AN XRF COMPOSITIONAL ANALYSIS OF OPAQUE WHITE GLASS BEADS FROM 17TH-CENTURY MISSION SANTA CATALINA DE GUALE, GEORGIA

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Previous analyses of the elemental composition of white glass beads have shown that the opacifier used during glass manufacture is temporally diagnostic, with a transition from tin to antimony to arsenic to fluorine. To date, most researchers using this fact for chronological purposes have focused on British, Dutch, and French contact sites in the northeastern United States and Canada. Many of these studies have relied on expensive, and sometimes minimally destructive, techniques. X-ray fluorescence spectrometry is a widely available, non-destructive technique that can be used to identify glass opacifiers extremely rapidly and inexpensively. This technique was used to analyze 783 specimens of four varieties of drawn white glass beads from burial contexts at Mission Santa Catalina de Guale, Georgia, demonstrating that the "opacifer-dating" method is also applicable to Spanish colonial sites in the southeastern United States.

INTRODUCTION

Glass beads have long been one of the most important artifact classes available to historical archaeologists, allowing investigators to address questions of trade and economy, religion, adornment, and mortuary practices. As objects of personal adornment that circulated widely they have considerable interpretive potential (Spector 1976) and can be used to explore complicated issues of embodied identity and colonial relationships (Hamell 1983, 1987; Loren 2009, 2010; Turgeon 2004). Of a more foundational nature, one of the most common, and more critical, roles in archaeology that glass beads have served is that of chronological marker (e.g., DeCorse 1989; Little 2010; Smith 1983). This is somewhat paradoxical for, despite their importance for this purpose, "glass beads, by and large, are extremely hard to date, and the vast majority possess no distinguishing features...." (Noël Hume 2001:54). This lack of distinguishing features means that a variety of approaches have been utilized to explore the chronological potential of glass beads.

As recently outlined by Marcoux (2012), this includes studies that have utilized quantitative frequency seriations (e.g., Kent 1983, 1984; Polhemus 1987), more qualitative assemblage-level sequencing (e.g., Wray 1983), approaches that identify the circulation dates for specific beads types that can serve as index fossils (e.g., Smith 1983), and Marcoux's (2012) own multivariate correspondence analysis method. Despite the success of all these approaches, each is limited by an inability to fully utilize the "non-diagnostic" beads – often with long periods of circulation – that tend to dominate most archaeological assemblages. Indeed, Marcoux (2012) excluded simple seed beads from his seriation "because they compose such a significant portion of every assemblage that they drown out the chronologically significant variability in the other bead types." These "non-diagnostic" bead types, however, are actually a largely untapped resource for dating purposes, underscoring Marvin Smith's (2002:60) observation that "the full potential of glass beads as chronological indicators has scarcely begun to be realized.

One approach that has emerged in recent decades that has both increased the potential of beads as chronological indicators and increased the utility of "non-diagnostic" types is the use of compositional analyses (e.g., Bonneau et al. 2014; Hancock 2005, 2013). As Kenneth Kidd (1983:3) noted, compositional analysis, combined with archaeological and archival investigations, is essential to illuminating past networks of bead manufacture and exchange – particularly compositional approaches that facilitate very large sample sizes and non-destructive approaches (*see also* Sprague 1985:100).

This article explores this potential by discussing the results of an x-ray fluorescence spectrometry (XRF) analysis of drawn white glass beads recovered from the cemetery of Mission Santa Catalina de Guale (SCDG), a 17th-century Franciscan mission located on St. Catherines Island, Georgia. This includes Kidd and Kidd (2012 [1970]) varieties IIa13, IIa14, IVa11, and IVa13. I begin by

arguing that XRF is an ideal method for non-destructively identifying chronologically significant glass opacifiers in large numbers of beads. I follow this by reviewing the evidence that supports the use of glass opacifiers as chronological markers, emphasizing both archaeometric and historical evidence. I conclude by presenting the results of the SCDG analyses, discussing how the compositional data neatly articulate with previous date estimates based on bead stylistic attributes (e.g., Smith 1983).

X-RAY FLUORESCENCE SPECTROMETRY AND GLASS BEADS

Numerous methods have been productively used for the compositional analysis of archaeological glass (for an overview of these methods, *see* Bonneau et al. 2014), including instrumental neutron activation (INAA) (e.g., Davison 1972; Glascock 2013; Hancock 2005; Hancock et al. 1994; Kenyon et al. 1995), laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) (e.g., Dussubieux et al. 2009; Gratuze 2013; Popelka et al. 2005; Robertshaw et al. 2010; Walder 2013), proton induced x-ray emission (PIXE) (e.g., Biron and Verità 2012; Gan et al. 2009; Kuisma-Kursula 1999; Šmit et al. 2012; Zucchiatti et al. 2007), and XRF (e.g., Karklins 1983; Hoffmann 1994; Polikreti et al. 2011; Shugar and O'Connor 2008; Veiga and Figueiredo 2002). The different techniques have various advantages and disadvantages, including relative cost, availability, destructiveness, sensitivity, and range of detectable elements (Bonneau et al. 2014).

For example, while INAA has been productively used for bead analysis and has excellent precision, accuracy, and sensitivity to many elements, several important elements for interpreting glass chemistries cannot be easily determined. These include lead, phosphorus, and bismuth. Both lead and phosphorus are important opacifying ingredients (Moretti and Hreglich 2013) and bismuth can be used as a marker for identifying the location of raw material procurement (e.g., Soulier et al. 1996). Additionally INAA is expensive, there are few research facilities where this analysis can be conducted, and as a bulk analytical technique, the method is not appropriate for multi-colored compound and complex bead varieties.

LA-ICP-MS solves some of these difficulties, for example, having the capability to identify lead, phosphorus, and bismuth concentrations. The technique is also more readily available than INAA and can perform spot analyses on compound and complex beads. The technique, however, is still relatively expensive, time consuming, and is minimally destructive to the archaeological specimen.

Like these other techniques, XRF[1] also has its limitations (*see* Hunt and Speakman 2015; Shackley 2010; Shugar 2013; Speakman et al. 2011; Speakman and Shackley 2013). For example, XRF is primarily a surface-only analysis, meaning glass corrosion can be a significant hurdle (Kaiser and Shugar 2012). Like INAA, XRF is also a bulk analytical technique and thus is not appropriate for multi-colored beads. Additionally, depending on instrumental parameters, XRF can have difficulty detecting many low-Z elements, including many elements important in the manufacture of glass, such as sodium and magnesium. The method also requires the creation of custom empirical calibrations using matrix-matched reference standards in order to obtain quantitative results. These, however, are extremely challenging to create because of the limited numbers of glass certified reference standards. Additionally, while many portable XRF instruments offer out-of-the-box fundamental parameters approaches to quantification, the results produced using these methods have not been shown to be valid or reliable for archaeological research purposes (Dybowski 2012; Hunt and Speakman 2015).

Despite these limitations, the use of XRF has a number of benefits. First, the instrumentation is becoming increasingly available, resulting in extremely low analytical costs. These reduced costs, along with very rapid data collection (about three minutes per sample) allow increasingly large sample sizes to be analyzed. Second, the analysis is completely non-destructive, an important consideration when one is working with museum specimens or collaborating with descendent communities.

XRF was selected for this project for all of the reasons just discussed: the need for non-destructive analysis of funerary objects, and the low cost and speed of analysis allowing for a large sample size (n=783). Additionally, the quantitative calibration concerns were side-stepped by focusing on an analytical question that could be addressed entirely by presence/absence information: what element was used to make each bead opaque?

GLASS OPACIFIERS AS TEMPORAL MARKERS

The compositional analysis of glass beads identifies the elemental content of the glass used to manufacture them, generally dividable into glass formers (e.g., silicon), modifiers and stabilizers (e.g., sodium, potassium, calcium), colorants (e.g., copper, cobalt, manganese), and opacifiers (e.g., tin, antimony, arsenic, fluorine). Patterned variation of these ingredients can successfully be used to identify place of manufacture and source of raw materials, and the variability in these elements often also has temporal implications. The

difficulty, however, in making temporal inferences from glass composition is that many glass ingredients vary according to the vagaries of raw material source and the practices of specific regional glass houses (Blair 2015a, 2016; McCray 1999a, 1999b); isolating compositional variability that primarily represents chronology, rather than one of these other factors, is profoundly difficult (Blair 2015a, 2016; Fitzgerald et al. 1995). The one exception to this – for beads of European manufacture dating to the 16th-19th centuries – is the glass opacifier. Numerous lines of evidence, both archaeometric and documentary, indicate that the choice of opacifier is independent of place of manufacture or raw material source.

Archaeometric Analyses of Glass Bead Opacifiers

The general sequence of glass opacifiers used in European glass production has been generally understood since the early analyses of Turner and Rooksby (1959, 1963) and Rooksby (1962). During the era of Roman glassmaking, calcium antimonate was the primary opacifier before being completely replaced by the use of a lead-tin calx, perhaps as early as the 12th century (Tite et al. 2008; Verità 2014). Lead-tin then remained the primary glass opacifier until sometime during the 17th century when it was replaced by antimony-based opacifiers, including both lead antimonate and calcium antimonate. Later, antimony was replaced by arsenic and then fluorine. Bone ash (calcium phosphate) has also been documented as an opacifier from the 14th century onward (Moretti and Hreglich 2013).

This sequence was first recognized as applicable to glass beads in a series of pioneering articles by Ron Hancock and his colleagues (Hancock 2005, 2013; Hancock et al. 1997, 1999; Moreau et al. 2002, 2006; Sempowski et al. 2000). In their original study, they analyzed 284 beads from 15 archaeological sites in Ontario thought to have short occupation periods (Hancock et al. 1997). They concluded that antimony replaced tin sometime in the late 17th century and that arsenic first appeared during the late 18th century. Fluorine was found to be an ingredient during the 19th and 20th centuries. In a subsequent study, a similar sequence was established for the Seneca region of New York (Sempowski et al. 2000). They postulated that the transition from tin to antimony was a gradual process, with both tin and antimony beads in circulation between 1625 and 1675, with tin finally disappearing as an ingredient by 1675.

A number of subsequent studies have produced compositional data for white glass beads (e.g., Bonneau et al. 2013; Dussubieux and Karklins 2016; Shugar and O'Connor 2008; Walder 2015). Table 1 synthesizes the data derived from a number of such studies. Samples included in the table were selected to include sites from the Americas and from European manufacturing locales, with a strong preference for studies that specifically note the bead type and glass color being analyzed. This synthesis of white bead opacifier data is useful for refining our understanding of the timing of glass bead opacifier transitions. Indeed, such refinement is needed because many of the studies just mentioned rely primarily on the archaeological *site* as the unit of analysis. Such coarse-grained resolution may well mask finer temporal variability across a site. For example, Sempowski et al. (2000) document both tin and antimony-rich beads on sites dated to 1625-1640, 1640-1655, and 1655-1675, and suggest that this correlates with a slow transition from tin to antimony during this period. Recently, however, Marcoux (2012) has argued that individual burial contexts are a better unit of analysis for refining the chronological resolution of glass beads, mitigating the confounding issue of occupational palimpsests (*see* Polhemus 1983, 1987).

Evidence from Recipe Books

Besides archaeometric data, one of the most important sources available for helping to interpret the evidence obtained via glass compositional analysis is glass recipe books. During the 16th and 17th centuries, glassmaking was primarily a skill that was learned and perfected through practice and experience (McCray 1999a, 1999b). This was primarily due to two factors. First, glassmaking and beadmaking during the 16th and 17th centuries was controlled by manufacturing guilds, operating within an apprenticeship system (Trivellato 2006). Second, the guild system was incredibly secretive and trade secrets were vigorously protected. While individual glass houses maintained internal recipe books, with few exceptions (e.g., Neri 1612, 1662 [1612]) these were not published for public consumption.[2] Many of these books, however, have been preserved and subsequently published, providing important insights into glassmaking practices, recipes, and ingredients and how they changed over time (Moretti and Hreglich 1984, 2013; Moretti et al. 2005).

Three recipe books in particular provide important evidence of glassmaking practices during the 16th and 17th centuries (Moretti and Hreglich 2005, 2013; Toninato and Moretti 1992). The chronologically earliest of these is an anonymous Venetian manuscript, initially transcribed by Moretti and Toninato (2001) and recently published and annotated in English (Watts and Moretti 2011). The volume was likely originally assembled between 1536 and 1567, and might be a copy of somewhat earlier recipes. The next is the aforementioned Neri volume, written by the Florentine

Table 1. Chronological Sequence for Opacifiers in White Glass Beads.

Site	Approximate Date	Location	Number of Samples				Source
			Sn	Sb	As	Other	
Middelburg	late 16th - early 17th centuries	Netherlands	4	1			Karklins et al. 2001
Cameron	1595-1610	Eastern Seneca, NY	8				Sempowski et al. 2000
Smith-Saeger	1600-1625	Ontario	4				Hancock et al. 1997
Chicoutimi	1600-1625	Quebec	x				Moreau and Hancock 2010
Dutch Hollow	1610-1625	Western Seneca, NY	27				Sempowski et al. 2000
Factory Hollow	1610-1625	Eastern Seneca, NY	5				Sempowski et al. 2000
Auger	1615-1630	Ontario	91				Hancock et al. 1997, 1999
Asd/Kg9; Kg10	1621-1657	Amsterdam	45	5			Bradley 2014; Karklins et al. 2002
Warren	1625-1640	Eastern Seneca, NY	10	5			Sempowski et al. 2000
Cornish	1625-1640	Eastern Seneca, NY	5	5			Sempowski et al. 2000
Bosley Mills	1625-1640	Western Seneca, NY	1	1			Sempowski et al. 2000
Hammersmith Embankment	1625-1650	London, England	8				Dussubieux and Karklins 2016; Karklins et al. 2015
Train	1625-1650	Ontario	2				Hancock et al. 1997
Orchid	1625-1650	Ontario	17				Hancock et al. 1997
Tipu	pre-ca. 1638-1641	Belize	x				Hancock and Graham 2006
Steele	1640-1655	Eastern Seneca, NY	5				Sempowski et al. 2000
Power House	1640-1655	Western Seneca, NY	5	5			Sempowski et al. 2000
Menzis	1640-1655	Western Seneca, NY		3			Sempowski et al. 2000
Dann	1655-1675	Western Seneca, NY	13	9			Sempowski et al. 2000
Marsh	1655-1675	Eastern Seneca, NY	2	17			Sempowski et al. 2000
Gillett Grove	17th century	Iowa		1			Walder 2015
Mormon Print Shop	17th century	Michigan		1			Walder 2015
Bead Hill	1670-1690	Ontario		2		2	Hancock et al. 1997
Beale	1675-1687	Eastern Seneca, NY		5			Sempowski et al. 2000
Boughton Hill	1675-1687	Eastern Seneca, NY		32			Sempowski et al. 2000
Rochester Junction	1675-1687	Western Seneca, NY		10			Sempowski et al. 2000
La Belle	1686	Texas		6/3			Walder 2015; Perttula and Glascock 2017
Snyder/McClure	1690-1710	Western Seneca, NY		15			Sempowski et al. 2000

Table 1. Continued.

Site	Approximate Date	Location	Number of Samples				Source
			Sn	Sb	As	Other	
Premier Palais	1700-1750	Quebec		2			Moreau et al. 2006
Dorion	1700-1800	Ontario		5			Hancock et al. 1997
Ashuapmushuan	1700-1800	Quebec		344			Moreau et al. 2002
Fort Michilimackinac	ca. 1715-1761	Michigan		11			Walder 2015
Magasins du Roy	ca. 1750-1760	Quebec		45			Moreau et al. 2006
Old Fort Niagara	mid-18th century	New York		324		6	Shugar and O'Connor 2008
Armours Point	1750-1800	Eastern Great Lakes		8			Hancock et al. 1997
Moose Factory III	1760-1850	Ontario		20	5		Hancock et al. 1997
Fort St. Joseph	1796-1814	Ontario		19	8		Hancock et al. 1997
Fort Malden	1797-1813	Ontario		8	9		Hancock et al. 1997
Sullivans Island	late 18th - late 19th centuries	Washington		x	x		Burgess and Dussubieux 2007
Fort Malden	1813-?	Ontario		15	6		Hancock et al. 1997
Dewar	ca. 1830	Ontario		3	12		Hancock et al. 1997
Camp Kitchi	1836-1856	Ontario		4	2	7 (bone ash?)	Hancock et al. 1997
Mohawk Village	1840-1860	Ontario		0	8		Hancock et al. 1997
Moose Factory I	1850+	Ontario		4	12	2	Hancock et al. 1997
Modern Souvenir	1903-1926	Ontario			5		Hancock et al. 1997

priest Antonio Neri and originally published in 1612 (Neri 1612). The final volume is the Darduin recipe book (Zecchin 1986). This volume contains several sets of recipes primarily compiled by Giovanni Darduin, a Muranese glassmaker and a later, unknown individual. The first section of the volume contains 16th-century recipes attributed to Giovanni's father, Nicolò Darduin (d. 1599), as well as Giovanni's own recipes that he continued to add to the volume until ca. 1654. Giovanni also transcribed and included an additional set of recipes from an anonymous 1523 document. The final portion of the manuscript, in different handwriting, was added by an unknown individual between 1693 and 1712 (Verità 1986).

These recipe books are particularly important for documenting different practices for producing opaque glass, including the use of tin dioxide, calcium antimonate, lead antimonate, lead arsenate, and bone ash (calcium phosphate). For my purposes, the temporal change in glass opacifiers is particularly relevant to this discussion. The primary opacifier for Venetian glasses, from the 14th century until the early- to mid-17th century, was tin dioxide, generally added to the glass mixture as calcined lead and tin. Three recipes in the anonymous Venetian manuscript describe the manufacture of white glass using this process (Watts and Moretti 2011:22), and the technique is repeatedly mentioned by Neri (2003 [1612], 2004 [1612], 2007 [1612]) and included in the Darduin manuscript.

At some point during the 17th century, however, the use of a lead-tin opacifier ceased and antimony-based opacifiers (both calcium antimonate and lead antimonate) came into use. For example, only one recipe in the 16th-century anonymous Venetian recipe book discusses opacification using calcium antimonate, and that recipe (XXXVI) is for an unusual silver mosaic glass (Watts and Moretti 2011:64). While calcium antimonate had been used as an opacifier in Roman times (Mass et al. 1996; Rooksby 1962; Turner and Rooksby 1959, 1961, 1963), with few exceptions it does not appear at all in the early Venetian recipe books. Antonio Neri

(2004 [1612]) only mentions its use for chalcedony glass and other specialized glasses, not as an opacifier. The Darduin manuscript contains the first mention (recipe CXLIV) of an opaque glass manufactured with lead antimonate in a recipe that dates to the mid-17th century. Commenting on this, Zecchin (1986:182 [translation mine]) states:

> This and the following are the first two recipes that use antimony as opacifiers in the glass in place of the traditional calc of lead and tin. As also indicated in the recipe, this substitution was dictated, rather than to improve the quality of the product, for economic reasons, probably because of the high cost of tin at the time. Antimony was a new component for the Venetian glass, which he had not used at least until the beginning of the 17th century.

Other glass opacifiers mentioned in the documents include bone ash (Watts and Moretti 2011), indicated by a high phosphorus content, and lead arsenate. The latter was first noted in a recipe dating 1 June 1693 (Zecchin 1986).

GLASS BEADS OF MISSION SANTA CATALINA DE GUALE

Santa Catalina de Guale was a Franciscan mission located on St. Catherines Island, Georgia. Following several sporadic, and generally failed, missionization attempts during the 16th century – most notably the 1595-1597 mission that was destroyed during the 1597 Guale rebellion (Blair and Thomas 2014; Francis and Kole 2011) – Santa Catalina was firmly established in its archaeologically known location by 1605. The mission was in operation until 1680 when, under attack from the British-allied Westos, the site was abandoned and the community relocated southward to Sapelo Island (Worth 2007, 2009a, 2009b). The original location of Mission Santa Catalina at Wamassee Head on St. Catherines Island was conclusively identified by David Hurst Thomas and the American Museum of Natural History in 1981, and over the next decade a number of structures were excavated, including the mission church, friary, and kitchen (Thomas 1987, 1988a, 1988b, 1993, 2010a).

Excavations beneath the floor of the church by Clark Spencer Larsen (1990) revealed the mission cemetery which contained a minimum of 431 Guale neophytes. These individuals were all buried in Catholic fashion: supine, feet oriented towards the altar, and arms crossed over the chest or abdomen. Almost all burials appear to have been interred in a simple shroud cloth; with few exceptions, coffins were absent. Recovered with these burials was an unusually large assemblage of grave furnishings, including whole majolica vessels, bells, chunky stones, Catholic devotional medals, religious medallions, finger rings, and nearly 70,000 trade beads (Blair et al. 2009; Thomas 1988a, 2010b).

The beads excavated at Mission Santa Catalina were primarily made of glass but also jet, amber, carnelian, and rock crystal. These objects were manufactured around the globe, likely including Venice, Amsterdam, Bohemia, China, India, and the Baltic region (Blair 2015a; Blair et al. 2009). Because of the historically well-documented dates for the mission cemetery (ca. 1605-1680), studies of this bead assemblage have generally focused on questions of origins, manufacture, exchange, and social networks, rather than chronology (Blair 2015a, 2015b, 2016, 2017). Lingering questions, however, about changing burial practices throughout the mission period (McEwan 2001; Thomas 1988a) have prompted more sustained examinations of bead chronology at Mission Santa Catalina.

Bead Chronology at Mission Santa Catalina

The beads recovered at Mission Santa Catalina have been previously evaluated for their temporal potential (Blair 2009:157-159). At that time, while also commenting on the possibilities of compositional analysis for dating purposes, several observations were made about specific bead types present in the assemblage:

1) Numerous eye beads (Kidd and Kidd type IIg) are present in the SCDG assemblage. Smith (1987:33) argues that these no longer circulated in Spanish-colonial contexts after ca. 1630.

2) Many charlottes (faceted seed beads; Kidd and Kidd IIf) are also present in the assemblage and also appear to date no later than the early 17th century (Smith et al. 1994:39).

3) Several blue beads with red-on-white stripes (IIbb24, IIbb27) are found in the SCDG assemblage. Kidd variety IIbb24 does not appear in the Susquehanna sequence until 1718-1743, while the very similar variety IIbb27 dates from 1575-1600 (Kent 1983:80-81).

4) A cobalt-blue bead with alternating red-and-white stripes (IIb71) is thought to be diagnostic of the early 17th century (Smith 1983:150, 1990:223).

5) Seed beads of compound construction, found in the thousands at SCDG, are most common from 1600 to 1630 (Smith 1987:33).

Despite the presence of a number of bead varieties in the SCDG assemblage that evidence suggests date earlier than 1630, they are relatively scarce compared to the enormous quantities of non-diagnostic beads of simple construction (e.g., IIa7, IIa13, IIa40, IIa55). Additionally, many of the temporally diagnostic compound and complex

varieties are restricted in distribution to only a handful of the presumed high-status burials in the mission cemetery. That is, the majority of individuals buried in the cemetery were not found with temporally diagnostic bead varieties. This was not unexpected. Smith (1987:33) notes that there are no bead varieties that are temporally diagnostic for the 1630-1670 period. This does not, however, mean that burials found with only a few beads of non-diagnostic types must date to the period 1630-1670. Such burials could easily date to any time during the 1605-1680 period when Mission Santa Catalina was in use.

COMPOSITIONAL ANALYSIS OF MISSION SANTA CATALINA BEADS

In 2007, I initiated a project to examine the elemental composition of the glass beads recovered at Mission Santa Catalina. While this project was initially designed to evaluate hypotheses about the production origins of certain bead varieties found at SCDG, particularly several hypothesized to have been manufactured in Bohemia and China (Francis 2009a:100, note 3, 2009b:84, note 8), it later developed into a broader study of the circulation and consumption of beads at the mission, using glass composition as a key metric in the identification of distinct social networks at SCDG (Blair 2015a, 2015b, 2016, 2017). Throughout, however, the project has evaluated the possibility of using elemental composition to refine our understanding of the SCDG chronology.

XRF Analysis of White Beads: Methods and Materials

The beads analyzed in this study consist of simple and compound white beads of Kidd varieties IIa13, IIa14, IVa11, and IVa13. All are of drawn manufacture.

IIa13 (AMNH Type 23; n=180). Of simple construction, these are opaque white (2.5 PB 10/0, 4.7Y 9/4, 4.5Y 9/1) and includes barrel, olive, oval, and spherical specimens. The beads are 3.51-7.99 mm in diameter and 2.51-13.0 mm in length. They were likely manufactured by members of the Paternostri guild in Venice; possibly also in France and the Netherlands (Francis 2009d, 2009e).

IIa14 (AMNH Type 15; n=33). Of simple construction, these are opaque white (4.5Y 9/1, 2.5PB 10/0) and ring shaped. They are 2.60-3.50 mm in diameter and less than 2.51 mm in length. This variety is thought to have been primarily manufactured in Venice by members of the Margareteri guild and are often referred to as simple white seed beads (Francis 2009c).

IVa11 (AMNH Type 38b; n=149). Of compound construction, these are composed of opaque white (4.5Y 9/1, 2.5PB 10/0) glass sandwiched between a transparent colorless core and a thin, clear exterior coat. The beads are ring and barrel shaped, ranging from less than 2.60 to 7.99 mm in diameter and from less than 2.51 mm to 4.50 mm in length. This variety, erroneously combined with type IVa13 in Blair et al. (2009), has been suggested to date to the period 1560-1630 (Smith n.d.). It is thought to have been manufactured by the Margareteri beadmaking guild in Venice (Francis 2009c).

IVa13 (AMNH Type 38a; n=421). Of compound construction, this variety is made of an opaque white (4.5Y 9/1, 2.5PB 10/0) glass with a transparent colorless core. In some specimens the white glass is heavily eroded and has developed a light yellow (4.3Y 9/7) hue. The beads are highly unstable and the opaque layer has eroded completely in some cases, leaving a separated core. The beads are ring and barrel shaped, ranging from less than 2.60 mm to 7.99 mm in diameter and from less than 2.51 mm to 4.50 mm in length. This variety was erroneously combined with type IVa11 in Blair et al. (2009). Smith (n.d.) suggests that this specific drawn, white, compound configuration post-dates 1630. It is thought to have been manufactured by the Margareteri beadmaking guild in Venice (Francis 2009c).

The elemental analysis of a sample of these beads was carried out using an evolving, multi-technique strategy. Samples were selected from all burial contexts with opaque white beads. For burial contexts with large numbers of beads, up to 50 specimens of each variety were selected for analysis. The initial analysis of the beads (n=783) was conducted using a Bruker Tracer III-V portable x-ray fluorescence spectrometer. Each bead was analyzed under vacuum for 180 seconds at 40 kV and 3 μA using a 0.001" Cu, 0.001" Ti, 0.012" Al filter. This analysis yielded spectral data for elements K, Ca, Ti, Mn, Fe, Co, Ni, Cu, Zn, As, Sr, Sn, Sb, and Pb. Net area under the peak values for each element were then extracted from each bead spectra in Artax 7, using a Bayesian deconvolution and Gaussian curve fitting method. These values were then exported to JMP 11 for exploratory data analysis. Additional compositional analyses using laser ablation - inductively coupled plasma - mass spectrometry (LA-ICP-MS)[3] were also conducted on a subsample of the SCDG beads in order to confirm the patterning identified via XRF analysis (Blair 2015b).

XRF Analysis Results

The XRF analysis of the Mission Santa Catalina white glass beads indicates that only two opacifiers were used to opacify them: 288 beads were opacified with lead-tin and 495 with calcium antimonate (Table 2). No beads were opacified with lead antimonate, lead arsenate, or bone

Table 2. Distribution of Bead Opacifiers in the Mission Santa Catalina Cemetery.

Individual / Burial	IIa13		IIa14		IVa11		IVa13		Additional Temporal Data
	Pb-Sn	Sb	Pb-Sn	Sb	Pb-Sn	Sb	Pb-Sn	Sb	
47	2	2							None
65	1								Bead types IIbb27 and IIb56 (pre-ca. 1630) present
70					37		1		None
77								22	None
86	1							50	None
88								2	Burial intrudes into Ind. 383
90		13						37	None
93								52	None
102				14				37	None
107								5	None
127		1							None
134								1	None
138	4								1 type IIf bead (pre-1630)
139 / 140				6				11	None
142		1						1	None
163		15						3	None
186	14								None
207					1				Stratigraphically earlier than Ind. 208
208	2				2				Numerous IIf beads, plus compound and complex varieties present; Ictucknee blue-on-white bowl, 1600-1650 (Deagan 1987:64-65)
212 / 218				1	1				None
226				10					Post-dates Ind. 228
228				2					Pre-dates Ind. 226
238		1						14	None
243								50	None
276		19						23	Dominated by wound varieties; stratigraphically late burial
282					18				Numerous IIf beads, plus compound and complex varieties
295 / 296								50	None

Table 2. Continued.

Individual / Burial	IIa13		IIa14		IVa11		IVa13		Additional Temporal Data
	Pb-Sn	Sb	Pb-Sn	Sb	Pb-Sn	Sb	Pb-Sn	Sb	
307	51				54		10		Stratigraphically earlier than Ind. 208; numerous IIf and compound varieties; pre-ca. 1630/40 and pre-ca. 1650 ceramics
318	30								Found with numerous pre-1630 artifacts
348 / 349 / 350	4				5				Found with numerous pre-1630 artifacts
363 / 364	5								None
383								2	Pre-dates Ind. 88
394								50	None
Burial B	14				30				Stratigraphically pre-dates Ind. 307 and 208; numerous faceted (IIf), compound, and and complex varieties
Burial E					1				Numerous eye (IIg) and complex (IIb56) beads
Total	**128**	**52**	**0**	**33**	**149**	**0**	**11**	**410**	**783**

ash. Figure 1 shows the spectral difference between these opacifiers. The spectrum in black shows the characteristic lead and tin peaks, while the spectrum in gray shows the distinct signature of antimony, indicating the ease with which bead opacifiers can be identified using XRF. All the analyzed beads fall into one of these categories with no evidence of mixed opacifiers or glass recycling, though the concentrations of the opacifying elements vary widely. This variation is attributable to bead variety. Beads of simple construction (e.g., IIa13) exhibit the highest elemental concentrations. The reduced amounts of tin and antimony found in compound varieties IVa11and IVa13 are the result of opacifier "dilution" caused by the combined bulk analysis of white opaque glass and the non-opacified clear glass layers.

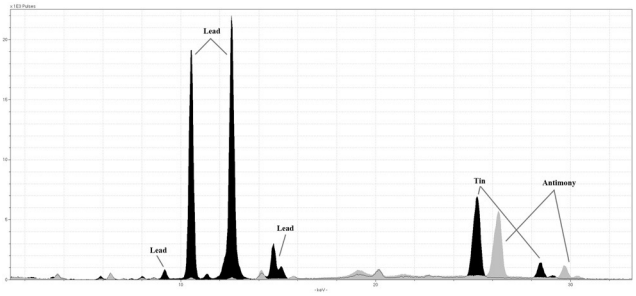

Figure 1. XRF spectra of two IIa13 beads from Mission Santa Catalina. The spectrum in black represents lead and tin; that in gray represents antimony.

Additionally, as I have explored elsewhere (Blair 2015a, 2015b, 2016), there is also considerable patterning in the SCDG XRF data that is not linked to opacifier choice (e.g., strontium, potassium, manganese, and iron). This variation, however, seems to have no clear and sustained relationship to chronology, but instead is primarily related to raw material sources and the specific practices of individual glass houses.

An interesting pattern emerges from the data in terms of bead variety. First, while IIa13 and IVa13 beads are split between the two opacifiers, all analyzed IIa14 bead are opacified with antimony and all IVa11 beads are opacified with lead-tin. This is consistent with Smith's (n.d.) observation that compound white glass beads with thick clear layers (IVa11) date earlier than those with thick white layers (IVa13).

The data presented in Table 2 also have significant temporal implications. With the exception of individual no. 47, found with both lead-tin and antimony beads, the remaining burials all have single-opacifier assemblages.[4] Indeed, the lack of mixed-opacifier assemblages in the Mission Santa Catalina cemetery strongly suggests that the transition from lead-tin to calcium antimonate was a relatively rapid process (*see* discussion below). Looking at Table 2, it is also clear that all burial contexts found with bead varieties and other artifacts dating prior to 1630 are

only found with beads opacified with lead-tin, supporting the temporal transition discussed earlier.

DISCUSSION

The analysis presented here has several important implications – some methodological, some specifically for Mission Santa Catalina and the southeastern United States, and some for thinking about bead chronology and interpreting the temporal position of "non-diagnostic" bead types more generally. First, the analysis demonstrates that XRF can be a highly effective technique for identifying the presence/absence of specific bead opacifiers. Because beads can be relatively dated based on the presence/absence of different opacifiers, XRF is an appropriate method for non-destructively analyzing very large samples at very low costs. While XRF, like all analytical techniques, has its limitations, the speed, cost, and non-destructive capability make it an excellent choice for this purpose.

This analysis also establishes that the opacifier chronology established at Northeastern archaeological sites (Sempowski et al. 2000) is also applicable to Spanish contexts in the Southeast. This is no surprise. All drawn beads circulating in North America during the 16th and 17th centuries likely came from the same manufacturing centers, primarily Venice (Karklins 2012:81). During the 17th century, an extensive trade in Dutch-made beads also occurred in both the Northeast and Southeast, though these products are difficult to distinguish and are largely derivative of their Venetian counterparts (Baart 1988; Francis 2009d; Hulst et al. 2012; Karklins 1974, 1983; Kenyon and Fitzgerald 1986; van der Sleen 1963a, 1963b).

Establishing the opacifier sequence as temporally valid for the Southeast also has other significant implications for understanding bead chronology in the region. Most importantly it provides another line of evidence that supports Smith's pioneering bead chronology (Smith 1983, 1987). As is evident in Table 2, burial contexts containing bead varieties that Smith dated prior to 1630 are *only* found in association with lead-tin opacified white beads. Some assemblages with lead-tin beads, however, lack beads diagnostic of the earlier period. Does this indicate that the lead-tin opacifier post-dates 1630, or is this merely indicative of the smaller quantities of good index fossil bead types in circulation? This raises the important issue: when and why did the transition from lead-tin to antimony occur?

Regarding the why question, Sempowsi et al. (2000) suggest the change was related to either the availability or cost of tin. Social and functional reasons for the change are also possible, but it seems probable that economics are the most likely factor. Hancock (2013:464) has noted that the amount of tin used in opaque white glass decreased over time, as glassmakers realized that lesser quantities were sufficient to produce opaque glass. This same pattern has also been documented for opaque turquoise-blue glass in the Southeast (Dalton-Carriger and Blair 2013, 2015). Similarly, as discussed above, Luigi Zecchin's (1986) analysis of the recipes in the Darduin manuscript suggests that the expense of tin likely led to its replacement by antimony.

If cost and economic concerns are the reason for the opacifier change, then it is highly significant that the ca. 1630 bead stylistic changes noted by Smith (1983, 1987), specifically the general disappearance of many complex and compound bead varieties, correlates with the documented use of a cheaper opacifier (Table 2). The trend toward simple beads and cheaper ingredients is consistent with an industry looking to cut costs in the production of inexpensive trade goods for colonial markets.

While Sempowski et al. (2000) suggest that there was a gradual transition in opacifier use, based upon several sites in the Northeast with mixed assemblages, the data from Mission Santa Catalina suggests a more rapid transition, perhaps as early as ca. 1630. The absence of burials possessing beads of both opacifier types suggests that lead-tin and antimony beads were not circulating simultaneously at SCDG. Additionally, stratigraphic relationships between burial pits at SCDG indicate that no burials with lead-tin-opacified beads are intrusive into burials with antimony-opacified beads. That is, all stratigraphically intersecting burial pits are consistent with the lead-tin to antimony transition.

A 1630 date for the opacifier transition is also largely consistent with the meta-analysis presented in Table 1. Several sites, however, do cause problems for this hypothesis; primarily the Steel and Marsh sites in the Eastern Seneca sequence and the Power House and Dann sites in the Western Seneca sequence. The high number of tin-opacified beads at the Dann site (ca. 1655-1675) in particular is problematic for this interpretation. How do we account for mixed assemblages found at sites spanning several decades in the Northeast? Perhaps the transition was indeed gradual but, of course, multiple site components, heirlooming, and the circulation of older beads could easily account for the presence of mixed assemblages, even over several decades. More likely, however, I suspect the issue will resolve itself if, as suggested by Marcoux (2012:159), short-duration contexts, rather than sites, are used as the primary unit of analysis in order to better establish bead contemporaneity. Additionally, some of the later sites in the Seneca sequence have not yet had the intensive temporal reevaluations that the earlier sites have had (Saunders and

Sempowski 1991; Sempowski and Saunders 2001; Wray et al. 1987, 1991). Such reanalysis could help clarify the timing of this transition.

CONCLUSION

The elemental composition of glass beads is an important but underutilized method for extracting chronological information from archaeological sites. XRF, in particular, as a fast, cheap, and non-destructive technology that can provide large samples of compositional data to sequence archaeological sites and features, should be more extensively utilized. At Mission Santa Catalina de Guale, the use of XRF on a large sample of white glass beads demonstrates that the opacifier sequence identified at Northeastern archaeological sites and in historic glass recipe books is also applicable to Spanish colonial sites in the Southeast. Additionally, while not fully explored here, the bead compositional data from the mission has significant potential for exploring temporal and social patterns within the site (Blair 2015a, 2015b, 2016, 2017). Indeed, the large sample sizes made accessible through the use of XRF should expand the possibilities of using glass beads to explore micro-scale intra-site patterns.

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ENDNOTES

1. In this discussion, I have chosen not to distinguish between the use of benchtop ED-XRF instruments and portable XRF. Although there are, of course, some practical differences between instruments, both "are subject to the same limitations... particularly with respect to sample preparation, instrument calibration, and ability to accurately quantify low-Z elements" (Hunt and Speakman 2015:626).

2. The importance of Antonio Neri's (1612) work for European glassmaking is widely acknowledged and is highlighted by the enormous number of editions and translations that have appeared over the last few centuries (Boer and Engle 2010; Engle 2014; Grazzini 2012; Turner 1963). At the same time, this complex and extensive sequence of editions and translations – beginning with Christopher Merret's 1662 English translation (Neri 1662 [1612]) – has actually resulted in an under-appreciation for the importance of Neri's writing for the history of beadmaking due to the repeated mistranslation of specific beadmaking terminology. Dillon (1907:183, n.1), for example, notes that *canne di conterie* (beadmaking canes) was translated by Merret as "rails for counting houses." This and similar errors were perpetuated in all subsequent editions based on Merret's translation, serving to delete any mention of glass beads from Neri's work (*see* discussions in Dillon 1907; Engle 2014; Francis 1988; Zecchin 1964) and leading many scholars to believe he had little to contribute to the topic (e.g., Turgeon 2001:66). Fortunately, Engle's recent three-volume translation of Neri has corrected these mistranslations and omissions (Neri 2003 [1612], 2004 [1612], 2007 [1612]).

3. These analyses were conducted at The Field Museum's Elemental Analysis Facility with the gracious assistance of Dr. Laure Dussubieux. These results will be presented in detail elsewhere.

4. Individuals no. 212 and 218 are another exception, but being a multiple burial with unclear temporal relationships, a mixed assemblage is not unexpected.

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