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HIGHLAND HOUSE, 117 LIDIARD GARDENS, PORTSMOUTH
CHEMICAL ANALYSIS OF WINDOW GLASS

TECHNOLOGY REPORT

David Dungworth and Roger Wilkes

ARCHAEOLOGICAL SCIENCE

ENGLISH HERITAGE

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CHEMICAL ANALYSIS OF WINDOW GLASS

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SUMMARY

The chemical analysis of thirteen fragments of window glass from Highland House was undertaken in an attempt to identify the composition of the glass originally installed during its construction in the 1880s. Two fragments have compositions which are similar to previously analysed late 19th-century glass. The remaining samples have compositions which suggest they are later replacements. The magnesium concentration of these later samples is considered and evidence for dividing them into an early group (1930–1960) with <3wt% MgO and a later group (1960+) with 3.5–4.1wt% MgO is discussed.

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DATE OF RESEARCH

2009–10

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INTRODUCTION

The analysis of fragments of window glass from Highland House, Henderson Road, Portsmouth forms part of a much larger project undertaken to investigate the chemical composition of window glass produced and used in Britain during the past five centuries. Samples of window glass have been selected from archaeological excavations (including glass production sites) and from historic buildings. These have been analysed to determine their chemical composition. A comparison of the chemical composition with the available dating evidence shows that a series of changes in window glass manufacturing took place during this period. The aim of this research is to provide a technique to date the manufacture of individual panes of glass in historic buildings. This knowledge will allow architects and others to make more informed judgements about which glass to retain and which can be replaced (Clark 2001). Historic window glass often has a surface which is not smooth (see Figure 2 below) and usually has an unintentional tint or colour; both of these contribute to the character of the glass and the building as a whole.

Almost all glass produced in Britain during the medieval period was produced using sand and terrestrial plant ashes (primarily bracken) and has a distinctive potassium-rich composition (Dungworth and Clark 2004). The arrival of French glassmakers in the late 16th century saw a change to a high-lime low-alkali (HLLA) glass. HLLA glass was probably made using sand and 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 sand and seaweed (kelp) ash (Dungworth *et al* 2009; Parkes 1823; Watson 1782). This kelp glass dominated the window glass industry until the early part of the 19th century when it was abandoned in favour of glass made using synthetic soda (Cooper 1835; Ure 1844; Muspratt 1860).

Nicholas Leblanc invented a process for the manufacture of synthetic soda at the end of the 18th century. Common salt was heated with sulphuric acid to produce sodium sulphate (soda saltcake), The sodium sulphate was then heated with lime and charcoal or coal to produce sodium carbonate. Initially, glass could only be made with sodium carbonate, but glassmakers soon discovered that the sulphate could be used directly if it was combined with charcoal or coal. Glass made for the century or so following the 1830s was a simple soda-lime-silica glass with low levels of impurities (Dungworth 2009).

The early decades of the 20th century saw the development of techniques for automatically drawing glass (Cable 2004; McGrath and Frost 1937) which initially had problems with glass devitrifying. These problems were solved by substituting a small amount of magnesia for lime and virtually all window glass made in Britain since 1930 has contained 2–5% magnesia (Smrcek 2005).

THE GLASS

Highland House was a detached building believed to have been built in the 1880s (*personal communication* John Pike, Head of Planning Services, Portsmouth City Council). The house is not shown on Ordnance Survey maps of 1879 and 1881 but is present from 1898. The building appears to have been used initially as the quartermaster's residence in associations with the former Royal Marines Barracks at Eastney, and later as a "sick house" for married families. By 1926 Highland House was used as the chaplain's house and by 1981 was in use as a driving test centre. By 2007 the property was vacant and planning permission was granted in 2009 for its demolition and the construction of new housing. Thirteen fragments of colourless window glass were recovered immediately prior to the demolition of Highland House (Figure 1).

Figure 1. Highland House, 117 Lidiard Gardens, Southsea in October 2009

METHODS

All of the fragments of glass were mounted in epoxy resin then 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 Highland House samples exhibited any substantial 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.

The SEM used was a FEI Inspect F which was operated at 25kV with a beam current of approximately 1.2nA. 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 chemical composition of the samples is 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 a wide range reference materials (Corning, NIST, DGG and Newton/Pilkington). A number of elements were sought but not detected: phosphorus, vanadium, chromium, cobalt, nickel, copper, zinc, arsenic, tin, antimony, rubidium, barium and lead.

Table 1. Minimum Detection limits (MDL) and analytical errors for each oxide

	SEM-EDS			EDXRF	
	MDL	Error		MDL	Error
Na_2O	0.1	0.1	V_2O_5	0.02	0.03
MgO	0.1	0.1	Cr_2O_3	0.02	0.03
Al_2O_3	0.1	0.1	MnO	0.02	0.03
SiO_2	0.1	0.2	Fe_2O_3	0.02	0.03
P_2O_5	0.1	0.1	CoO	0.02	0.02
SO_3	0.1	0.1	NiO	0.02	0.03
Cl	0.1	0.1	CuO	0.02	0.01
K_2O	0.1	0.1	ZnO	0.02	0.01
CaO	0.1	0.1	As_2O_3	0.02	0.01
TiO_2	0.1	0.1	SnO_2	0.1	0.05
BaO	0.2	0.1	Sb_2O_5	0.15	0.07
			Rb_2O	0.005	0.005
			SrO	0.005	0.005
			ZrO_2	0.005	0.005
			PbO	0.02	0.02

RESULTS

All thirteen samples of window glass from Highland House are soda-lime glasses (Table 2). Samples 1, 6 and 11 share almost identical compositions and were almost certainly made at the same time and place. In addition, both samples 8 and 9 and samples 10 and 12 share almost identical compositions.

Table 2. Chemical composition of the Highland House window glass

	Na_2O	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	Fe_2O_3	SrO	ZrO_2
#01	14.92	2.89	0.31	72.3	0.24	<0.1	9.18	0.12	<0.005	0.008
#02	14.49	2.82	0.24	72.9	0.22	<0.1	9.16	0.15	<0.005	0.015
#03	12.32	0.12	1.50	71.9	0.30	0.65	12.94	0.22	0.010	0.007
#04	11.91	<0.1	1.42	72.0	0.28	0.59	13.53	0.21	0.010	0.008
#05	13.42	2.97	1.48	72.5	0.18	0.66	8.57	0.23	0.008	0.007
#06	14.96	2.90	0.31	72.3	0.25	<0.1	9.17	0.12	0.005	0.009
#07	13.91	3.94	1.27	72.3	0.21	0.39	7.81	0.15	0.028	<0.005
#08	14.63	3.97	1.02	71.9	0.19	<0.1	8.09	0.17	<0.005	0.021
#09	14.37	3.92	1.08	72.0	0.21	<0.1	8.22	0.17	<0.005	0.026
#10	15.45	3.62	0.68	71.7	0.37	0.18	7.76	0.17	0.020	0.006
#11	14.95	2.88	0.29	72.3	0.21	<0.1	9.23	0.12	<0.005	0.006
#12	15.49	3.61	0.67	71.8	0.35	0.19	7.74	0.17	0.022	0.005
#13	14.80	2.89	0.24	72.7	0.16	<0.1	9.03	0.13	0.005	0.012

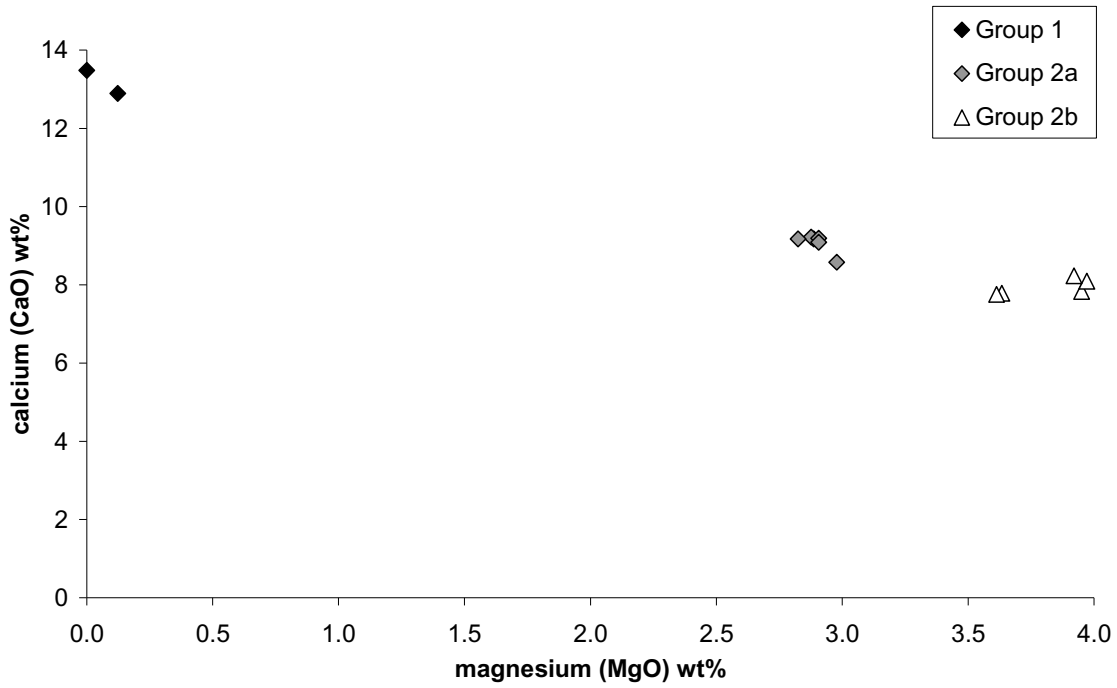

Figure 1. Plot of magnesium and calcium content of the Highland House window glass

The variations in chemical composition suggest three major groups: the first with negligible magnesium (<0.2wt% MgO) and the second with 2.8–4.0wt% MgO (Figure 1). Magnesia was deliberately added to window glass from the late 1920s onwards in order to overcome problems of devitrification during forming (Turner 1926; Cable 2004). It is likely that the glass in Group 1 windows was produced before 1930 and that Groups 2 and 3 were produced after 1930.

DISCUSSION

Table 3 shows the Highland House Group 1 glass samples have compositions which are very similar to those of Wentworth (1877, Dungworth and Wilkes 2010) and Welch Road (1894–5, Dungworth in preparation a). The Group 1 glass probably represents the composition of the glass installed at Highland House in the 1880s.

Table 3. Chemical composition of some 19th-century glass
(1 = Dungworth 2009; 2= Hatton 2004; 3 = Dungworth and Wilkes 2010;
4 = Dungworth 2010a)

	Source	Date	Na_2O	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	Fe_2O_3	As_2O_3	SrO
Chatsworth	1	1837–40	14.0	<0.1	0.7	70.3	0.34	<0.1	14.1	0.20	0.41	0.015
Nailsea	2	1830–70	13.1	0.2	0.8	68.9	0.60	0.1	13.5	0.33	0.22	0.022
Wentworth 1	3	1877	11.9	0.4	0.7	71.5	0.24	0.3	14.3	0.28	<0.02	0.026
Welch Road	4	1895	11.6	0.1	1.5	72.5	0.30	0.6	13.1	0.20	<0.02	0.019

The Highland House glass with significant levels of magnesium has been divided into two sub-groups (2a and 2b). These sub-groups recur in other samples of analysed post-1930 window glass which contain >2wt% MgO (Figure 2). The first group (cf Highland House Group 2a) contains 2.7–3.0wt% MgO while the second group (cf Highland House Group 2b) contains 3.5–4.1wt% MgO). A review of published analyses of 20th-century window glass by Smrcek (2005) suggests that relatively small proportions of magnesia were used by British glassmakers between 1930 and 1960, but that higher proportions of magnesia were employed after the development of the float process at the end of the 1950s.

The available data suggests, therefore, that it may be possible to distinguish between British window glass manufactured between 1930 and 1960 and glass manufactured since 1960 on the basis of the magnesium content.

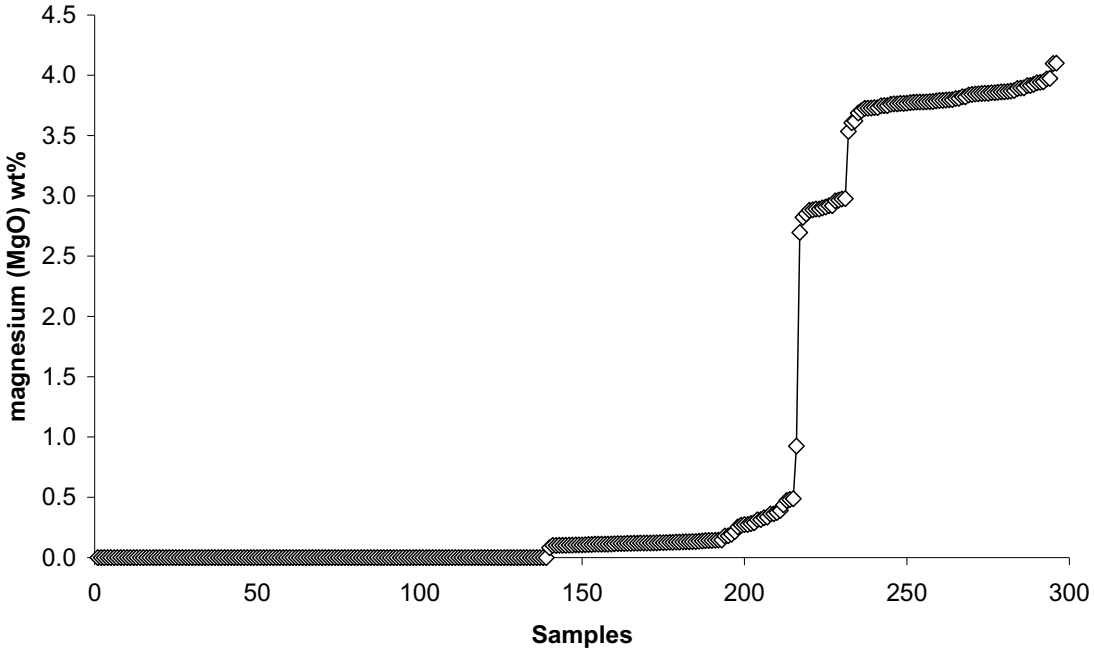

Figure 2. Magnesium concentration of 296 samples of 19th- and 20th-century soda-lime glass in rank order (data from this report; Dungworth 2009; 2010a; 2010b; Dungworth and Loaring 2009; Dungworth and Wilkes 2010)

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