glass are both of HLLA I type but are otherwise indistinguishable from the remaining HLLA1 glass from Oxburgh Hall (although it is possible that all the HLLA1 glass was imported). The analysis of dated HLLA2 glass (Dungworth 2011) suggests that this glass was produced after the first decade or so of the 17th century. It is possible that this change coincided with the development of coal-fired furnaces. While most HLLA2 window glass contains modest levels of strontium (0.06–0.12wt% SrO) a small proportion contains higher levels (0.2– 0.3wt%). High levels of strontium are usually associated with the use of kelp (Dungworth 2013) although the levels of strontium in HLLA2 glass are slightly lower than most true kelp glass (0.3–0.6wt% SrO). This high strontium HLLA2 glass has been noted at Apethorpe House (Dunster 2011) and Cullompton (Girbal and Ford 2010). In the latter case, the archaeological context suggested a very late 17th-century or early 18th-century date. It is possible that high-Sr HLLA2 represents a very late phase of HLLA production

which maintained some traditional elements of HLLA glass production but introduced some of the emerging kelp glass technology.

A single pane of glass at Oxburgh Hall is of a façon de venise type. This glass was not encountered during the first phase of English Heritage research into historic window glass (Dungworth 2011) but has been noted at Apethorpe (Dunster 2011). Façon de venise glass was produced in the Low Countries from the 15th century onwards and in London from the late 16th century onwards. It is uncertain when glass of this type ceased being manufactured, however, it is most unlikely that production continued after the introduction of Leblanc soda into glassmaking (c1835 in England). Façon de venise glass is most commonly associated with the production of high status tableware; however, contemporary documents refer to the manufacture of plate glass for mirrors and some windows. Such glass would have been considerably more expensive than ordinary window glass as some of the raw materials used to façon de venise glass were imported from the Mediterranean.

Only one pane of glass from Oxburgh hall is of a kelp glass. As this glass type was widely used during the 18th century and the first three decades of the 19th century, its virtual absence from Oxburgh is consistent with the documentary evidence for few repairs during this period. The presence of a soda-lime-silica glass of 19th-century type (SLS4) is consistent with known periods of building in the 1830s and 1860s. This includes seven panes of studio glass and eleven panes of plain glass. Latter soda-lime-silica glass (SLS5a), which includes four panes of studio glass and nineteen panes of plain glass, suggests repairs after c1930.

With the exception of the painted Flemish glass, all of the analysed glass from the King's Room is relatively modern. A small proportion is studio glass but much is plain glass of the 19th or 20th centuries. The presence of painted 17th-century Flemish glass (which was widely traded in the early 19th century) and the absence of any early (pre-c1835) plain glass in this room indicates that the glazing is probably a 19th-century creation. The presence of SLS5a glass (c1930-c1960) suggests that some restoration (and possibly more) has occurred in the 20th century.

While the windows in the King's and Queen's Rooms Turrets contain a greater proportion of historic glazing (mostly HLLA probably produced before the 18th century) it is likely that this has been subject to a degree of restoration (and possibly much more) in the 19th and 20th centuries. It is perhaps significant that windows K3 and Q3 which both look directly out from the Hall contain the greatest proportion of historic (prec1835) glazing. The windows to either side contain some historic glass in among plain and studio glass of the 19th and 20th centuries. The layout of the glazing is consistent with a programme of restoration which selected as much historic glass as possible and placed this in the most prominent positions, while modern glass was used for the peripheral windows.

# CONCLUSIONS

The pXRF analysis of 91 panes of window glass at Oxburgh Hall was undertaken to confirm the suitability of the methodology and to shed some light on the history of glazing at the site. The technique can successfully identify individual panes with compositions that can be assigned to types that are known to have been produced during specific periods in the past. It is likely that the original glazing would have been of 'forest glass' but none of this glass now survives. Slightly more than half of the analysed glass belongs to a type (HLLA) which was originally produced before the 18th century but most of the remaining glass would have been made in the 19th or 20th centuries. A consideration of the arrangement and distribution of the glass suggests that most of the windows are 19th-century creations.

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English Heritage undertakes and commissions research into the historic environment, and the issues that affect its condition and survival, in order to provide the understanding necessary for informed policy and decision making, for the protection and sustainable management of the resource, and to promote the widest access, appreciation and enjoyment of our heritage. Much of this work is conceived and implemented in the context of the National Heritage Protection Plan. For more information on the NHPP please go to http://www.english-heritage. org.uk/professional/protection/national-heritage-protection-plan/.

The Heritage Protection Department provides English Heritage with this capacity in the fields of building history, archaeology, archaeological science, imaging and visualisation, landscape history, and remote sensing. It brings together four teams with complementary investigative, analytical and technical skills to provide integrated applied research expertise across the range of the historic environment. These are:

- \* Intervention and Analysis (including Archaeology Projects, Archives, Environmental Studies, Archaeological Conservation and Technology, and Scientific Dating)
- \* Assessment (including Archaeological and Architectural Investigation, the Blue Plaques Team and the Survey of London)
- \* Imaging and Visualisation (including Technical Survey, Graphics and Photography)
- \* Remote Sensing (including Mapping, Photogrammetry and Geophysics)

The Heritage Protection Department undertakes a wide range of investigative and analytical projects, and provides quality assurance and management support for externally-commissioned research. We aim for innovative work of the highest quality which will set agendas and standards for the historic environment sector. In support of this, and to build capacity and promote best practice in the sector, we also publish guidance and provide advice and training. We support community engagement and build this in to our projects and programmes wherever possible.

We make the results of our work available through the Research Report Series, and through journal publications and monographs. Our newsletter *Research News*, which appears twice a year, aims to keep our partners within and outside English Heritage up-to-date with our projects and activities.

A full list of Research Reports, with abstracts and information on how to obtain copies, may be found on www.english-heritage.org.uk/researchreports

For further information visit www.english-heritage.org.uk

