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A non-destructive analytical technique used for analyzing a variety of glassware samples in hopes to determine their elemental composition based off their qualitative and quantitative data results.

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A non-destructive analytical technique used for analyzing ancient glassware and pottery to determine their elemental composition

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Introduction

- X-ray fluorescence spectroscopy (XRF) was used in determining the elemental composition of old glass samples. The non-destructive analytical technique measures the fluorescent X-rays which are emitted from the material tested, once excited by a primary source. This technique is often used for its elemental analysis capabilities while examining metals, glass, and artwork such as paintings or pottery. XRF analysis is case specific to that of large samples and materials containing high abundances of elements, unlike rocks and minerals, that are generally analyzed by other techniques.
- In this study, XRF analysis was used to examine and identify specific glassware samples, exported from remote locations, in hopes to determine any correlation between the glass samples being tested. During experimentation, there were 41 glass samples and 3 glass standards that were examined and quantitatively analyzed to determine the elemental compositions of all samples. To ensure optimal sensitivity of these different materials, four methods were utilized to confirm accurate data is collected and to improve all quantitative data results. These four methods contain distinct experimental factors and were purposely selected due to their specific current, voltage, filter, time, and vacuum. The purpose of changing any of these experimental factors, specifically the voltage and filter, is to maximize sensitivity to the elements of interest.

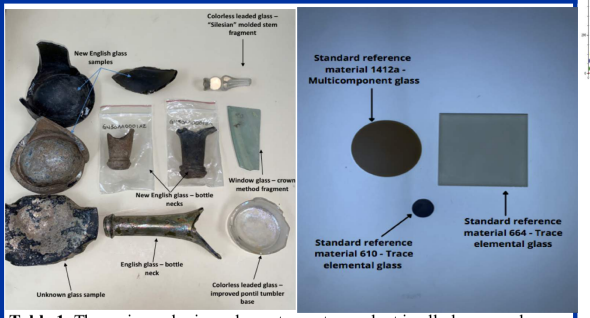


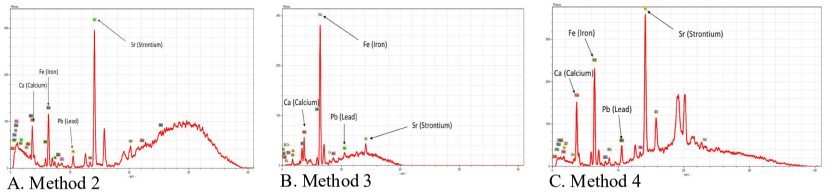
Table 1. The major and minor elements most prevalent in all glass samples. Typically, the major elements that were present in these glass samples could be used to help identify specific glassware.

Major elements	Minor elements
Si	Zn
Ca	Mn
As	Cr
Pb	Zr
Mg	Nb
K	Rb
Fe	Rh
Na	Sn
Sr	
Se	

Type of Instrumentation:

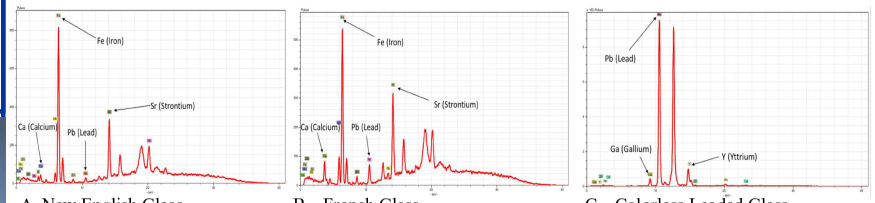
The image to the left provides a visual of the XRF analytical technique that allowed for qualitative and quantitative data results of all the glass samples utilized during experimentation.

Qualitative Results



A. Method 2 B. Method 3 C. Method 4

Figure 1. Methods 2,3, and 4 were used to determine the elemental composition of an old English glass sample. The methods used for analyzing each glass sample were selected based on the experimental factors that allowed specific groups of elements to be optimized.



A. New English Glass B. French Glass C. Colorless Leaded Glass

Figure 2. All three graphs of spectra utilized method 4 in all the different glass samples. Method 4 efficiently excited the elements from Ti to Ag K-lines and the W to Bi Lines.

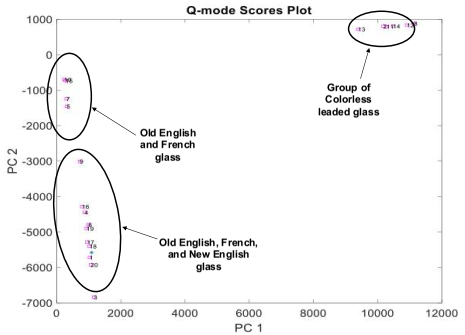


Figure 3. The principle component analysis plot above was used to distinguish our lead glass from our English and French glass. PCA was used to reduce the dimensionality of these large data sets into smaller ones, while still containing majority of the information in the large set.

Table 2. The table below contains all lead, English, and French glass items alongside their corresponding average Pb peak areas for all three methods. The intensity of each peak was used to determine the concentration of lead for each glass sample.

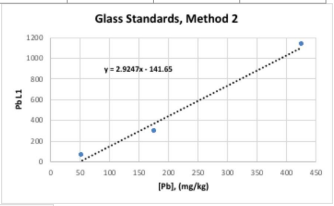
Glass Item	Average Pb (three methods)
CLG	27170
CLG	22098
CLG molded	17662
Window	700
CLG	20635
English	61
French	177
English	109
CLG	22515
CLG	27494
Green	684

Quantitative Results

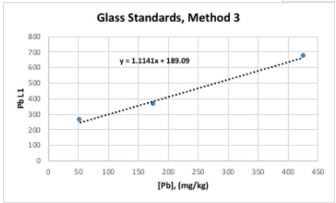
Table 3. The table below list all the lead glass items used during experimentation. The basic approach to XRF analysis was to utilize the area under the fitting peaks as a quantifiable value to determine the abundance of the element, Pb (lead). The concentrations of Pb were determined by using the given peak area values associated with each lead glass sample and the net peak areas (K_i) that were calculated using the given concentrations of Pb from all standard reference materials.

Lead glass items	M2 Pb	Pb, (mg/kg)	M3 Pb	Pb, (mg/kg)	M4 Pb	Pb, (mg/kg)
OBJ-17BA	36024.3	19396.911	13433.56	4686.1901	32053.17	7677.46313
OBJ-20AA	21586.58	11623.0703	13803.33	4815.18141	30905.38	3306.21144
OBJ-10FA	11073.01	5962.14748	11569.69	4035.99394	30343.7	2771.20386
OBJ-99ZA	not analyzed	not analyzed	12554.82	4379.64867	28714.47	6877.76856
OBJ-17DA	not analyzed	not analyzed	12929.48	4510.34582	32100.6	3096.90449
OBJ-03CA	37112.48	19982.8302	13155.18	4589.07946	32214.61	7716.13169

A. Glass standards 610, 664, 1412a – Method 2



B. Glass standards 610, 664, 1412a – Method 3



C. Glass standards 610, 664, 1412a – Method 4

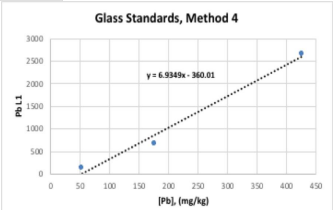


Figure 4. Scatter plots A,B, and C above all indicate a linear correlation between the net peak area of Pb Lα line and the amount of Pb in each glass standard.

Methods

- A total of 41 glass samples and three glass standards were analyzed using Bruker Tracer-III X-ray fluorescence spectrometer equipped with a rhodium target X-ray tube and a Si-PIN detector.
- All measurements were first performed using Method 1(LabRat Mode), with a X-ray tube voltage of 40 kV and current of 40 mA with a 12 mm Al/1 mm Ti/6 mm Cu filter at ambient pressure.

	Method 1	Method 2	Method 3	Method 4
Current (μV)	40	40	40	4.2
Voltage (kV)	40	40	15-20	40
Time (s)	60	60	60	60
Filter	None	Green	Blue	Yellow
Vacuum	None	None	None	None

Conclusions

Future work:

The goal of this research project was to determine the elemental composition of different glass samples and glass standards by using an analytical technique known as X-ray fluorescence spectroscopy (XRF). In terms of continuing future research, the goal is to become proficient in other analytical methods that are used to determine the elemental make-up of different glassware and other materials. As well as, incorporating other analytical techniques like inductively coupled plasma mass spectrometry (ICP-MS) and X-ray diffraction (XRD) to compare the effectiveness of those techniques to XRF analysis. The results will include comparing additional methods not used in this research project that aid to optimizing XRF elemental group analysis and comparing the efficiency of XRF spectroscopy to other regularly exercised techniques.