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18th- and 19th-Century Scottish Laboratory Glass: Assessment of Chemical Composition in Relation to Form and Function

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The glass manufacturing industry in Scotland began in 1610, and its early focus was on the manufacture of bottles and other vessels.[1] Over time, other items, such as windows, mirrors, and specialized pieces, were made in Scottish glasshouses.[2]

The 18th century marked the emergence of chemistry teaching and research in Scotland, particularly at the Universities of Glasgow and Edinburgh. The glassware used in these early lectures and experiments is the focus of this article. Little is understood about the ingredients and techniques involved in the manufacture of specialized glass items employed in early Scottish scientific work—or about how specialized these items were.

In the 18th century, there was a rapid increase in the volume and diversity of glass manufactured in Scotland.[3] During this period, most Scottish glass was made with two main ingredients: silica, sourced from sand, and an alkali flux to reduce the melting temperature of the silica.[4] Other minor ingredients may also have been included to improve the chemical stability and durability of the glass, to remove gas bubbles, or to add or remove color.[5]

The ingredients used to manufacture glass evolved over time. From the 18th century to about 1840, ashes of seaweed were employed as the main alkali flux. This type of glass—known as kelp-fluxed glass—was used for all manner of glass items, including bottles and window glass.[6] Around 1790, the French scientist Nicolas Leblanc (1742–1806) invented an industrial process to make sodium carbonate (synthetic soda), an alternative alkali flux. In 1825, Charles Tennant opened a factory in St. Rollox, Glasgow, Scotland, United Kingdom, to manufacture Leblanc's synthetic soda. Within 10 years, Tennant's factory had become the largest chemical works in Europe, with synthetic soda as its primary output.[7] Throughout the 1830s and 1840s, the alkali flux used in manufacturing Scottish glass moved away from kelp and toward synthetic soda.[8]

The transition from kelp fluxing to synthetic soda caused a change in the chemical makeup of glass. Kelp-fluxed glass is characterized by the presence of strontium (~0.4%), magnesium (~5%), potassium (~4%), and calcium (~10%); early synthetic soda glass contains no detectable strontium, magnesium, or potassium, but it does contain a higher degree of calcium (~14%) and a small amount of arsenic (~0.2%).[9] Calcium acts as a stabilizer, ensuring that the glass is durable. Kelp-fluxed glass contained calcium naturally, whereas synthetic soda did not. For this reason, lime was introduced as an ingredient in synthetic soda glass batches to provide calcium and to make the glass stable.[10]

Scottish window glass of the 18th and 19th centuries includes these significant chemical markers,[11] which allow us to deduce the manufacturing type, using scientific methods. For example, in kelp-fluxed glass, the presence of both potassium and strontium is identified using portable X-ray fluorescence; identifying the glass type allows us to infer approximate dates of manufacture. First-generation synthetic soda glass, dating from the 1830s to the 1870s, can be identified by the level of calcium and the presence of arsenic, which was added to help purge gas bubbles from the melt.[12] Other analytical techniques, such as scanning electron microscopy, can also be employed, but these may involve taking a small sample from a collection piece, which is not desirable for historically important samples.

"Standard" glassware, such as windows and bottles, can sometimes be characterized chemically and assigned an approximate date of manufacture using these methods. Until now, the chemical profile of Scottish laboratory glass has not been examined. This leads to several questions: Was Scottish laboratory glass manufactured in the same way as standard bottles but shaped differently, or were these items manufactured with custom-made ingredients? If unique constituents were used, was this to confer any specific qualities on the glassware, and could this indicate a high level of expertise in the Scottish glass industry at that time?

To begin to answer these questions, 18th- and 19th-century laboratory glass from the University of Edinburgh is investigated in this article. Samples for analysis were taken from two sources: (1) the Playfair Collection, and (2) samples excavated archaeologically from the Old College Quadrangle.

The Playfair Collection

In 1858, Prof. Lyon Playfair donated items from the chemistry laboratories of the University of Edinburgh to the Industrial Museum of Scotland. These items, termed the Playfair Collection, consisted of laboratory equipment used by Professor Playfair's predecessors as professors of chemistry at Edinburgh: Joseph Black (1766–1795), Thomas Charles Hope (1795–1844), and William Gregory (1844–1858).[13]

Joseph Black, one of the most famous names in Scottish scientific history, is credited with the discovery of carbon dioxide. Black succeeded Prof. William Cullen at the Universities of Glasgow and Edinburgh in 1766; by that time, chemistry and medicine were already established subjects with strong numbers of students. Over the next 92 years, the teaching of chemistry expanded rapidly to allow students to undertake practical experiments, the language of lectures changed from Latin to English, and class sizes increased.[14]

During the mid-19th century, the University of Edinburgh expanded, with the construction of new laboratories. When he assumed the chair of chemistry, Professor Playfair inventoried obsolete equipment that was no longer being used in teaching or experiments, and, instead of destroying these items, he donated them to the Industrial Museum. Today, the National Museums Scotland in Edinburgh houses the Playfair Collection.

Robert Anderson, a museum curator and historian of chemistry, extensively catalogued the various items of the Playfair Collection in 1978.[15] He identified which professor had purchased some of this equipment, allowing us to consider a range of dates for when these items were manufactured. For example, Anderson attributed an air thermoscope (cat. no. 1858.275.9) to Professor Hope, and so we can assign approximate dates of manufacture and purchase to 1795–1844. The professors who purchased some items are not known, and so a wider range of dates must be considered for those.

Old College Quadrangle: Evaluation and Excavation

In 2010, the University of Edinburgh contracted with Addyman Archaeology for an assessment and archaeological investigation of the interior courtyard of the university's main building complex, known as the Old College Quadrangle, before a proposed redevelopment with a new scheme of paving and landscaping.[16] The quadrangle, which forms the central area of the university, is surrounded by buildings dating to the late 18th and early 19th centuries. It is situated close to the heart of medieval Edinburgh. The present building was constructed in 1789–1792 and completed about 1819–1833 by the architects Robert Adam and William Playfair respectively.

An evaluation of the site, conducted in March 2010, targeted the expected remains of previous buildings. Exposure and recording of the entire court surface took place from June to October 2010, with numerous individual sondages and wider excavation areas opened up to investigate particular areas, features, and deposits. The ground level in the courtyard had been lowered and leveled, sometime around 1829, as part of William Playfair's completion of Adam's partly built quadrangle building. The excavation also uncovered substantial remains of the pre-existing college buildings, including the common hall building of 1619, the library of 1642–1646, courtyards, and other structures.

Two of the deeper sondages, made within the library, recorded about 10 glass artifacts and brightly colored chemical compounds. The glass appeared to be laboratory glass, with artifacts including thermometer rods, flasks, and bottles. The conical base of one glass item contained a quantity of what appeared to be a chemical compound. This suggested to the archaeologists that this glass was related to the practice of chemistry in the late 18th and early 19th centuries. These findings led to further excavation in this area in June 2011 to obtain a larger sample and to better understand the circumstances of deposition.

It is important, in the context of this study, that the finds excavated from the library building included laboratory glass. In 1777, Joseph Black moved into that building, and a reference in the Edinburgh City Archives records that in 1800, a year after Black's death, Thomas Charles Hope stored Black's old chemistry apparatus in its cellar.[17] The excavation of the site in June 2011 produced more than 50 glass samples. In addition to chemical glassware, the archaeological excavations recovered window glass, wine bottles, and drinking vessels.

Manufacture of Scottish Laboratory Glass

The period during which glassworks manufactured and sold early laboratory glassware to the University of Edinburgh is of interest in the context of the Scottish glass industry and it manufacturing practices. Compared to other nations, relatively little is known about the Scottish glass industry of this period. Anderson argues that the glassworks in Leith, Edinburgh, produced the glassware for the laboratories.[18] He goes on to suggest that the relationship between the university and the glassworks was so strong that Joseph Black would often visit the latter in person. The basis of this relationship was the friendship between Black and the owner of the Leith glassworks, Archibald Geddes, a former student of Black's. Black subsequently became a director of the glassworks. The Leith glassworks manufactured standard glass items such as bottles and windows,[19] making it possible that these same glassblowers in the same glasshouse manufactured these items for the university.

What was asked of the glassworks, and how did it produce its wares? Was the glass that was made for the university's laboratories simply window or bottle glass that was shaped uniquely, or were the batch ingredients also altered to provide different properties to the glass? To answer these questions, we undertook scientific analysis of glass excavated from the University of Edinburgh and from the Playfair Collection in an effort to better understand the manufacturing process of laboratory glassware in the 18th and 19th centuries.

Materials and Methods

In this study, we analyzed 14 pieces from the Playfair Collection and 14 samples excavated from the Old College Quadrangle using portable X-ray fluorescence to determine their chemical characteristics. Some of the pieces from the Playfair Collection are in several parts. For example, the differential thermoscope, or "photoscope" (cat. no. 1858.275.10; Fig. 1), consists of two glass spheres blown at each end of a glass capillary that was bent in two right angles. The catalog number refers to two differential thermoscopes, so, for this item, four spheres and two capillary tubes were analyzed.

Table 1 details the analysis of the samples and lists the chemistry professors with whom Anderson associated them.[20] A Niton XL3t system was employed for the portable X-ray fluorescence analysis, following the procedure of Kennedy, Murdoch, and Kirk.[21] With the surface of the glass placed against the nose cose, the instrument generated X-rays for 50 seconds over an area eight millimeters in diameter. To improve light-element detection, we flushed the nose cone with helium. The lightest element detectable by the XL3t is magnesium.

Of the nine options available through the XL3t (alloy, alloy electronics, dental alloy, precious metals, mining, soil, exploration, lead paint, and thin sample and plastic), we selected the Cu/Zn mining mode because it allowed us to detect more than 20 elements, especially those of interest in the analysis of historical glass. The software uses a Fundamental Parameters algorithm to calculate concentrations, in parts per million, of each element. The values recorded were divided by 10,000 and then multiplied, according to a standard element oxide conversion table, to produce a weight percentage of each oxide. Kennedy, Murdoch, and Kirk noted that the XL3t is very accurate in detecting concentrations of heavier elements, although there is less confidence in the accuracy of the figures given for the lightest elements and, within a glass matrix, silicon.[22]

Results

Table 2 presents the results of the X-ray fluorescence examinations of the Playfair Collection. Overall, the samples from this collection fall into two broad categories. The first category is high-lead glasses, in which the lead content is between 23 and 38 percent. Accompanying this lead content are elevated levels of arsenic (0.65%–2.1%) and potassium (1.5%–7.2%). The second category contains no lead, but it does have a high amount of calcium (up to 26.4%), as well as magnesium, aluminum, strontium, iron, titanium, and sulfur. The high-calcium glasses reveal the presence of strontium, indicating that kelp was used as the alkali flux. This is in keeping with the date range of these pieces: 1752–1843. Window glass in Scotland was manufactured using kelp until at least 1839.[23]

High-lead glasses constitute the largest part of the samples analyzed. Most of these glasses are attributed to the time of Professors Hope and Gregory, from 1795 onward. The two exceptions are the bottle with a faucet (cat. no. 1858.275.34; Fig. 2), for which no professor was named, and a collection of bottles and flasks (cat. no. 1858.275.39; Fig. 3) tentatively assigned to Professors Black and Hope, in which only two of the five bottles analyzed had a high lead content.

Most of the high-calcium glasses date from the time of Professor Black and, in some cases, perhaps Professor Hope. This supposition supplies us with 1752–1843 as the date range for these pieces. In general terms, it would appear that the earlier glasses, dating from Professor Black's time, were high-calcium items, and that later glasses, dating from the time of Professors Hope and Gregory, were high-lead glasses. There may be some overlap during Professor Hope's time, given that several of the items could not be assigned to one particular professor. Moreover, given the exceedingly long tenure of Professor Hope, it becomes even more difficult to declare with certainty the dates of manufacture of some glass items.

The samples excavated from the Old College Quadrangle present a chemical profile similar to that of the objects from the Playfair Collection (Table 3). Some of these samples have high proportions of lead (over 30%), while others display no lead but a high amount of calcium oxide.

From within the quadrangle finds, butnot the Playfair Collection, a third category of samples emerges. There is a distinct difference between this category and the categories from the Playfair Collection and other quadrangle samples. Four samples show a low level of lead oxide (14%–25%), along with small quantities of iron, strontium, calcium, and potassium.

Another interesting aspect of these results is that the green glass exhibits between 0.26% and 2.28% iron oxide. There are several possible sources for the inclusion of iron oxide; it can be found in the sand or added separately in small amounts as a decolorizer or at higher levels as a colorant.[24] In its reduced form, Fe^{2+}, iron produces a strong blue color in glass; in its oxidized state, Fe^{3+}, a yellow-green color results.

Discussion

Lead glass was introduced commercially in Scotland during the 17th century.[25] During the 18th century, the use of lead glass increased for scientific experiments, particularly among scientists whose work relied on high-quality lenses, such as those found in telescopes.[26] Early attempts at lead crystal manufacture showed that low-lead glass had issues with water and stability, in which the glass seemed to "weep." The English merchant and glassmaker George Ravenscroft (1632–1683) solved this problem in the late 17th century by raising the lead content of glass from 15 percent to 30 percent.[27] In 1849, the British glassware manufacturer Apsley Pellatt (1791–1863) wrote that, for much of the 18th century, the only heavy glass used for telescopes in Britain and elsewhere in Europe was English flint glass, which used a standard flint glass recipe with an additional 10 percent of lead.[28]

Lead is a strong flux that reduces the melting temperature of the silica and produces qualities desirable for laboratory glassware, including a high refractive index, high transparency, and the ability to withstand a wide range of temperatures.[29] The Playfair lead glasses have characteristics that are slightly different from those of modern lead glass; they also display similar levels of lead and potassium, but higher levels of arsenic.[30] It is interesting to note that Sir James Standsfield (d. 1687), a prominent Scottish entrepreneur and glassworks owner who listed the ingredients needed to make flint (lead) glass in the late 17th century, included (in some cases) white arsenic and yellow arsenic.[31] Arsenic acts as a decolorizer of lead glass.[32] Among the samples analyzed in our study, increased levels of lead appear to be accompanied by increased levels of arsenic (Fig. 4).

David Dungworth and Colin Brain, who analyzed English lead drinking glasses of the late 17th century,[33] sometimes found levels of lead comparable to those in the Playfair Collection. However, they did not detect arsenic as part of the chemical makeup of their glasses. Their samples showed a relationship between the relative levels of lead and potassium, in which the later samples had higher levels of lead and lower levels of potassium. The results of our study do not match the findings of Dungworth and Brain, indicating that the lead glasses produced for the university's laboratory glassware was manufactured using different ingredients and techniques to those used for tableware.

Among the glasses excavated from the Old College Quadrangle is a set of samples with moderate levels of lead (below 25%). These samples contain strontium (0.17%–0.18%) and iron (~0.5%), which are not observed in glasses with a higher proportion of lead. In addition, as the lead content decreases in these samples, the calcium content increases (Fig. 5). There are numerous possibilities for explaining why the glasses with moderate levels of lead contain these other elements. One is that the samples—the wall and base of a vial—

were designed this way to confer certain physical properties, such as transparency and stability. A second possibility is that these glasses were made with cullet of nonlead glass (e.g., window or bottle glass). Cullet was, and still is, used extensively in glassmaking.[34] By introducing broken glass of one type into a melt of another type, a “hybrid” sample of glass can be produced. Similar examples have been observed in window glass, with characteristics of two glass types in one sample.[35]

The composition of laboratory glassware of this type has not been previously analyzed. The fact that many of the analyzed items contain lead is understandable, given the emergence of lead glass in the late 17th century and its application for scientific purposes.[36] This suggests that Scottish glassmakers, like their counterparts in other countries, were able and willing to manufacture pieces made of such glasses for various purposes, employing unusual ingredients and skills.

The types of glasses examined from the Playfair Collection and the Old College Quadrangle excavations are not chemically similar to window or bottle glasses manufactured in Scotland at about the same time. Although calcium is a feature of window glass, it tends not to be present in quantities above 15 percent.[37] Most of the high-calcium glasses in the Playfair Collection and the quadrangle excavations had calcium levels above 20 percent. The difference can be explained by the durability of “standard” glass compositions needed for chemical and thermal experiments. Reducing the levels of sodium and increasing the levels of magnesium or calcium improves the resistance of glass both to attack from acidic or basic solutions and to thermal expansion.[38] These attributes are desirable for glassware to be employed in laboratory experiments. In our study, the levels of magnesium observed in the high-calcium glasses are similar to those in historical Scottish window glass,[39] but the calcium levels are significantly higher. The high-calcium glasses in the Playfair Collection include cucurbits, bottles, flasks, and other vessels that were probably used to hold or store chemicals. In addition, tubes to produce electrostatic charges were manufactured using this form of glass.

The manipulation of the chemical composition of glassware to improve durability predates the Scottish glass industry. In the 13th century, Venetian glassmakers increased the levels of calcium and magnesium in their laboratory products[40] to make stills and other apparatus more stable. Given the international stature of the glass industry in the 18th and

19th centuries,[41] we can speculate that the techniques employed in producing special wares such as laboratory glass were known to Scottish glassmakers at that time.

Conclusion

The chemical composition of samples from the Playfair Collection and the Old College Quadrangle excavations was determined using portable X-ray fluorescence. The results demonstrate a range of compositions that can be divided into three broad categories: high lead, moderate lead, and high calcium. The original purpose of the analyzed items appears to be related to their chemical composition. The high-lead items, for example, are those that would probably have required a high degree of transparency and thermal resistance. The high-calcium items were probably used to store harmful chemicals, and thus they would have needed an enhanced degree of stability that greater proportions of calcium provide. This suggests that the glassmakers were proficient in determining these factors when they embarked on the production of laboratory ware. The purpose of the moderate lead glasses is more open to speculation; nonlead cullet may have been used in a lead glass melt, or perhaps they were made specifically to fulfill a purpose that is not known to us.

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FIGURE CAPTIONS

FIG. 1. Differential thermoscope, or "photoscope," from the Playfair Collection (cat. no. 1858.275.10).

FIG. 2. Bottle with a faucet, from the Playfair Collection (cat. no. 1858.275.34). This bottle contained a high proportion (30.94%) of lead oxide.

FIG. 3. Five of the bottles from the Playfair Collection (cat. no. 1858.275.39).

FIG. 4. Relationship between lead and arsenic levels in samples from the Playfair Collection (*top*) and the Old College Quadrangle excavations (*bottom*). An approximately linear relationship exists for samples in which the lead content is below 30 percent and the arsenic content is below 1.5 percent.

FIG. 5. Relationship between lead and calcium levels in samples from the Old College Quadrangle excavations. As the amount of lead decreases, the amount of calcium increases.

ABSTRACT

Glassware used in the laboratories at the University of Edinburgh in the 18th and 19th centuries was analyzed using portable X-ray fluorescence. The samples came from two sources: the Playfair Collection, an assortment of vessels and equipment currently in the care of the National Museums Scotland, and excavations at the university's Old College Quadrangle.

A high degree of commonality was observed between the Playfair and quadrangle samples. High-lead glasses contained over 30-percent lead oxide, along with small amounts of arsenic and potassium. High-calcium glasses contained over 20-percent calcium, alongside magnesium, aluminum, strontium, iron, titanium, and sulfur. Moderate-lead glasses contained less than 25-percent lead and also strontium and iron, which were not observed in the high-lead glasses, indicating that cullet from another glass type may have been used in their manufacture.

High levels of lead were found in items that require thermal stability and a high degree of transparency. High levels of calcium were seen in vessels that may have been used to hold and store chemicals, requiring a high degree of chemical stability.

These items suggest that Scottish glassmakers were capable of producing high-quality items of a relatively uncommon nature, using unusual ingredients in the melt.

FOOTNOTES

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