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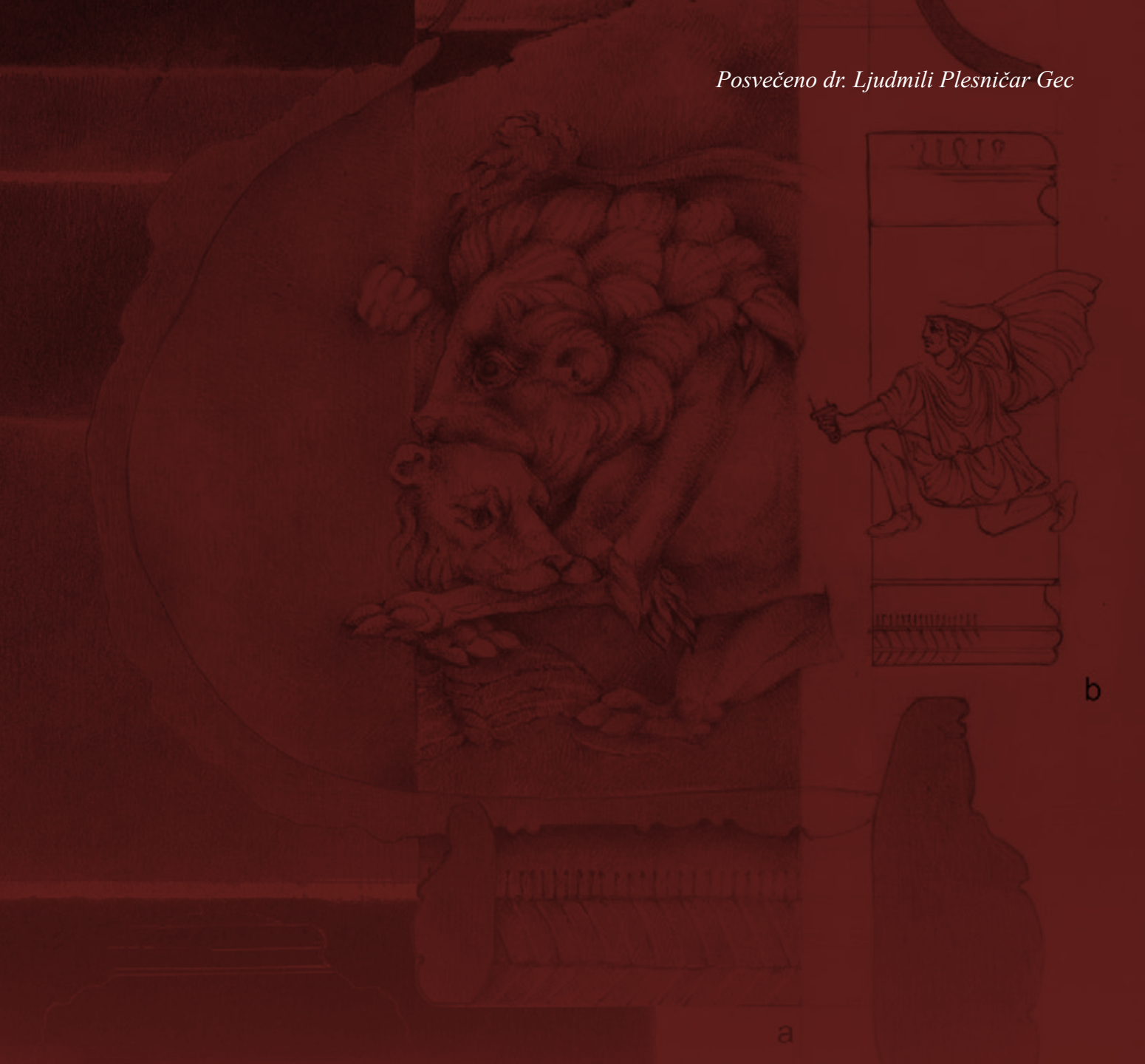

EMONA

med Akvilejo in Panonijo
between Aquileia and Pannonia

Zbrali in uredili / *Edited by*
Irena Lazar & Bernarda Županek

KOPER 2012

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Janka ISTENIČ, Žiga ŠMIT

A RAW GLASS CHUNK FROM THE VICINITY OF NAUPORTUS (VRHNIKA)

ABSTRACT

The paper reviews archaeometric research of Roman glass, gives a description of the analytical procedure carried out by the techniques of proton induced X-ray and gamma-ray spectrometry (PIXE, PIGE), and presents analytical results for an undated chunk of raw glass from the vicinity of Vrhnika as well as their comparative evaluation and interpretation. The results indicate that the piece of raw glass is Roman.

Keywords: *raw glass, glass, Roman period, PIXE, archaeometry*

Ključne besede: *surovo steklo, steklo, rimska doba, PIXE, arheometrija*

Introduction

In its Roman collection the Archaeological Department of the National Museum of Slovenia keeps a relatively large chunk of raw glass (Inv. No. R 1886; fig. 1). It was found in 1886 near Vrhnika (Roman *Nauportus*) and came directly to the museum, where the curator Alfons Müllner inventoried it. He put down in the inventory only very scanty information on where it was found: »Grüner Glasfluss, Oberlaibach«. More information can be found in his short paper on the glass chunk (Müllner, 1893, 171–172): it was found in 1886 to the east of Vrhnika, three metres deep in clay, in the area delimited by the River Ljubljanica in the south, the Vrhnika-Ljubljana road in the (north)west and Sinja gorica in the (north)east, where the clay for brickworks was dug. It was a stray find.

Jana Horvat briefly discussed the location of the find-spot of this glass chunk. In the area described by Müllner, i.e. the triangle between the Ljubljanica, the Vrhnika-Ljubljana road and Sinja gorica, she noticed depressions caused by clay extraction, which support Müllner's report on where the glass chunk was found (Horvat, 1990, 181, map 1: 14).

Our main interest lay in the question of whether the glass chunk is Roman or not. We had to proceed from the object itself, because the chunk was not found in a context that would allow its dating.

Description of the raw glass chunk (fig. 1)

The glass chunk is of irregular shape, green in colour and transparent, weighs 3.14 kg and measures 21 x 17.8 x 10.5 cm. Concentric lines are visible at the surface of several sides of the glass chunk; they indicate that it was broken from a large glass slab.

A slightly curved and about 14 cm long line of air bubbles (diam. *c*. 3 mm) is visible in the glass.

Research issues

From comparison of the object in question with published Roman raw glass chunks (Foy, Nenna, 2001, figs. 6, 12, 15, 18) it was clear that it could be Roman. Therefore we decided to approach the question of its approximate date from its elemental composition.

Roman and post-Roman glasses form a group of relatively homogeneous soda-lime-silica glasses, with little variation in major element composition. The source of sodium in Roman and post-Roman glasses was *natron*, i.e. a mixture of relatively pure sodium salts, therefore they are low in potash and magnesia (less than 1–1.5 %[1] of each oxide) and contain nearly no phosphorus. The preferred source of *natron* was Egypt (Freestone, 2005; Picon, Vichy, 2003; Shortland et al., 2006).

Glasses made from *natron* were the dominant type of glass in the Mediterranean and surrounding regions from the mid-first millennium BC to around 850 AD. They were produced on a very large scale, probably in a small number of primary workshops, which appear to have been located near the sources of raw materials in Egypt and Palestine. The slabs of raw glass were broken up into chunks of glass that were traded across the Mediterranean and further to secondary workshops, where they were remelted to make glass vessels (Freestone, 2005; Shortland et al., 2006).

Twelve different compositional groups of Roman and post-Roman *natron* glasses have been established. Only four of them (Groups 1–4) have so far been identified in the West. Groups 1 and 2 seem to be relatively late, dating to the 5th and from the 6th to the beginning of the 8th century, respectively (Picon, Vichy, 2003, 18–22; Foy et al., 2003). Glasses of Group 3, most probably produced in the region of Syria and Palestine, predominate in the West, in the Mediterranean as well as in the Near East. They are dated at least from the 3rd century BC until the 9th century AD (Picon, Vichy, 2003, 18–26; Foy et al., 2003, 61–62, 80).

The core of Group 4 are glass vessels and raw glass chunks from the cargo of the late 2nd/beginning of the 3rd century Ouest-Embiez I shipwreck (southern France, Dept. Var; Thirion-Merle, Vichy, 2007; Fontaine, Foy, 2007).

1 Note that all percentages in the paper refer to wt %.

Figure 1: Raw chunk glass from the vicinity of Vrhnika. Not to scale. Photo: T. Lauko, Narodni muzej Slovenije.

There is another category of soda-lime-silica glass that used sodium-rich plant ash from salt resistant, halophytic plants growing in coastal, salt marsh and desert regions, or seaweeds as a source of sodium. In contrast to *natron* glasses, the glasses made with sodium from plant ash have relatively high magnesia and potash (typically more than 1.5–2 % of each oxide); they also contain a low percentage of phosphorus, which is practically absent in *natron* glasses. In the Near East this kind of glass presumably coexisted with the production of *natron* glass. It seems that during the 9th century AD glass made of sodium-rich plant ash replaced *natron* glasses in south-western and Mediterranean Europe as well as in Egypt (Foy, Nenna, 2001, 26; Shortland et al., 2006; Freestone, Gorin-Rosen, Hughes, 2000, 69–70).

A new procedure for glass production and its organisation are characteristic of the Middle Ages in Europe: glasses were produced locally from river sand and potash-rich plant ash (i.e. forest plant ash, mostly ash of beech or ferns), which provided potassium (potash) to act as a flux. The products of individual glass workshops have different elemental composition. They can be differentiated from *natron* glasses, as well as from soda glasses made with soda from plant ash, by their high potassium and by relatively high phosphorus contents. It seems that in central and northern parts of western Europe potassium glasses gradually replaced *natron* glasses around the 9th century AD (Foy, Nenna, 2001, 25–26; Freestone, 2005; Shortland et al., 2006).

Experimental

Analyses were made by proton induced X-rays and gamma rays (PIXE-PIGE), using a proton beam in air. The advantages of both techniques are: i) no sample has to be taken, ii) very small radiation damage is caused, and iii) they yield good sensitivity for a large number

of elements. Both methods have been used for the analysis of glass from different periods, e.g. Greek (Sokaras et al., 2009), Roman and early medieval (Zucchiatti et al., 2007; Šmit et al., 2009a) and Venetian-type glass (Kos, Šmit, 2000; Šmit, 2007; Šmit et al., 2009b).

The measurements were made at the Tandetron accelerator of the Jožef Stefan Institute in Ljubljana. The proton impact energy at the target was 2.7 MeV. Two X-ray spectra were measured, one with a 5.7 cm air gap as the only absorber, and one with an additional absorber of 0.1 mm aluminium foil, which increased the sensitivity for mid-Z elements around strontium and antimony to about 10 μg/g.

The lightest elements, Na, Mg and Al, were determined according to the yields of the respective gamma rays induced by the reactions of inelastic proton scattering. The concentrations of these three elements were determined using the standard glass NIST 620 as reference. The measurements were made in three selected spots. The proton current was measured by a thin wire mesh intersecting the beam in vacuum; the transmission of the mesh was 58 %. The integrated dose was 10 μC for measurements on the glass chunk, and 8 μC for measurement on NIST 620 glass, which contains a larger (3.69 %) amount of MgO.

The measurements did not provide values for phosphorus. In X-ray spectra, the phosphorus K lines are overlapped by the escape peak of calcium. Phosphorus also emits a gamma line at 1266 keV, though the strong Compton background of sodium limits the sensitivity for phosphorus to a few percent.

The measurements provided results in which the accuracy for major elements lay within ±5 %; the accuracy for minor elements (concentration below 1 %) decrease to 10–20 % and even less in the vicinity of the detection limits. For magnesium, which was determined according to its gamma line at 585 keV, the detection limit was about 0.2 %. This line overlaps with a gamma line of 583 keV from the natural background; its contribution was estimated from the measured count rate and measuring time and was subtracted from the total yield.

Analytical results and discussion

Analyses were made at three spots, in order to get information regarding the homogeneity of the material. The results are given in Table 1.

For several minor elements, notably lead, the differences in concentrations exceed the uncertainty levels, which reflects the inhomogeneity of the sample.

The low potassium and magnesium contents of the raw glass chunk found near Vrhnika indicate the use of *natron* as a flux, suggesting a typical Roman and post-Roman glass production (cf. paragraph Research issues above). Therefore we compared its elemental composition to Groups 1–4 of *natron* glasses, i.e. the groups which occur in the West. We used data from Foy et al., 2003 (79–85, Appendices 1–3) for comparison of Groups 1–3 and from Thirion-Merle, Vichy, 2007 (267, table 1) for group 4. They give mean values and standard deviations of the elemental composition for each group, and in the case of late (5th to 7th century) subgroups 3.1, 3.2 and 3.3, also the elemental composition of individual samples.

The low contents of iron, manganese and titanium oxides clearly differentiate the raw glass chunk under discussion from the late Roman (5th century) and post-Roman (middle of the 6th to beginning of the 8th century groups I and II (Foy et al., 2003, 45–48, Appendices 1, 2). It can also be distinguished from Group 4, which is characterised by relatively low values for the oxides of calcium, aluminium and iron (mean values for 12 raw glass chunks: 5.26 % CaO, 1.80 % Al_2O_3, 0.28 % Fe_2O_3), as well as the relatively high antimony content (Thirion-Merle, Vichy, 2007, 267, Table 1) which served as a decolourant in this group of glass.

The elemental composition measured in the raw glass under discussion is most similar to Group 3. Values for this group derive from analyses of 123 samples (Foy et al., 2003, 61–62, Table 12; Thirion-Merle, Vichy, 2006, 268, Table 2), which comprise at least 16 raw glass chunks (Foy et al., 2000, 53, Fig. 1). They match relatively well the raw glass chunk from Pompei (De Francesco et al., 2010, 14, Table 2: RG), which also seems to correspond to Gro-

	spot 1	spot 2	spot 3
Na_2O	14.1 ± 0.5	14.1 ± 0.5	15.0 ± 0.5
MgO	0.60 ± 0.08	0.63 ± 0.08	0.70 ± 0.08
Al_2O_3	2.68 ± 0.10	2.77 ± 0.10	2.76 ± 0.10
SiO_2	70.3 ± 1.5	71.2 ± 1.0	69.7 ± 1.0
SO_3	0.52 ± 0.10	0.14 ± 0.10	0.53 ± 0.10
Cl	1.11 ± 0.05	1.10 ± 0.05	1.15 ± 0.05
K_2O	0.56 ± 0.03	0.42 ± 0.03	0.47 ± 0.03
CaO	8.51 ± 0.40	8.15 ± 0.40	8.01 ± 0.40
TiO_2	0.067 + 0.010	0.051 ± 0.010	0.071 ± 0.010
MnO	1.05 ± 0.05	1.01 ± 0.05	1.05 ± 0.05
Fe_2O_3	0.44 ± 0.03	0.36 ± 0.03	0.40 ± 0.03
CuO	0.006 ± 0.002	0.003 ± 0.002	0.003 ± 0.002
ZnO	0.003 ± 0.001	0.001 ± 0.001	0.001 ± 0.001
Br	0.0008 ± 0.0002	0.0004 ± 0.0002	0.0006 ± 0.0002
Rb_2O_3	0.0008 ± 0.0002	0.0004± 0.0003	0.0006 ± 0.0002
SrO	0.056 ± 0.003	0.053 ± 0.003	0.058 ± 0.003
ZrO_2	0.0049 ± 0.0010	0.0024 ± 0.0010	0.0045 ± 0.0005
PbO	0.018 ± 0.0005	0.0011 ± 0.0005	0.0010+ ± 0.0005

Table 1: Composition of the chunk of raw glass from the vicinity of Vrhnika. Values refer to wt. %.

up 3. Of the major elements, it contains similar percentages of silica (66.3 % SiO_2) and CaO (7.7 %) as the raw glass found near Vrhnika, but significantly higher Na_20 (21.9 %). Values of most of the minor elements, such as MgO (0.58 %), K_2O (0.55 %), TiO_2 (0.05 %), MnO (0.49 %), Fe_2O_3 (0.33 %) and Al_2O_3 (1.97 %), also match relatively well the item from the vicinity of Vrhnika. The difference in the Na_2O values can be explained by variation in Na_2O % within glasses of Group 3. Na_2O concentration around and above 20 % are not beyond the limits of the group. Indeed, they also occur in raw glass from the group (cf. Foy et al., 2003, 70, Table 14, 85, Appendix 3; raw glass: samples VRR 48, 166). Analyses of raw glasses from seventeen late Byzantine period (6th to 7th century) glass furnaces from Bet Eli'ezer (Israel) provided evidence that the furnaces were charged in a number of batches throughout the firing process, and that the upper ones contain more soda. Presumably the glass makers added a batch with excess *natron* towards the end of a firing, to maintain the fluidity of the glass as the temperature of the furnace began to fall (Freestone, Gorin-Rosen, Hughes, 2000, 67, 71, fig. 5).

Group 3 of *natron* glasses comprises more than nine tenths (9/10) of Roman and Early Mediaeval glasses in the West; it is also the best represented group in the Mediterranean as well as in the Near East. In the West and the East it is present at least from the 3rd century BC until the 9th century AD; it seems that in the West its presence before 1st century AD was only sporadic. The glasses of this group were most probably produced in the region of Syria and Palestine (Picon, Vichy, 2003, 18–26; Foy et al., 2003, 61–62, 80).

In comparison to Egypto-Palestinian glass as represented in the scheme by Freestone, Gorin-Rosen, Hughes (2000, 72–73), the chunk from the vicinity of Vrhnika matches well the Levantine I Group (in the scheme of the French authors, this group is comprised in Group 3 –cf. Picon, Vichy 2003 and Foy et al., 2003), which typically contains lime (CaO) at around 8–9% and alumina (Al_2O_3) at 2.5–3 %. The group comprises 4th–7th century glass produced in the Near East from River Belus sand (Gorin-Rosen, Hughes 2000, 72–74, 82, fig. 8). However, this agreement does not exclude an earlier dating of the Vrhnika chunk.

Conclusions

The raw glass chunk from Vrhnika is a soda-lime-silica glass made with *natron*. This provides a clue for its dating from the 3rd century BC/1st century AD to the 8th/9th century AD. It seems probable that it is Roman.

The information that the raw glass chunk was found in a considerable depth of clay suggests that it was deposited at a place where, afterwards, probably in the Roman period, a thick alluvial clay layer was formed. Such a place might have been a stream channel or a depression (e.g. from clay extraction) in terrain (information kindly provided by Tomaž Verbič, Arhej d.o.o., Sevnica).

Deposition in a stream channel, connected to the River Ljubljanica, which was an important transport route in the Roman period (Istenič, 2009a; Istenič, 2009b), might suggest that the raw glass chunk was lost during transport by water.

POVZETEK

Kos surovega stekla, najden konec 19. stoletja pri Vrhniki v slabo poznanih okoliščinah, ki ne omogočajo datacije iz najdiščnega konteksta, je bil avtorjema povod za datiranje na podlagi kemijske sestave stekla. Rezultati so pokazali, da gre za natronovo steklo, značilno za rimsko in poznoantično obdobje. Članek vključuje tudi kratek pregled stanja raziskav na področju arheometričnih analiz stekla.

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