Attribution Enhanced: An SEM/EDS - LA-ICP-MS Investigation of a Suspected Mid-18th-Century Wistarburgh Glass Globular Bottle

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ABSTRACT

The Wistarburgh glassworks (Alloway, New Jersey, ca. 1739–1782) was the first commercially successful glass manufactory in what is now the United States. It mostly produced pane glass and bottles, the latter with a distinctive yellowish green color. Analyzed Wistarburgh bottle glass has calcium-rich compositions, but so too does some of the glass produced by other prominent 18th-century Germanic American glass manufacturers on the eastern seaboard. Distinguishing between the wares originating at these factories has traditionally relied on aesthetic criteria, but compositional data, particularly trace elements, provide an objective means of identifying them.

Compositional data for an intact, blown glass globular bottle suspected to have been made at Alloway are consistent with a Wistarburgh origin for this artifact. Of the 46 components determined, phosphorus (P), zirconium (Zr), niobium (Nb), thorium (Th), scandium (Sc), lead (Pb), silicon (Si), potassium (K), vanadium (V), and chromium (Cr) best distinguish the products of this glassworks from other prominent 18th-century Germanic American glass for which analytical data are available. Based on the depiction of similar bottles in contemporary paintings, this form appears to date to the early years of this glassworks, and may in part predate the more familiar chestnut bottles commonly attributed to Wistarburgh.

Introduction

Compositional data are increasingly being used to authenticate various artifacts made of various types of media, including porcelain, glass, alloys, and precious metals. Analytical data provide a degree of objectivity that supplements subjective assessments made using aesthetic criteria. This study presents major, minor, and high-precision trace element data for a blown glass globular bottle suspected to have originated at the Wistarburgh glassworks (ca. 1739–1782), one of three important Germanic American glassworks operating in the Mid-Atlantic states during the 18th century. The data are compared with an analytical database (Greenough and Owen 2016) for 42 samples of this glass. The results support a Wistarburgh origin for this artifact.

Sample Description

The analyzed sample is a yellow-olive green glass, thick-walled, globular (shaft and globe) bottle with an elongated, conical neck and a sheared lip (Figure 1). Although its color closely resembles bottle glass generally attributed to the Wistarburgh factory, and more specifically that of a seal bottle in the Corning Museum of Glass marked "RW" (evidently for Richard Wistar, a proprietor of the glassworks), its globular form is highly unusual. The "RW"-marked seal bottle notwithstanding, most Wistarburgh bottles have flattened sides; these are the so-called chestnut bottles.

The glass from which the globular bottle described here was made contains numerous tiny seed bubbles; larger air pockets are concentrated near its outward flaring top. The bottle has an unground pontil mark from which particles were removed for chemical analysis. Its base shows considerable wear, more so than the "RW" bottle. The bottle is 13.3 cm (5.2 in.) high, with a maximum diameter of 9.5 cm (3.7 in.). Similar bottles were produced by several 18th-century American glassworks (McKearin and Wilson 1976:plate 51, #6), but they are much less common than chestnut bottles. The sheared lip of the bottle differs from the rolled lip typical of chestnut bottles (and the "RW" bottle) produced at the Wistarburgh glassworks (Marschell 2007). However, its distinctive color closely resembles much of the bottle glass recovered from the Wistarburgh factory site, as well as intact bottles attributed to this glassworks. Furthermore, its glass contains "gall," a whitish yellow substance caused as particulate grains breaking away from glass furnace walls become embedded within molten glass. The presence of gall is diagnostic of some Wistarburgh glass artifacts, particularly around their pontil marks (Historical American Glass n.d.), but is not unique to the products of this factory.

Figure 1. Photographs of the analyzed globular bottle: (*left*) overview (note the sheared, rather than rolled, lip); (*right*) base showing pontil mark (note wear). The bottle is 13.3 cm (5.2 in.) tall. (Photo by Patrick Cruise, 2015.)

History

The three most famous American glassworks that operated on the eastern seaboard during the 18th century were founded by German immigrants: Caspar Wistar, John Frederick Amelung, and Heinrich Wilhelm Stiegel. The backgrounds of these individuals and the history of their enterprises have been described by numerous authors (Palmer 1976, 1989a, 1989b; Lanmon et al. 1991; Bigelow 2012), so are not repeated in detail here.

Wistar established the glassworks that bears his name, Wistarburgh, in Alloway, New Jersey, in 1739. This area has abundant silica sand (typically >98 wt.% SiO_2) essential for the production of good-quality glass, clay to make firing crucibles, and, at the time, abundant wood to fire glass furnaces and to make potash commonly used as a flux to help melt glass batches. Caspar's son Richard took over the glassworks when the elder Wistar died in 1752. He operated the factory for 29 years, whereupon his own son briefly operated the glassworks until its closure in 1782. The Wistarburgh glassworks produced flat glass, bottles, a variety of vessels, and, famously, globes for the electrostatic machines that Benjamin Franklin used for scientific research.

Heinrich Stiegel established a glassworks in Manheim, Pennsylvania, making flat glass and bottles. Convinced he could produce higher quality glass, he replaced the Manheim factory in 1770, making the furnaces himself. He succeeded in making exemplary glass vessels, but none are signed. Stiegel's advertisements, however, show that his factory produced tableware, some of which was cut and engraved. This colorless glass has potash-lead compositions, which contrast with the green soda-lime glass used to make flat glass and bottles. The factory ceased operation in 1774.

In 1784 John Frederick Amelung established the community of New Bremen, Maryland, and a glassworks near Frederick, Maryland. The New Bremen factory acquired equipment from a nearby glassworks founded by former employees of Stiegel's Manheim enterprise. The New Bremen glassworks produced flat glass, green glass bottles, and, eventually, colorless glass to make tableware, some of which was elaborately engraved. This factory survived until ca. 1795, whereupon the glassworkers migrated to Baltimore, Philadelphia, and elsewhere.

Analytical Methods

The major and minor element composition of the bottle was determined using an LEO 1450VP scanning electron microscope (SEM) operated with a beam current of 20 kV, and equipped with an Oxford Instrument INCA X-max 80 mm^2 silicon drift EDS detector.

Trace elements were determined by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS). This method uses a laser beam focused on the surface of the medium being analyzed to generate minute particles that are then transported to the excitation source of the ICP-MS for digestion and ionization. The excited ions are then fed through the mass spectrometer for analysis. Detection limits are very low, and analytical data produced by this method are highly precise. The method is also minimally destructive: analysis can be undertaken on a tiny (<1 mm) flake of sample. In the case of blown glass, this is best removed from near the pontil mark. The ablation procedure leaves a tunnel a few tens of microns wide through the irradiated sample.

The equipment employed for the present investigation uses a Resonetics RESOlution M-50 193 nm ArF excimer laser ablation system coupled to a Thermo X Series II quadrupole mass spectrometer. Ten analyses consisting of 30 seconds of washout/background followed by 30 seconds of ablation were made with a spot size of 36 µm, laser repetition rate of 6 Hz, and a fluence of 6 J/cm^2. Data were processed with Iolite software (Paton et al. 2011) using NIST 612 glass as an external reference and the silicon concentrations determined by EDS as an internal reference.

Results

The globular bottle consists of potassium-bearing, calcium-rich ($CaO/(CaO+Na_2O+K_2O) = 0.81$) soda-lime glass (Tables 1, 2). It contains 65% SiO_2, 2.4% Al_2O_3, and mod-

Table 1. Composition of the Globular Glass Bottle, Compounds.

Symbol	Name	wt%
SiO_2	silicon dioxide	65.11
TiO_2	titanium dioxide	0.27
Al_2O_3	aluminum oxide	2.36
FeO_t	iron oxide (total Fe as FeO)	0.89
MnO	manganese oxide	0.63
MgO	magnesium oxide	1.67
CaO	calcium oxide	22.74
Na_2O	sodium oxide	2.91
K_2O	potassium oxide	2.3
P_2O_5	phosphorus pentoxide	0.78
SO_3	Sulfur trioxide	0.05
	Total	99.71

Table 2. Composition of the Globular Glass Bottle, Elements.

Symbol	Name	ppm
Rb	rubidium	22.14
Cs	cesium	0.25
Sr	strontium	957.40
B	boron	123.62
Ba	barium	2295.40
La	lanthanum	84.00
Ce	cerium	51.60
Pr	praseodymium	12.80
Nd	neodymium	47.40
Sm	samarium	7.70
Eu	europium	1.50
Gd	gadolinium	6.80
Tb	terbium	0.90
Dy	dysprosium	5.30
Ho	holmium	1.00
Er	erbium	2.70
Tm	thulium	0.30
Yb	ytterbium	2.10
Lu	lutetium	0.30
Y	yttrium	35.23
Zr	zirconium	182.62
Nb	niobium	5.09
Ta	tantalum	0.36
Th	thorium	3.02
U	uranium	1.10
Sc	scandium	5.77
V	vanadium	17.79
Cr	chromium	17.23
Co	cobalt	4.60
Ni	nickel	25.47
Cu	copper	20.07
Pb	lead (*plumbum*)	855.50
As	arsenic	23.30
Sn	tin (*stannum*)	19.90

erate amounts of femic components (0.9% FeO_t, 0.6% MnO, 1.7% MgO). In terms of its lime, soda, and potash contents, it plots on the low-sodium, high-calcium edge of the common field of analyzed soda-lime glass from the Wistarburgh, Amelung, and Stiegel factories (Figure 2). The bottle is enriched in phosphate (0.8% P_2O_5), a signature component in some Wistarburgh soda-lime glass recovered from the factory site (Owen 2004), and it plots in the Wistarburgh field on P_2O_5 vs. CaO and P_2O_5 vs. SiO_2 diagrams (Figure 3B, C).

In terms of trace elements, the bottle has an anomalously high barium content (2,295 ppm Ba) given its modest concentrations of potash (2.3% K_2O) and rubidium (22 ppm Rb), suggesting the inclusion in the glass batch of a source of this component unrelated to alkali feldspar. The glass is enriched in the rare earth elements (REEs) and yttrium (Y), a proxy for the REEs.

Origin of the Globular Bottle

Compared with several other historical (19th-century) glassworks from New Jersey and Connecticut, soda-lime bottle glass produced by the Wistarburgh factory has high $CaO/(CaO+Na_2O+K_2O)$ ratios (>0.7) and is enriched in various trace elements, notably the REEs, Y, and barium (Owen 2004). However, analysis of glass recovered from the sites of the two other prominent 18th-century Germanic American glassworks—the Stiegel and

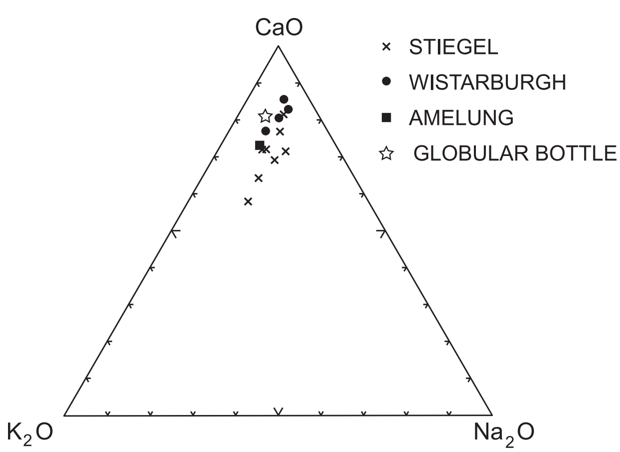


Figure 2. $CaO-K_2O-Na_2O$ plot (wt.% data) comparing the composition of the globular bottle with soda-lime glass sherds from the Wistarburgh, Stiegel, and Amelung factories. (Graphic by Randolph Corney, 2015.)

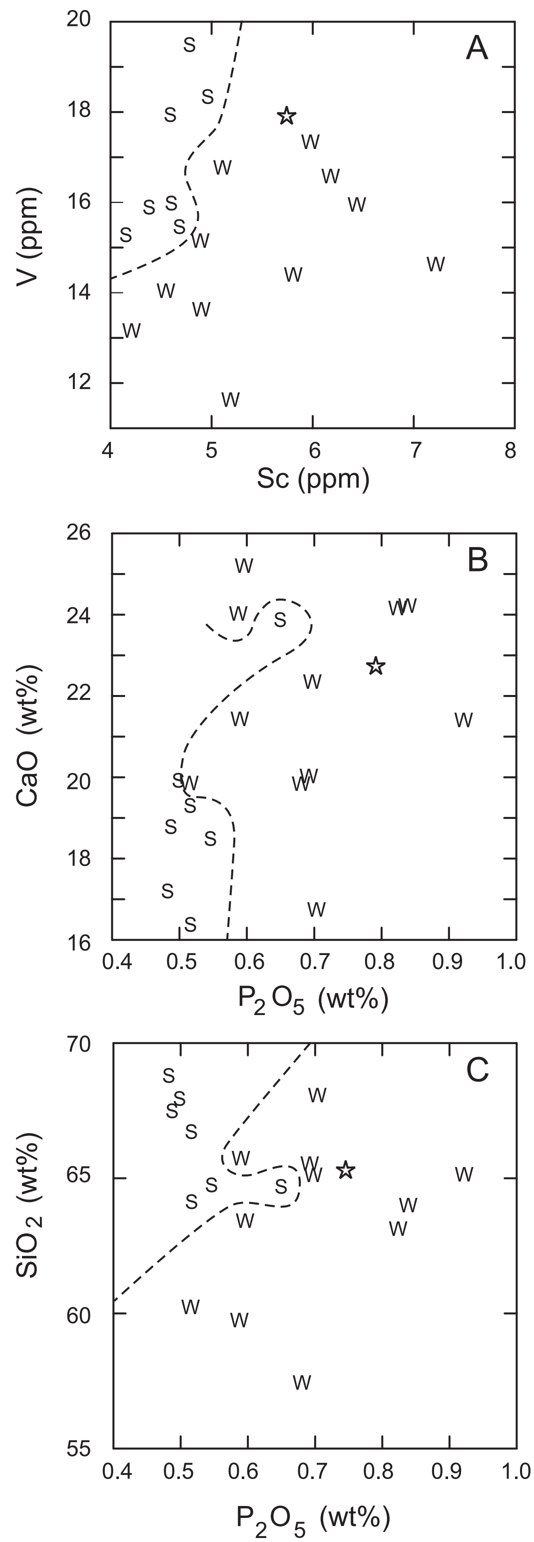


Figure 3. Binary discrimination diagrams used to distinguish between Wistarburgh ("W") and Stiegel ("S") glass: (*A*) V vs. Sc; (*B*) CaO vs. P_2O_5; (*C*) SiO_2 vs. P_2O_5. Star - analyzed glass globular bottle. (Modified from Greenough and Owen 2016.)

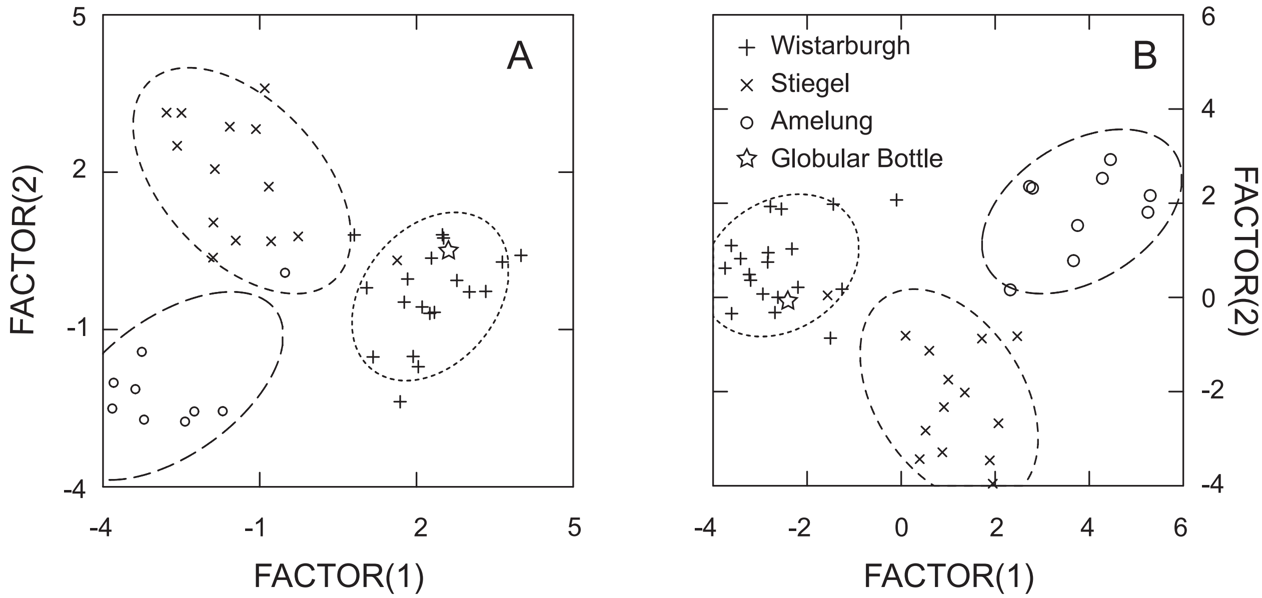
Amelung factories—shows that their soda-lime glass can not only be lime-rich, but is also enriched in these trace elements. Indeed, these compositional commonalities point to (1) linkages (possibly migrant workers) between these three glassworks and/or (2) the inclusion of similar raw materials in glass batches prepared at each glassworks. Some components are associated with heavy minerals present as impurities in silica sand. These include phosphorus (P) in phosphate minerals (e.g., apatite, monazite), selected rare earths (lanthanum [La], cerium [Ce], neodymium [Nd]), and thorium (Th) in monazite, niobium (Nb) and tantalum (Ta) in titanium (Ti) oxides (e.g., rutile), vanadium (V) in oxide minerals such as ilmenite and magnetite, and zirconium (Zr) in zircon. Other components can be associated both with heavy minerals and with stabilizers or fluxes used in glass batches. Strontium is an example. This trace element is geochemically linked with calcium, a major constituent of apatite and calcite (either in the form of limestone or shells), used to stabilize glass. Alkalis are another example. Sodium and potassium are associated with alkali feldspar, which can be preserved in small amounts in silica sand, but alkali carbonate minerals, traditionally derived from plant ash, are the major source of these components in some types of historical glass. Lead was added in the form of lead oxide (e.g., read lead or litharge) as a flux and/or to add brilliance to finished glass objects with alkali-lead compositions.

Unless heavy minerals are removed from silica sand, their geochemical signatures can be preserved in glass, and this characteristic can be exploited to identify the sources of sand used by historical glassmakers (Owen et al. 2005; Owen and Greenough 2008). It also forms the basis for establishing glass-discrimination diagrams on which the compositions of glass from different factories cluster in different (and sometimes overlapping) fields. In short, glass compositions vary with the composition and proportion of the ingredients used in the batches from which they are made. The only other significant variable is the loss of some volatile components (notably lead and possibly alkalis) during firing (melting; Hallse and Cook 2006).

Notwithstanding the calcic composition of soda-lime glass made by Amelung, Steigel, and Wistarburgh, there are some components that serve to distinguish the products of these factories, and a Wistarburgh origin for the globular bottle described here is suggested not only by its P_2O_5, CaO, and SiO_2 contents, but by its concentration of V and scandium (Sc) (Figure 3A). However, it has a lower strontium/nickel (Sr/Ni) ratio (= 38) than previously analyzed Wistarburgh soda-lime glass (Sr/Ni = 70–120); in terms of these two components, it more closely resembles Stiegel soda-lime glass (Sr/Ni = 29–44). Clearly, simple binary plots are not ideally suited for distinguishing between these types of glass. Consequently, the use of exploratory statistical methods is required to tease out systematic compositional differences between 18th-century Germanic American soda-lime glass.

Greenough and Owen (2016) showed that various minor and trace elements, including P, Zr, Nb, Th, Sc, and lead (Pb), best serve to distinguish Wistarburgh, Amelung, and Stiegel glass irrespective of glass type. The composition of the globular bottle plots in the Wistarburgh field on the discriminant analysis plot based on these components (Figure 4A). It also lies in the Wistarburgh field on a second such plot that incorporates these six components as well as silicon (Si), potassium (K), V, chromium (Cr), and tin (Sn) (Figure 4B). These plots not only provide compelling evidence that the globular glass bottle was produced at the Wistarburgh glassworks, but the good field separation on them testifies to the lack of extraneous material (e.g., imported glass) included in the analytical database on which these diagrams are based (Greenough and Owen 2016). The components on which these diagrams are based include those affiliated with silica sand (silica itself and heavy mineral impurities) and fluxes, the compositions and proportions of which therefore appear to be diagnostic of each of the three types of 18th-century Germanic American glass described here.

Given that Caspar Wistar not only hailed from Germany, but also hired German glassblowers, it stands to reason that northern European styles of glass were produced at the newly opened Wistarburgh glassworks. Bottles of similar form are depicted on northern European paintings dating to the 1730s–1750s (e.g., William Hogarth's "A Midnight Modern Conversation," ca. 1732). Furthermore, based on depictions of the evolution of wine bottle shapes from 1700 to 1800, the shape of this bottle indicates a manufacturing date corresponding to the early years of Wistarburgh (Hancock 2009:368). It possibly predates the more familiar and common chestnut bottles attributed to this glassworks.



F1 = -4.849917 + (6.290871*P_2O_5) + (-0.019889*Zr) + (2.283043*Nb) + (-2.607949*Th) + (0.412847*Sc) + (0.000008*Pb)

F2 = -0.537822 + (0.518716*P_2O5) + (0.002249*Zr) + (0.476003*Nb) + (0.752341*Th) + (-0.784419*Sc) + (0.000017*Pb)

F1 = -5.309887 + (0.190298*SiO_2) + (-0.138901*K_2O) + (-7.557678*P_2O_5) + (0.021435*Zr) + (-2.718733*Nb) + (2.058282*Th) + (-0.267955*Sc) + (-0.017973*V) + (0.136342*Cr) + (-0.00001*Pb) + (0.142826*Sn)

F2 = -1.703146 + (0.018674*SiO_2) + (0.012772*K_2O) + (0.596644*P_2O_5) + (-0.000566*Zr) + (-0.391671*Nb) + (-1.397033*Th) + (0.896917*Sc) + (-0.009879*V) + (0.072964*Cr) + (-0.000017*Pb) + (-0.163765*Sn)

Figure 4. Discrimination diagrams useful in distinguishing between Wistarburgh, Stiegel, and Amelung glass, based on (*A*) including P, Zr, Nb, Th, Sc, and Pb and (*B*) including P, Zr, Nb, Th, Sc, Pb, Si, K, V, and Cr. Note that the composition of the globular bottle plots in the Wistarburgh field on both diagrams. Analyses of other glass objects can be plotted by calculating values for F1 and F2 using the equations provided. Star - analyzed glass globular bottle. (Graphic by Randolph Corney, 2015.)

During the first half of the 18th century, the globular shape was the primary style of bottle used for table decanters or serving bottles. This accounts for the lack of a string rim or rolled lip on the bottle described here as a cork would have been used to close it, but without the need to have the cork secured, as was possible with string-rim or rolled-lip bottles. Wear on the base of the bottle shows that it was repeatedly and heavily used, further pointing to its use as a decanter.

Aesthetic criteria such as form and shape alone are insufficient for rendering sound judgment on the origins of suspected early Germanic American glass, partly because of possible pressure placed on German glassblowers to imitate English imports (Palmer 1989b). Consequently, compositional data provide an objective basis for evaluating attributions based solely on subjective criteria. Regrettably, the analytical database for early American glass is minute compared with ancient glass. For example, less than 10% of the approximately 2,000 analyses of glass and related materials reported in Brill (1999) are for American artifacts. Although sufficient analytical data are available to support a Wistarburgh origin for the globular bottle described here, many more analyses not just of Germanic American glass, but of the products of the hundreds of glassworks that operated along the eastern seaboard and elsewhere in America during the 18th and 19th centuries are required before the use of compositional data will become routine in provenance studies of domestically produced American glass. Fortunately, only tiny (tens of microns diameter) fragments of glass are required to allow the determination of high-precision analyses of major, minor, and trace elements by microbeam techniques.

Conclusions

This study compares the major, minor, and trace element composition of a globular bottle to an analytical database for 18th-century Germanic American glass (Amelung, Stiegel, and Wistarburgh). Of the 46 components determined, P, Zr, Nb, Th, Sc, Pb, Si, K, V, and Cr best distinguish the products of these glassworks. The composition of the globular bottle is consistent with a Wistarburgh origin. Based on the depiction of bottles of similar form on contemporary northern European paintings and progressive changes in wine bottle shapes during the 18th century, it is interpreted as being an early product of this glassworks.

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