# NORTHINGTON GRANGE, NORTHINGTON, HAMPSHIRE TECHNICAL EXAMINATION OF GLASS FROM THE CUPOLA

**TECHNOLOGY REPORT** 

David Dungworth





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# Northington Grange, Northington, Hampshire

# Technical examination of glass from the cupola

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#### **SUMMARY**

Four samples of glass from a cupola or dome previously installed at the top of a stair case at Northington Grange were examined. The glass is thick but curved; and smooth on one side but rough on the other side. The form of the glass suggests that it was cast and rolled then heated and bent over formers to achieve the desired shape. The glass is a soda-lime-silica glass made using a synthetic or industrial form of soda. This suggests that the glass would probably have been manufactured after c1835. A slightly earlier manufacture cannot be entirely ruled out as there is insufficient data on the nature of early plate glass. The presence of arsenic (presumably used as a refining agent) is most consistent with manufacture prior to c1870.

#### **ACKNOWLEDGEMENTS**

I would like to thank Pam Braddock who alerted me to the fact that the cupola was in storage at fort Brockhurst, provided access and allowed sampling.

#### **ARCHIVE LOCATION**

The remains of the cupola are stored at Fort Brockhurst, Gosport. The samples examined in this report are held in the laboratories at Fort Cumberland, Portsmouth.

#### DATE OF RESEARCH

2012

#### **CONTACT DETAILS**

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#### INTRODUCTION

This report provides data on the nature of the curved glass panels from a cupola from Northington Grange. The cupola was installed during the 19th century and removed in the 1970s. The technical examination was undertaken to provide information on the likely date of manufacture and so inform its conservation.

## ARCHITECTURAL CONTEXT

The Grange at Northington, Hampshire (SU 5622 3615) was an outlying farm of Hyde Abbey Winchester (Geddes 1983; Pevsner and Lloyd 1967). Following the Dissolution the farm passed through several owners until Robert Henley had a large house built in the late 17th century (designed by William Samwell). In the first decade of the 19th century the then owner (Henry Drummond) began to have the building completely transformed by the architect William Wilkins into a Greek Revival temple (Brock 2009). The existing house appears to have been completely encased in 'a Parthenon portico of tremendous pathos' (Pevsner and Lloyd 1967, 258). In 1817 the Grange was sold to the Baring family who had the house extended to the west, including the dining hall and conservatory. In 1868, the architect John Cox carried out an extensive internal re-design of the house to provide more light and space. From the late 19th century onwards the Grange suffered from neglect until, in the early 1970s, the fittings of the house were auctioned off, its roof removed and the western extension demolished. To prevent further demolition the Grange was taken into guardianship in 1975. A replacement roof was installed in the 1970s but a glass dome or cupola which had previously provided extra light for the stair case was not incorporated and has remained in storage at Fort Brockhurst, Gosport ever since.

The architectural context of the Northington Grange cupola suggests that it was probably manufactured and installed at some stage during the 19th century. Pevsner and Lloyd (1967, 259) note that Horace Walpole (1717–1797) observed a 'small vestibule with a cupola', but this must have been part of the Samwell building and is unlikely to have survived the remodelling by Wilkins (Brock personal communication). The fragments of cupola possibly formed part of Wilkins's original Arcadian vision for the Grange (1809), however, contemporary illustrations indicate that no cupola was present in c1830 (Brock 2009, Fig 16). The cupola is likely to have been inserted during Cox's alterations in 1868. The technical examination of samples of the glass was undertaken with the hope that this would provide some basis to distinguish between these two dates.

#### GLASS TECHNOLOGY BACKGROUND

Previous technical examination of hundreds of samples of historic window glass has provided the basis for dating surviving glass through its chemical composition (see Dungworth 2011; 2012a; 2012b). Three types of glass were employed in the manufacture of domestic window glass in the 19th century (Table 1). Throughout the 18th century and into the fourth decade of the 19th century, the mostly widely used glass for windows was made using sand and the ash of seaweed (kelp). This plant-ash glass is easily recognised through chemical analysis as it contains small amounts of phosphorus ( $P_2O_5$ ) and (for glass) exceptionally high concentrations of strontium (SrO).

Table 1. Average chemical composition of domestic window glass during the 19th century (after Dungworth 2011)

	kelp	synthetic l	synthetic2	mechanised I
	1700–1835	1835–1870	1870-1930	1930-1960
Na <sub>2</sub> O	7.9±0.7	12.7±0.9	12.9±2.1	13.9±0.5
MgO	5.3±0.3	0.2±0.1	$0.2\pm0.2$	2.8±0.2
$Al_2O_3$	2.6±0.6	0.6±0.1	1.2±0.3	$0.9 \pm 0.6$
SiO <sub>2</sub>	66.5±0.4	$70.8 \pm 1.2$	71.9±0.4	72.2±0.7
$P_2O_5$	1.1±0.2	< 0.2	< 0.2	< 0.2
$SO_3$	0.7±0.1	0.4±0.1	$0.4\pm0.2$	$0.4 \pm 0.2$
Cl	0.6±0.1	$0.1 \pm 0.1$	<0.1	<0.1
$K_2O$	4.2±0.2	$0.1 \pm 0.1$	$0.5 \pm 0.2$	$0.1 \pm 0.1$
CaO	10.4±1.0	14.0±0.8	12.9±0.6	9.7±0.8
$Fe_2O_3$	$0.71 \pm 0.14$	0.22±0.06	$0.21 \pm 0.06$	$0.13 \pm 0.03$
$As_2O_3$	< 0.2	0.22±0.16	< 0.2	< 0.2
SrO	0.45±0.10	0.03±0.01	0.02±0.01	$0.01 \pm 0.01$

The development of a synthetic soda industry based on Nicholas Leblanc's technique for converting common salt (NaCl) into sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) in the 1830s led to the abandonment of kelp as a flux in glass making. The earliest synthetic soda glasses are broadly similar to modern window glass: they are soda-lime-silica glass and these three oxides typically account for 95–98wt% of the glass. Synthetic soda glasses contain little or no phosphorus and have low iron (Fe<sub>2</sub>O<sub>3</sub>) contents which render them colourless in almost all applications. The very low levels of impurities in most 19th-century glass (eg iron and aluminium, Al<sub>2</sub>O<sub>3</sub>) suggests that high quality white sands were employed.

Around 1870 a slight shift in window glass composition is discernible: there is a drop in the arsenic  $(As_2O_3)$  content and a rise in the potassium content. Arsenic had been used to improve the clarity of glass; it can do this in several ways but one of the most important was the removal of small bubbles of air from the molten glass (refining). Later 19th-century sources warn of the health implications of using arsenic and offer alternative refining agents, such as saltpetre (potassium nitrate, KNO<sub>3</sub>).

The development of mechanical methods of drawing sheet glass directly from the furnace in the 1920s and 1930s can be detected in the chemical composition of contemporary

window glass. The time required to draw large sheets of hot and slightly plastic glass from the furnace and manipulate these led to problems with devitrification (crystallisation) defects. These problems were largely overcome by replacing a proportion of the lime (CaO) by magnesia (MgO).

The development of a window glass dating technique using its chemical composition has been highly successful, however, its application on some high status buildings has illustrated that for some customers glass of quite unusual or exotic compositions could be employed (Dungworth 2010; 2012b; Girbal and Dungworth 2011).

## THE GLASS

The glass cupola comprises several curved sheets of glass. These vary in thickness from 5.6 to 7.0mm. The concave surface is rough while the convex surface is smooth and shiny. The latter surface bears occasional surface irregularities. The form of the glass suggests that it was prepared as plates which were then heated and bent (McGrath and Frost 1937, 77, 536). The glass is largely free from any discernible colour or tint. Four samples of broken glass were collected for technical examination.

#### METHODS OF TECHNICAL EXAMINATION

The four fragments of glass were mounted in epoxy resin and ground and polished to a 3-micron finish to expose a cross-section through the glass. The samples were inspected using an optical microscope (brightfield and darkfield illumination) to identify corroded and uncorroded regions. None of the samples exhibited any substantially corroded surfaces. The samples were analysed using two techniques to determine chemical composition: SEM-EDS and EDXRF. The energy dispersive X-ray spectrometer (EDS) attached to a scanning electron microscope (SEM) provided accurate analyses of a range of elements while the energy dispersive X-ray fluorescence (EDXRF) spectrometer provided improved sensitivity and accuracy for some minor elements (in particular manganese, iron, arsenic, strontium and zirconium) due to improved peak to background ratios (Table 2).

The SEM used was a FEI Inspect F which was operated at 25kV with a beam current of approximately I.2nA (polished samples were coated in carbon to ensure they were earthed). The X-ray spectra generated by the electron beam were detected using an Oxford Instruments X-act SDD detector. The quantification of detected elements was achieved using the Oxford Instruments INCA software. The EDS spectra were calibrated (optimised) using a cobalt standard. Deconvolution of the X-ray spectra and quantification of elements was improved by profile optimisation and element standardisation using pure elements and compounds (MAC standards). The EDXRF used was an EDAX Eagle II

which was operated at 40kV and 1mA with a 300µm capillary. The EDXRF spectra were deconvoluted and quantified using the Vision32 software.

The chemical compositions of the samples are presented in this report as stoichiometric oxides with oxide weight percent concentrations based on likely valence states (the exception being chlorine which is expressed as element wt%). The accuracy of the quantification of all oxides was checked by analysing relevant reference materials (NIST and DGG) using both SEM-EDS and EDXRF. A number of elements were sought but not detected: phosphorus, titanium, vanadium, chromium, cobalt, nickel, copper, zinc, tin, antimony, rubidium and barium.

Table 2. Minimum Detection limits (MDL) and analytical errors (two standard deviations) for each oxide

	SEM-EDS			EDXRF	
	MDL	Error		MDL	Error
Na <sub>2</sub> O	0.1	0.1	MnO	0.02	0.03
MgO	0.1	0.1	$Fe_2O_3$	0.02	0.03
$Al_2O_3$	0.1	0.1	CoO	0.02	0.02
$SiO_2$	0.1	0.2	NiO	0.02	0.03
$P_2O_5$	0.1	0.1	CuO	0.02	0.01
$SO_3$	0.1	0.1	ZnO	0.02	0.01
Cl	0.1	0.1	$As_2O_3$	0.005	0.005
$K_2O$	0.1	0.1	$SnO_2$	0.1	0.05
CaO	0.1	0.1	$Sb_2O_5$	0.15	0.07
$TiO_2$	0.1	0.1	$Rb_2O$	0.01	0.005
BaO	0.2	0.1	SrO	0.005	0.005
			$ZrO_2$	0.01	0.005
			PbO	0.02	0.02

Both surfaces of one sample of glass (sample #3) were examined using the SEM to provide information on the topography of the surfaces. In this case the glass was coated in a thin layer of gold to ensure that it was earthed and the images produced using a secondary electron detector.

#### CHEMCIAL ANALYSIS OF THE GLASS

The chemical compositions of the four samples of glass are given in Table 3. It is clear that two of the samples (#2 and #4) share virtually identical chemical compositions which do not differ by more than the analytical error or precision. These two samples probably derive from glass made in the same place and at the same time. It is possible that these two samples derive from the same section of the cupola.

Despite the variation in chemical composition between the remaining samples (#1, #3 and #2/4) these all appear to be closely related to each other. No phosphorus was detected (<0.2wt%  $P_2O_5$ ) in the glass confirming that the flux was not directly derived from a plant ash. All samples are soda-lime-silica glasses with compositions similar to 19th-and 20-century synthetic soda glass. Nevertheless, the sodium content (15-16wt%  $Na_2O$ ) is higher than most 19th- or 20th-century domestic window glass (13-14wt%) and the calcium content is lower than normal (4.9-6.2wt% CaO compared to usual values of 9-15wt%). As such, the Northington Grange glass has no exact parallel among domestic window glass of the 19th and 20th centuries. The absence of magnesium is consistent with manufacture prior to 1930 and the presence of substantial proportions of arsenic has strong links to glass manufactured between c1835 and c1870.

Table 3. Chemical composition of four samples of glass from the Northington Grange cupola

	#1	#2	#3	#4
Na <sub>2</sub> O	16.1	15.4	16.1	15.5
MgO	<0.1	<0.1	<0.1	<0.1
$Al_2O_3$	0.32	0.31	0.27	0.32
$SiO_2$	76.4	76.5	75.6	76.3
$P_2O_5$	< 0.2	< 0.2	< 0.2	< 0.2
$SO_3$	0.71	0.68	0.75	0.70
Cl	0.49	0.29	0.38	0.29
$K_2O$	0.13	0.12	<0.1	0.14
CaO	4.89	5.76	6.16	5.76
$TiO_2$	<0.1	<0.1	<0.1	<0.1
MnO	< 0.02	< 0.02	< 0.02	< 0.02
$Fe_2O_3$	0.105	0.117	0.065	0.117
$As_2O_3$	0.59	0.61	0.44	0.62
$Rb_2O$	< 0.01	< 0.0	< 0.0	< 0.0
SrO	< 0.005	< 0.005	< 0.005	< 0.005
$ZrO_2$	<0.01	< 0.0	< 0.0	< 0.0
PbO	< 0.02	< 0.02	< 0.02	< 0.02



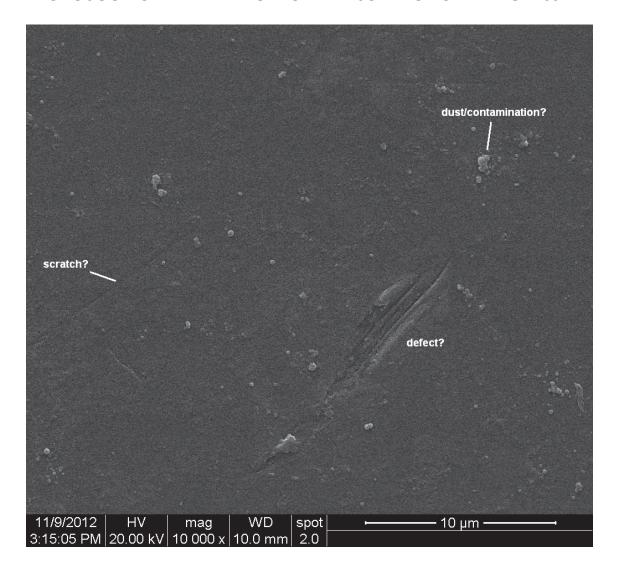


Figure 1. SEM image (secondary electron detector) of the smooth surface of sample #3. Note some surface scratches, defects and probable dust/contamination.

The smooth surface of the glass has occasional small defects and scratches (Figure I) but is otherwise largely free from surface features. The small extent of most of the defects is consistent with those that might be produced during bending rather those produced by scratches during the removal/recovery of the glass from the building. The rough surface of the glass (Figure 2) has no apparent scratches but is covered with impact craters. These vary in size from slightly less than 10µm to just over 100µm although most are close to 20µm across with a few around 100µm. The surface has the microscopic characteristics of a lapped surface, ie an abraded surface where the abrasive medium is free to move. The size distribution of the impact craters suggests that the penultimate abrasive medium was about 200µm in diameter but that not all traces of this stage of lapping had been removed. The final stage probably used abrasive particles around 50µm in diameter.

These sizes suggest the use of the very finest grades of sand or possibly emery (McGrath and Frost 1937, 36–37).

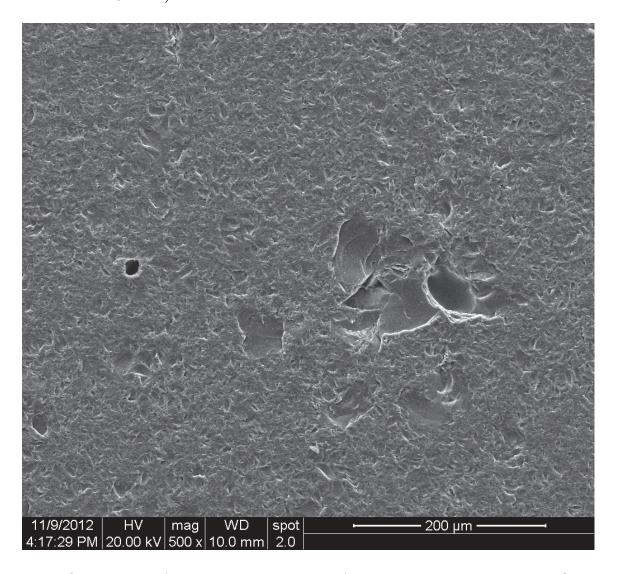


Figure 2. SEM image (secondary electron detector) of the rough surface of sample #3.

## DISCUSSION

The glass from the Northington Grange cupola has a chemical composition which suggests manufacture after the introduction of synthetic soda in the 1830s. The composition of the glass does not exactly match any contemporary domestic window glass, however, this is likely to be because of the way in which the cupola was produced. The glass would have been produced initially as flat cast plates which were ground and polished (to varying degrees on both surfaces). The plates would then have been cut and then heated and bent to shape. The investigation of domestic window glass (Dungworth 2011a; 2011b; 2012) has made use of glass which was in almost all cases blown and relatively little is known about the production of cast plate glass. Analysis of plate glass in the 19th century (Mayer and Brazier 1850) shows that this glass usually contains low levels of calcium (Table 4) which closely parallels the Northington Grange samples.

Table 4. Early 19th-century analyses of British plate glass (after Mayer and Brazier 1850). (nr = not reported)

No.	$Na_2O$	MgO	$Al_2O_3$	$SiO_2$	$K_2O$	CaO	MnO	$Fe_2O_3$
5	11.6	nr	2.7	78.7	1.3	6.1	nr	trace
6	13.1	nr	trace	77.4	3.0	5.3	nr	0.9
7	12.4	nr	3.6	77.9	1.7	4.9	trace	nr

The use of synthetic soda glass for the Northington Grange cupola would (in relation to the data from domestic window glass) suggest manufacture in the years following the introduction of the Leblanc process (ie post c1835). The limited discussion of the use of fluxes in the plate glass industry would, however, suggest that production could pre-date c1835. Contemporary sources often stress the use of the best possible ingredients that would be free from impurities (eg Brayley 1846; Muspratt 1860; Parkes 1823) and by 1823 the Ravenhead works started to make use of a form of synthetic soda produced from the double decomposition of common salt and potashes (Brayley 1846, 156; Parkes 1823, 208–9). Thus soda-lime-silica glass could, for prestigious glazing projects, pre-date c1835.

The chemical composition of the glass from Northington Grange cupola is consistent with manufacture after c1835 and before c1870; however, slightly earlier production cannot be ruled out.

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