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TECHNOLOGY REPORT

David Dungworth





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SUMMARY

The chemical analysis of sixteen fragments of window glass from four separate houses in Welch Road was undertaken in an attempt to identify the composition of the glass originally installed during the construction (1894–95). Nine fragments (including at least one fragment from each property) share virtually identical compositions and so are interpreted as original glass. The original window glass is a soda-lime-silica glass and the composition is compared with available data for earlier 19th-century and 20th century window glass.

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DATE OF RESEARCH

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INTRODUCTION

The analysis of fragments of window glass from Welch Road, Southsea 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 This HLLA glass remained in use until the end of the 17th century when it was superseded by a glass made using sand and seaweed (kelp) ash (Dungworth *et al* 2009; Parkes 1823; Watson 1782).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

The east side of Welch Road was built in two sections in 1894 and 1895 (Figure 1) and window glass from these houses has the potential to provide information on the composition of window glass used in Britain in the last decade of the 19th century. Over the past few years every opportunity to sample glass has been taken and samples have been obtained from four houses. In three cases window glass was collected during the installation of double glazing, while in the remaining case sampling was undertaken during repairs (in this case the glass was returned to its original position after sampling). Most of the glass sampled for analysis has a slightly wavy surface (Figure 2) and occasionally bubbles, however, the surface textures were not sufficiently distinctive to allow immediate identification of the forming technique.



Figure 1. Moulded ceramic plaques commemorating the construction of Rowland Terrace and Welch Terrace (the eastern side of Welch Road)



Figure 2. A typical window from Welch Road with a characteristic surface texture indicating that it was manufactured before the development of the float glass process in 1960

Table 1. Provenance and description of samples

House	Pane	Thickness	Description
9		2.63mm	Colourless, wavy surface, occasionally bubbles
9	2	3.26mm	Colourless, wavy surface, occasionally bubbles
9	3	3.84mm	Colourless, wavy surface, occasionally bubbles
13	1	3.10mm	Colourless, wavy surface, occasionally bubbles
13	2	2.37mm	Amber, flat surface
13	3	2.54mm	Colourless, acid-etched design
13	4	1.76mm	Colourless, acid-etched design
15	I	2.93mm	Colourless, wavy surface, occasionally bubbles
15	2	2.69mm	Colourless, wavy surface, occasionally bubbles
15	3	3.98mm	Colourless, flat surface
19	1	3.08mm	Colourless, wavy surface, occasionally bubbles
19	2	3.23mm	Colourless, flat surface
19	3	2.89mm	Colourless, wavy surface, occasionally bubbles
19	4	3.47mm	Colourless, wavy surface, occasionally bubbles
19	5	2.85mm	Colourless, wavy surface, occasionally bubbles
19	6	3.20mm	Colourless, wavy surface, occasionally bubbles

METHODS

All of the 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 Welch Road 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 I.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 (two standard deviations) for each oxide

	SEM-E	:DS		EDXR	F	
	MDL	Error		MDL	Error	
Na ₂ O	0.1	0.1	V ₂ O 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	0.03	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 ₂ O	0.005	0.005	
			SrO	0.005	0.005	
			ZrO_2	0.005	0.005	
			PbO	0.03	0.02	

RESULTS

All of the Welch Road glass is rich in sodium, calcium (Figure 3) and silicon with low concentrations of other elements. These glasses have chemical compositions which indicate that they were made with relatively pure raw materials. Two samples (#15.3 and #19.2) have low lime content compared to the other samples and high concentrations of magnesia indicating that they were probably manufactured after 1930. Sample 13.2 has a high manganese content, however, this was the only example of analysed glass which was not colourless. The manganese may have helped to provide this glass with its amber colour. Figure 4 shows the concentrations of two minor oxides in the glass (alumina and potassium oxide). This illustrates that nine fragments (including at least one fragment from each of the four houses) have chemical compositions which are almost identical (the variations in chemical composition between these samples is rarely greater than the analytical precision, as shown by the error bars).

Table 2. Chemical composition of the Welch Road glass samples

#	Na ₂ O	MgO	Al_2O_3	SiO ₂	SO ₃	Cl	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	SrO	ZrO_2
9.1	11.66	0.11	1.39	73.7	0.26	< 0.05	0.56	12.06	<0.05	< 0.02	0.167	0.023	0.009
9.2	11.97	<0.1	1.55	71.5	0.36	< 0.05	0.58	13.68	0.06	< 0.02	0.193	0.012	0.010
9.3	11.77	<0.1	1.44	72. I	0.34	< 0.05	0.57	13.27	0.12	< 0.02	0.190	0.015	0.008
13.1	11.66	0.12	1.58	71.6	0.29	< 0.05	0.60	13.48	0.07	< 0.02	0.186	0.008	< 0.005
13.2	16.91	0.31	0.73	68.0	<0.1	0.12	<0.1	14.44	0.07	0.27	0.187	0.020	< 0.005
13.3	13.44	0.49	0.53	69.8	0.34	< 0.05	<0.1	14.84	< 0.05	< 0.02	0.175	0.027	0.010
13.4	13.47	0.45	0.54	70. I	0.29	< 0.05	<0.1	14.86	0.06	< 0.02	0.175	0.026	0.009
15.1	12.97	0.27	1.04	70. I	0.40	< 0.05	<0.1	14.64	0.06	< 0.02	0.295	0.018	0.006
15.2	11.73	0.13	1.46	73.7	0.24	< 0.05	0.54	12.03	< 0.05	< 0.02	0.168	0.019	0.007
15.3	12.84	3.78	1.11	72.8	0.21	0.06	0.52	8.71	< 0.05	< 0.02	0.126	0.009	0.008
19.1	12.12	0.92	0.67	70.8	0.36	< 0.05	<0.1	14.48	< 0.05	< 0.02	0.283	0.022	0.008
19.2	15.44	3.53	0.13	71.3	0.39	0.08	<0.1	9.17	< 0.05	0.11	0.067	0.012	0.007
19.3	11.35	0.14	1.49	72.6	0.28	< 0.05	0.56	13.27	0.06	< 0.02	0.245	0.024	0.009
19.4	11.40	0.11	1.47	72.5	0.31	< 0.05	0.59	13.28	0.08	< 0.02	0.244	0.026	0.007
19.5	11.41	0.10	1.57	72.4	0.29	< 0.05	0.59	13.23	< 0.05	< 0.02	0.245	0.025	0.007
19.6	11.62	0.11	1.39	72.3	0.29	<0.05	0.52	13.60	0.09	< 0.02	0.180	0.023	0.007

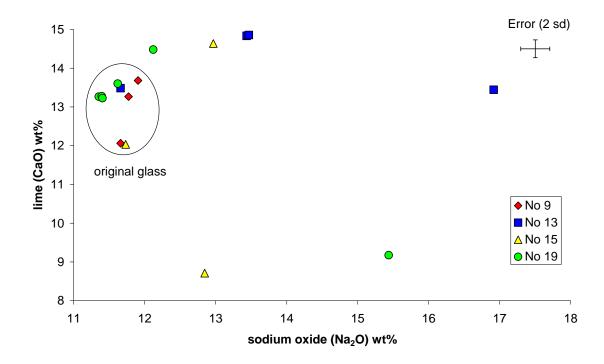


Figure 3. Plot of sodium oxide and lime content of the Welch Road window glass. The ellipse indicates those samples which share similar compositions and are likely to be original

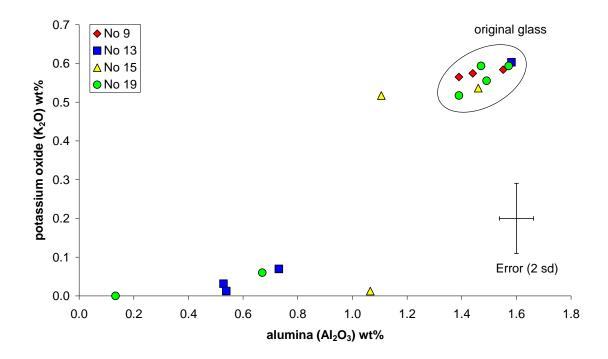


Figure 4. Plot of potassium oxide and alumina content of the Welch Road window glass. The ellipse indicates those samples which share similar compositions and are likely to be original

DISCUSSION

The sixteen samples of Welch Road window glass analysed include nine fragments with virtually identical compositions which probably represent the glass that was originally installed in these houses when they were built in 1894 and 1895. The remaining seven samples have rather varied compositions are some of these are likely to represent piecemeal replacement of windows in the decades which followed. Three of the samples from No 13 Welch Road were from a decorative window (one amber and two acidetched) and may be original but obtained from a different source. Sample #15.1 has a low magnesium content and probably represents replacement before 1930. Samples #15.3 and #19.2 have a high magnesium content and probably represent replacement after 1930.

The Welch Road glass contributes to an understanding of the changes in the manufacture of window glass in the 19th and 20th centuries (Table 3). The glass used in 1894–5 at Welch Road closely resembles that glass used for the construction of the Wentworth Conservatory in 1877 (Dungworth and Wilkes 2010a). The glass from both Welch Road and Wentworth contains relatively modest concentrations of sodium compared window glass made earlier in the 19th century or in the 20th century. It is possible that the low sodium content of the late 19th-century window glass was the result of developments in furnace technology which enabled glassmakers to achieve higher temperatures

(Dungworth and Wilkes 2010a). A second difference between the earlier and later 19th-century glass can be seen in the concentration of arsenic; while arsenic is present in the earlier glass it could not be detected in any of the later 19th-century samples (see also Dungworth and Wilkes 2010b).

Table 3. Chemical composition of some 19th- and 20th-century flat glass (Sources: I = Dungworth 2009; 2 = Hatton 2004; 3 = Dungworth and Wilkes 2010a; 4 = this report; 5 = Dungworth 2010; 6 = Smrcek 2005)

	Source	Date	Na ₂ O	MgO	Al_2O_3	SiO ₂	SO ₃	K ₂ O	CaO	Fe ₂ O 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 I	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
Fort Cumberland	5	1940	14.3	2.9	0.3	72.5	0.25	<0.1	9.4	0.13	< 0.02	0.008
Drawn	6	1930-60	14.6	2.1	1.0	72.0	0.45	0.1	9.8	0.12		
Wentworth 2	3		13.3	3.8	1.4	72.5	0.19	0.7	7.9	0.22	< 0.02	0.007
Wentworth 3	3		13.3	3.8	1.1	72.4	0.19	0.6	8.4	0.15	< 0.02	0.006
Float	6	1960–99	13.8	4.1	1,1	71.9	0.19	0.6	8.1	0.19		

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