An Early Byzantine glass workshop at Argyroupoli, Crete: Insights into complex glass supply networks

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Abstract

Archaeometric studies on early Byzantine glass excavated in Greece are extremely scarce in the literature and almost exclusively related to small groups of samples, mainly glass tesserae. The aim of this study is to present archaeometric data of a large assemblage of early Byzantine glass excavated in ancient Lappa, modern town of Argyroupolis, SW of Rethymno in Crete.

A series of salvage excavations unearthed a complex of 5 rooms, identified as a secondary glass workshop, yielding more than 1500 glass fragments of objects (mainly rims and stems of glass goblets) and glass working debris (mainly test drops, chunks etc.). The glass and the architectural remains date to the 4th to 7th c. AD.

The glass is a typical soda lime silica glass, with close similarities between the chemical composition of the glass working debris and the objects found in the complex. The glass working debris can be divided in three main compositional groups, including the two well-known mineral-natron based groups Levantine I and Foy Série 2.1. The third compositional group of samples identified in the assemblage has a strong plant ash signature. This group, similar to one previously identified in Egypt, has been noticed here for the first time outside Egypt. There are only a few examples of Foy Série 3.2, a composition that circulated widely in the Mediterranean during the early Byzantine period. This differentiation into four compositional groups can be also broadly linked to object types, while the glass working debris covers all compositional groups.

1. Introduction - Archaeological background

Rescue excavations by the Greek Archaeological Service in 2003 to 2007 in the outskirts of the modern village of Argyroupolis yielded an early Byzantine building complex with workshop and habitation areas (Figure 1). Argyroupolis is situated west of Rethymnon in the area of ancient Lappa, one of the significant cities of Crete during Hellenistic and Roman times. Since the beginning of the 20th century, rescue excavations in the area of the ancient city have revealed habitation areas, workshops, bath houses and parts of the extended cemetery of the city (Gavrilaki, 2004). Some fine examples of sculpture as well as mosaic pavements testify the prosperity of the city. During the early Byzantine period Lappa was the see of a bishop and at least three basilicas are known from that period (Sanders, 1988: 120, 163).

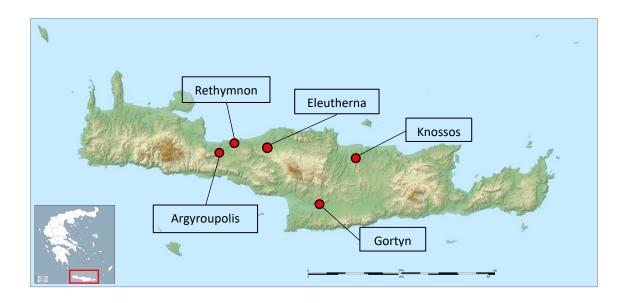


Figure 1. Map of Crete indicating major sites during the early Byzantine period.

The building complex under discussion was constructed in front of a tall retaining wall (Figure 2). The wall was built with roughly dressed stones, mortar and architectural members reused from earlier structures; on its base it had two semi-circular projections in the form of buttresses. The building consisted of five rooms arranged around an open space. The rooms were made of stones and their roofs were covered with tiles; in some cases, the bedrock had been cut to accommodate the walls. There are indications, such as the trace of a staircase on the retaining wall, that some of the rooms had a second floor. The building complex was built on a backfill of the 6th – early 7th century, which had been levelled to form the floors. Traces of an earlier building phase, possibly connected with the backfill, can be seen in the interior of some of the rooms. The building was destroyed sometime during the 7th century as indicated by the pottery of the destruction layer.

The excavation yielded more than 1500 fragments of glass vessels, glass tesserae and a great number of glass refuse, which led to the suggestion that part of the building housed a workshop for manufacturing glass objects. The study of the glass fragments, until now, has shown that one of the main products of the workshop were lamps. For the typology of the vessels only preliminary notes can be made since the glass findings are not published yet. At least three different types can be discerned; the majority were stemmed cups with a discoid base ('goblets'), while only a few fragments could be identified as lamps with a conical body and a pointed base, and lamps with a tall cylindrical stem for use in polycandela. All three types appear in the fifth century and spread during the next two. Especially the first type, that of the stemmed cup, was a very common form during the 6th and the 7th centuries. Because of its shape it could be used either as a drinking vessel or as a lamp (Antonaras, 2008, 26,28). Among the glass findings are also fragments of jugs and unguentaria. The vessels were made mostly of green, light blue or white transparent glass and only a few of them in dark blue glass. Furthermore, a few fragments of window panes in green or light blue glass were also found; it is not certain whether they were products of the workshop or they were used for recycling.

The significant number of tesserae found during the excavation, more than 80 pieces have been identified so far, may also possibly linked to the workshop, either as raw material for recycling, or as a product. The tesserae are of various colours, red, blue, green or yellow, the most outstanding among them, though, are the two examples with leaves of gold.

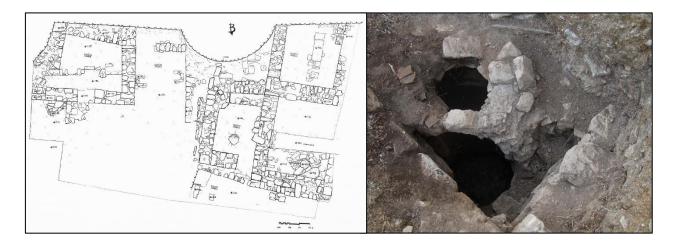


Figure 2. Top view of the complex with the five rooms and the small circular construction.

Glass manufacturing can also be connected with earlier habitation in the area since under the floor of one of the rooms a small circular construction was discovered, which belongs to an earlier building phase (Figure 2 right). The construction has been tentatively interpreted by the excavator as a glass furnace. Its size and formation resembles similar constructions of Imperial and Byzantine periods in Greece (for indicative examples cf. Antonaras 2009; Raptis 2010; Vasilakis 2011; Antonaras 2014; Gounaris 2004; Antonaras and Chrysostomou, 2015). The diameter of the furnace is c. 1.20 m, its lower part, probably the combustion chamber, was dug into the soft bedrock and its upper part, in the shape of a dome and a height of c. 1.10 m, was made of tiles and stones. On the base of the dome a ring was formed on the bedrock and a narrow, arched opening on the periphery of the dome led to its interior. Pottery forms of the 4th and 5th centuries, that could be related to the construction of the furnace, point to the glass activity in the area already from that period (Fiolitaki, 2007, 1312-1314, Fiolitaki, forthcoming).

2. Materials and methods

Seventy-six samples from the Argyroupolis assemblage were selected for analysis, divided in two major categories (Table 1): fragments of glass objects, and glassworking debris. The selection of objects (55 fragments in total) focussed on stems of goblets, the main vessel type of early Byzantine contexts, and on rims of unspecified glass vessels. The glass working debris consists of deformed glass masses, chunks and test drops (21 fragments in total). All fragments are transparent with characteristic natural hues such as aqua blue, greenish and olive green, while there are only few fragments of transparent cobalt blue glass (Table II, supplementary material). Interestingly, there is only one characteristic fragment of a foot-base of a glass beaker, which otherwise is very common in early Byzantine contexts.

Table 1. Assemblage division in four major glass categories according to their chemical composition.

Rethymno glass categories	Objects	Glassworking debris	Total
Levantine	N=26 Stemmed goblets n=9 Vessel rims n=17	N=5 Glass chunks n=1 Test drops n=1 Deformed glass n=2 Furnace wall n=1	N=31
Série 2.1	N=19 Stemmed goblets n=5 Vessel rims n=13 Vessel body n=1	N=6 Glass chunks n=2 Test drops n=2 Deformed glass n=1 Furnace wall n=1	N=25
PA II	N=5 Stemmed goblets n=1 Vessel rims n=4	N=9 Glass chunks n=3 Test drops n=2 Deformed glass n=4	N=14
Série 3.2	N=2 Stemmed goblets n=2	N=1 Glass chunks n=1	N=3
unidentified	N=3 Stemmed goblet n=1 Vessel rims n=2	-	N=3
Total	N=55	N=21	N=76

A small fragment of glass, measuring less than $2 \times 2 \text{ mm}^2$, was removed from each sample using pincers and diamond cutting wheels attached to a Dremel hand drill. The small fragments were mounted in resin blocks and ground with silicon carbide papers of various grits (600, 800, 1200, 2500 and 4000). Finally, the blocks were polished using 3-6 μ m diamond suspensions, resulting in a flat surface, suitable for SEM analysis.

The glass samples (n=76) were analysed using a Zeiss Evo 15 scanning electron microscope (SEM), coupled with an Ultim Max EDS Detector (Oxford Instrument) housed at the Science and Technology in Archaeology and Culture Research Center (STARC), Cyprus Institute. The accelerating voltage was set at 20 kV, with a beam current of 1nA and a working distance of 8.5 mm. Analyses were done in 3-5 areas of c. 100 by 100 µm and the mean values calculated. The accuracy of the instrument calibration was tested using NIST 620, NIST 621, and Corning A and B standard reference materials (Table 2). The detection limit for most of the oxides analyzed is better than approximately 0.3 wt%. When the concentrations of the analyzed samples are above this threshold, the analytical results have an error margin lower than 10% (and in most cases lower than 5%). Data on the precision of the SEM-EDS analyses is included in the Supplementary material (SRM all sessions).

Part of the samples (n=45) were further analysed to detect their trace element composition using a Resonetics M50E excimer laser working at 193 nm coupled with a Thermo Fisher Scientific ELEMENT XR mass spectrometer at the Institut de recherche sur les archéomatériaux, Centre Ernest-Babelon (IRAMAT-CEB). The excimer laser was operated at 5 mJ with a repetition rate of 10 Hz. As a precaution, the beam diameter was adjusted from 40 μ m to 100 μ m to avoid saturation from elements such as manganese, copper, tin, antimony or lead. Even though no excessively high values for any of these

elements were expected, they could be concentrated locally as small inclusions. A pre-ablation time of 20 s was set and the signal was acquired for 27 s corresponding to 9 mass scans from Li to U (Gratuze 2014 and 2016). One to three ablations were carried out on each sample. Calibration for trace elements was performed using the NIST 612 standard reference material. Information on the precision of the LA-ICPMS is included in the Supplementary material (SRM all sessions). The results for minor and major oxides are very closely similar between the two methods used (Fig. 3). The only exception is in the data for titania which the SEM-EDS software systematically over-estimates by about 10% relative to the LA-ICPMS analyses as well as compared to the published Corning A data; accordingly, we have corrected our TiO_2 data in the table where we give the SEM-EDS data for minor and major oxides for all analysed samples, and the trace element data where available from the additional LA-ICPMS analyses.

Table 2. The measured, certified and relative error values in wt% for the standard reference materials as analysed by SEM-EDS. Certified / recommended values (Cert.) for SRM 620 and 621 from National Institute of Standards and Technology (NIST) and published values (Publ.) for CornA and B from Adlington (2017).

SRM621	Na₂O	MgO	Al ₂ O ₃	SiO ₂	SO₃	K ₂ O	CaO
(n=20)							
Meas.	12.22	0.27	2.81	71.76	0.11	1.99	10.83
Cert.	12.74	0.27	2.76	71.13	0.13	2.01	10.71
Error (% rel.)	-4.0	0.6	1.8	0.9	-11.9	-0.8	1.1

SRM620	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	SO₃	K ₂ O	CaO
(n=20)	Na ₂ O	IVIGO	A12O3	3102	303	K2O	CaO
Meas.	13.85	3.66	1.82	72.77	0.29	0.39	7.23
Cert.	14.39	3.69	1.80	72.08	0.28	0.41	7.11
Error (% relative)	-3.8	-0.9	0.9	1.0	2.7	-3.9	1.6

CornA				6:0										
(n=20)	Na₂O	MgO	Al ₂ O ₃	SiO ₂	SO₃	K₂O	CaO	TiO₂	MnO	FeO	CoO	CuO	Sb ₂ O₃	BaO
Meas.	14.19	2.65	0.94	67.61	0.17	2.90	5.15	0.86	1.05	0.99	0.17	1.23	1.59	0.49
Publ.	14.3	2.66	1.00	66.56	0.14	2.87	5.03	0.79	1.00	0.98	0.17	1.17	1.58	0.46
Error (% rel.)	-0.8	-0.3	-5.8	1.6	23.6	0.9	2.4	8.5	5.4	1.5	0	5.0	0.8	5.9

CornB	No O	Mao	41.0	c:o	D.O.		CI	к о	CoO	M=O	F=0	CO	7:0	sh o	DLO
(n=18)	Na₂O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO₃	CI	K₂O	CaO	MnO	FeO	CuO	ZnO	Sb ₂ O ₃	PbO
Meas.	16.7	1.0	4.2	62.5	0.8	0.6	0.2	1.1	8.6	0.3	0.3	2.8	0.2	0.3	0.5
Publ.	17.0	1.03	4.36	61.55	0.82	0.49	0.16	1.00	8.56	0.25	0.31	2.66	0.19	0.41	0.61

Error (% rel.)	-1.5	-3.0	-3.5	1.6	-6.1	21.4	4.2	5.2	0.8	0.7	-2.0	5.9	1.8	-15.6	-11.9

Table 3. The measured, certified and relative error values in ppm for selected trace elements in the standard reference materials and in in wt% for major and minor elements as analysed by LA-ICPMS. Certified / recommended values (Cert.) for SRM 612 from National Institute of Standards and Technology (NIST) and published values (Publ.) for CornB from Adlington (2017).

NIST612 (n=6)	Li	В	Ti	v	Cr	Mn	Со	Ni	Cu	Zn	As	Rb
Meas.	41.2	34.8	42.1	38.3	38.4	40.1	35.2	36.1	35.3	33.7	32.2	32.5
Cert.	40.2	34.3	44	38.8	36.4	38.7	35.5	38.8	37.8	39.1	35.7	31.4
Error (% rel.)	2.5	1.6	-4.4	-1.4	5.4	3.5	-0.8	-6.9	-6.5	-13.7	-9.9	3.6

NIST612 (n=6)	Sr	Υ	Zr	Sn	Sb	Ва	La	Ce	Nd	Pb	Th	U
Meas.	80.4	39.6	39.0	33.4	33.0	36.4	38.8	38.8	34.3	31.9	37.9	37.5
Cert.	78.4	38.3	37.9	38.6	34.7	39.3	36	38.4	35.5	38.6	37.8	37.4
Error (% rel.)	2.5	3.5	2.8	-13.5	-5.0	-7.3	7.6	1.0	-3.4	-17.4	0.3	0.3

CornB (n=6)	Na₂O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	CI	K₂O	CaO	MnO	FeO	CuO	ZnO	Sb ₂ O ₃	PbO
Meas.	16.67	1.02	4.43	62.52	0.83	0.22	1.01	8.51	0.24	0.37	2.68	0.21	0.43	0.41
Publ.	17.00	1.03	4.36	61.55	0.82	0.16	1.00	8.56	0.25	0.31	2.66	0.19	0.41	0.61
Error (% rel.)	-2.0	-0.9	1.6	1.6	1.5	36.1	0.5	-0.6	-2.3	20.9	0.7	12.3	5.0	-32.9

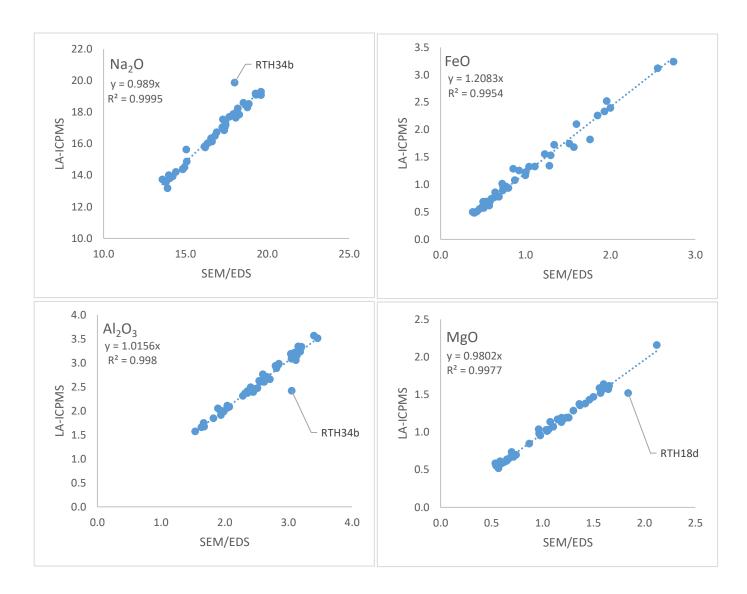


Figure XX. Comparison between the two techniques in specific oxides. In most cases the R² of the trendline is over 95% showing the good agreement between the two techniques. More comparison graphs can be found in the Supplementary material.

3. Results

Most of the analysed samples belong to the general soda-lime-silica category based on mineral natron flux (Table S1). The main glass former, silica (SiO_2), ranges between 63.0 to 73.5 wt% and is derived from sand, as indicated by the levels of iron oxide (0.3 to 2.7 wt% FeO) and alumina (1.5 to 3.5 wt% Al_2O_3) as impurities in the glass. Soda (Na_2O), the main flux used to melt the glass, ranges from 13.2 to 19.7 wt%. Lime (CaO), likely deriving from shell fragments naturally occurring in the sand, varies from 5.1 to 11.5 wt%. The origin of the lime from shells rather than limestone fragments is indicated by the amount of strontium in the samples, which ranges from about 350 to nearly 700 ppm Sr. Roughly speaking, sea shell fragments found in sands can add 300 to 600 ppm Sr to the glass (Brems et al. 2014), while limestone typically adds lower concentrations of Sr to the glass (Freestone et al. 2009: 35). Sand was fused predominantly with mineral natron, as is evident by the low amounts of

potash (0.4-1.5 wt% K_2O) and magnesium (0.4-1.6 wt% MgO) in most samples, almost all below the accepted upper threshold for mineral natron glass of 1.5 wt% K_2O and MgO, respectively (Lilyquist et al. 1993). However, one compositional group contains consistently higher levels of both oxides (1.4-2.1 wt% MgO and 1.1-1.7 wt% K_2O), as well as high phosphate (0.2-0.5 wt% P_2O_5), more in line with the use of plant ash. The overall glass composition is rather typical of glass found at the eastern Mediterranean from the period under study (Foy et al. 2003; Henderson 2000; Rehren and Freestone 2015; Ceglia et al. 2015). Systematic differences in major, minor and trace element composition indicate that the assemblage falls into different compositional groups, relating to separate glassmaking traditions and raw materials. Important for the interpretation of the site is the observation that the compositional differences between objects (obj) and glass working debris (glsw) are minimal across the entire analysed assemblage, as shown in whisker boxplots (Figure 3).

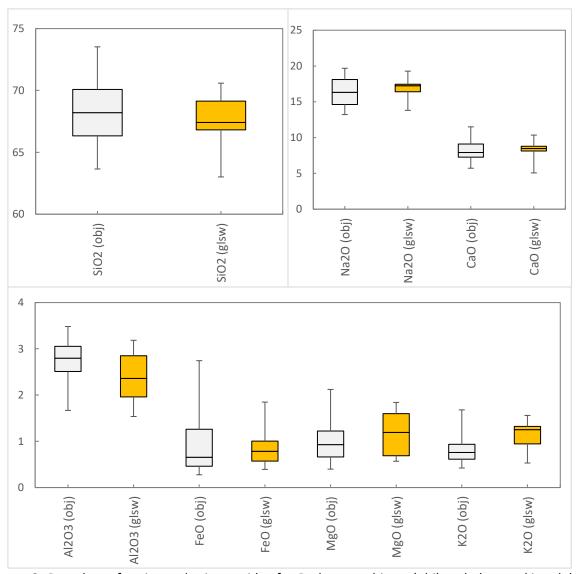


Figure 3. Box plots of major and minor oxides for Rethymno objects (obj) and glassworking debris (glsw). There is no significant difference between the two groups of samples. The whiskers show minimum and maximum values while the line in the box represents the median values of the corresponding oxides.

Recent research on glass from the mid- to late-1st millennium AD resulted in the identification of several broad compositional groups based on major and minor oxides as characteristic diagnostic elements. The main groups with a super-regional distribution include: Levantine I and II (Freestone 2005) manufactured in the Levantine coast in large primary glass production sites (Apollonia, Bet Eli'ezer etc.), Egypt I and II manufactured most likely in Egypt (Gratuze 1988), Série 2.1 and Série 3.2 which were also identified as an Egyptian manufacture (Foy et al. 2003), and finally the glass high in iron, manganese, and titanium, the so-called HIMT glass (Freestone 1994; Freestone et al. 2018), first identified by Mirti et al. (1993) and labelled Groupe 1 in Foy et al. (2003), which was also manufactured in Egypt (Nenna 2014). A number of other compositional groups were used during this period, too, but are mostly only of local or regional significance.

These seven main groups of first millennium glass can be easily distinguished in an Al_2O_3/SiO_2 vs TiO_2/Al_2O_3 biplot. These three oxides are incorporated in the glass as part of the sand and, therefore, their correlation can show possible distinctive different glassmaking regions through the use of different sands.

In Figure 4 the glass from Rethymno correlates well with Levantine I, Série 2.1 and Série 3.2, while in a first impression there is also a good correlation with Egypt II glass. However, the levels of MgO and K_2O in Egypt II glass average only 0.5 and 0.3 wt% respectively, while the corresponding samples from Rethymno have significantly higher mean values of both oxides (1.3 and 1.6 wt% respectively), as well as much higher phosphate levels than those found in other mineral natron glass groups. The excess in these three oxides is potentially an indication of the use of plant ash for the fusion of sand (see below). A very similar glass compositional group has recently been identified by Rosenow and Rehren (2018) in glass samples from Upper Egypt, dating to a similar period as the Rethymno glass, and labelled as PA II. Because of this similarity (Table 4), this group of glass will be referred to as PA II from here onwards, until a larger set of data becomes available to determine their potential relationship more clearly. There are also three samples (marked as outliers) that cannot be assigned to any of the aforementioned groups.

Table 4. Comparison of the mean values for the major and minor oxides between RETH PA II data, PA II (Rosenow and Rehren 2018) and Série 2.1 and Série 3.2 (Foy et al. 2003). In bold the values that are similar between the groups. It seems that RETH PA II group has more similarities with PA II (Rosenow and Rehren 2018).

	Na₂O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO₃	Cl	K ₂ O	CaO	TiO ₂	MnO	FeO
RETH PA II	17.38	1.63	1.87	67.21	0.32	0.31	0.86	1.31	8.15	0.21	0.18	0.88
PA II (RoRe 2018)	16.51	1.48	2.25	66.20	0.32	0.33	0.71	1.22	8.14	0.15	0.25	0.97
Foy 2.1	17.91	1.11	2.54	66.08	0.17	0.39	0.8	0.9	7.55	0.19	0.96	1.43
Foy 3.2	18.72	0.67	1.97	68.55	0.07	0.34	0.96	0.57	6.77	0.11	0.76	0.57

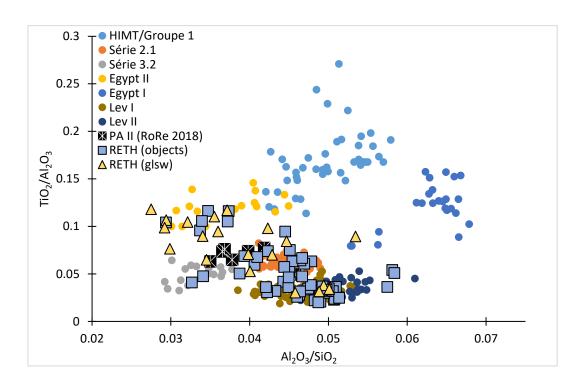


Figure 4. The correlation of objects (obj) and glassworking debris (glsw) from Rethymno compared to some of the main compositional glass groups of late antiquity. Each of the four identified compositional groups is represented by glassworking debris as well as object fragments. There are no samples from Rethymno belonging to the HIMT and Egypt I groups. Underlying data used in this graph are from Freestone 2005 (Lev I and II), Foy et al. 2003 (HIMT/Groupe 1, Série 2.1, Série 3.2), Rosenow and Rehren 2018 (PA II), and Gratuze 1988 (Egypt I and II).

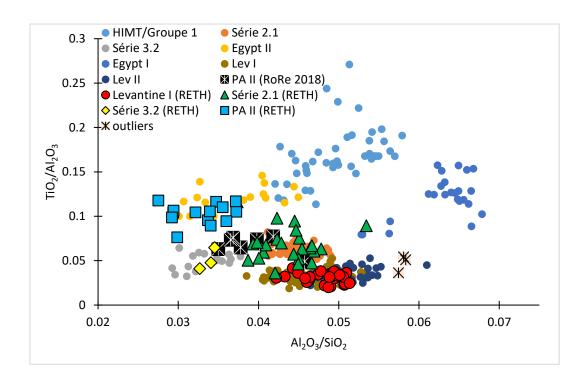


Figure 5. The glass from Rethymno can be distinguished in four groups with chemical compositions matching published data, namely Levantine, Série 2.1 and Série 3.2, and one more group that even though it correlates well with Egypt II it does not belong to this group due to higher MgO and K_2O content. Three samples cannot be assigned to any of the groups. Underlying data are as given in Figure 4.

In Figure 5 we assign the Rethymno glass (both objects and glassworking debris) to the four compositional groups of glass offering a clearer view of the corresponding groups. The Levantine glass from Rethymno forms a coherent group (red circles) as also seen in the Na₂O/SiO₂ against CaO/Al₂O₃ graph (Figure 6). Recently, Phelps et al. (2016: 60-1) highlighted the separation of the Levantine I group sensu Freestone into two chemically distinct groups for Apollonia and Jalame, respectively, and suggested to use these more specific production site names where possible. However, the Rethymno Levantine glass data straddles the Apollonia-type and Jalame-type glass compositions (Fig. 6); accordingly, we use the more generic label Levantine I to refer to this compositional group. Both Série 2.1 and PA II groups are somewhat more dispersed in this presentation than Levantine I, despite consisting of fewer glass samples, while Série 3.2 group is represented only by three samples.

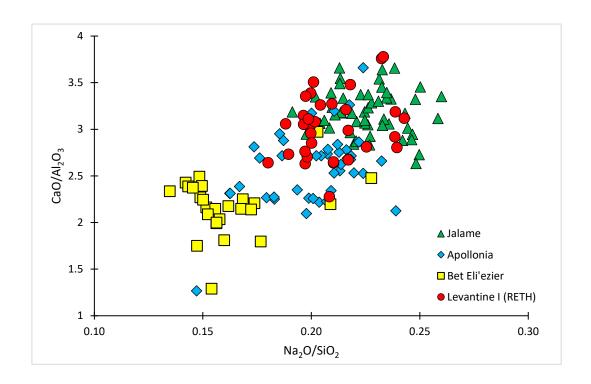


Figure 6. CaO/Al_2O_3 vs. Na_2O/SiO_2 plot demonstrating the separation between the main Levantine groups (Phelps et al. 2016). The Levantine I group is represented by data from Jalame and Apollonia (Brill 1988; Freestone et al. 2000; Tal et al. 2004; Freestone et al. 2008), and Levantine II by the Bet Eli'ezer data (Freestone et al. 2000). The Rethymno samples correlate with the Levantine I group, matching both Jalame and Apollonia data.

Gratuze (2013) used the scatter plot Y_2O_3 vs ZrO_2 to show a systematic difference between glass from Egypt (high in ZrO_2) and glass made in the Levant, which is proportionately richer in Y_2O_3 . Applying this test to the glass samples from ancient Lappa confirms that the PA II glass is consistent with an origin in Egypt, falling between Egypt I and Egypt II (Figure 7) — as had already been indicated by the close overlap with Egypt II in the initial graph based on minor oxide (Figure 5).

In this plot we also notice that the three samples not assigned to any compositional group are probably of Levantine origin, although their high soda content (>18 wt% Na₂O) and elevated titania (>0.15 wt%) point to an Egyptian origin. We further note that the Série 2.1 glass falls between the broad trend lines for Egypt and the Levant, respectively, with no clear correlation to either of them. Interestingly, the PA II group can be further distinguished in two groups, both having positive correlation, but with different slopes, and each including both objects and glassworking debris.

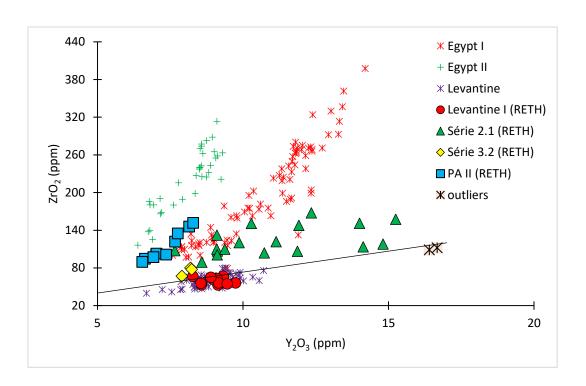


Figure 7. ZrO_2 vs. Y_2O_3 . The PA II group falls between the two established Egyptian glass groups (data from Schibille et al. 2019). The line represents the separation between Levantine and Egyptian glass compositions as proposed by Gratuze (2013); the three outlier samples are likely of Levantine origin.

The potash (K₂O) against magnesium (MgO) plot (Figure 8) separates the samples in two groups (indicated by the two dashed ellipses), which is more evident in the glass working debris samples. The first group on the left of the graph exhibits rather constant values of MgO (around 0.6-0.7 wt%) and varying K₂O content (0.5-1.5 wt%), while the second group shows values of MgO above 1.0 wt%. It seems that some of the samples, including the outliers, most of the Série 2.1 samples and all PA II samples have a positive correlation, within which the PA II glass seem to form a continuum with Série 2.1 samples. This continuum, however, disappears when we test the sum of the plant ash discriminative oxides i.e. MgO, K₂O and P₂O₅ against MnO (Rosenow and Rehren 2018, Fig. 12.4). The average ratio of MnO/Sum for the Série 2.1 glasses is 0.44, while for the PA II it is 0.05, clearly distinguishing the two groups.

Furthermore, a strong positive correlation is also noticed in the potash (K_2O) and phosphorus (P_2O_5) plot (Figure 9). The PA II glasses have the highest amounts of both oxides, potentially indicating that they were fused using plant ash. For the rest of the samples, which were fused with natron, this positive correlation seamlessly continues to lower concentration levels, suggesting that a single compound adds both elements in the glasses at a rather stable proportion to each other. It is unlikely that this would have been a consciously added component, but could be explained as contamination from fuel ash during the melting of the glass. In a series of glass melting experiments Paynter (2008) noticed significant accumulation of potash in glass batches depending on the time the glass batch remained in the furnace. Glass heated for more than 30 hours showed potash values of around 2.0 wt%; when heated for much longer potash levels raised over 2.5 wt%, due to the exposure to fuel ash vapour and particles for longer time and more often, resulting in an increase of both potash and phosphorus oxide (Rehren et al. 2010: 75-76).

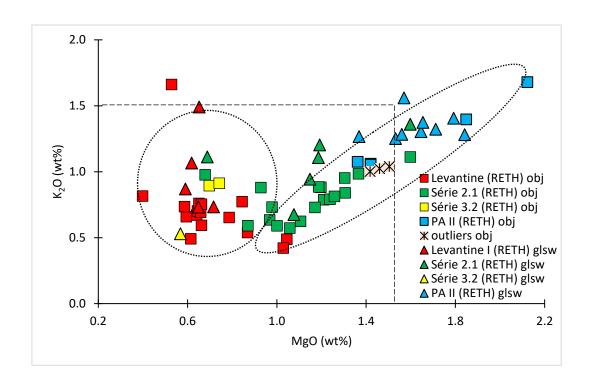


Figure 8. The correlation between magnesia (MgO) and potash (K_2O) for the Rethymno objects (obj) and the glassworking debris (glsw) for the four corresponding groups. The assemblage can be separated in two groups, one of which presents a positive correlation and high levels of both magnesia and potash. The straight dashed lines represent the separation threshold values between natron and plant ash glass as suggested by Lilyquist et al. (1993). The two ellipses indicate possibly two separate groups as discussed in the text.

4. Discussion

The assemblage consists of four different compositional groups representing different glassmaking centres. Three are from more widely-known productions in modern-day Israel and Egypt, respectively, namely Levantine I, Série 2.1, and Série 3.2, while one is of more limited distribution of likely Egyptian origin, namely PA II; a further three samples could not be assigned to either of these groups. Each of the four groups is represented as fragments of objects and as working waste, suggesting at a first glance that the workshop processed all four different types of glass.

The analysed material may represent different periods of production, ranging from the 4th and 5th century AD as indicated by the pottery associated with the circular feature tentatively identified as a glass furnace, to the destruction layer of the 7th century AD. Due to the levelling activity and disturbance of the soil from building activity it is not possible to stratigraphically date the glass fragments, and their degree of fragmentation prevents morpho-typological dating, too. However, elsewhere the different compositional groups have been linked to broad chronological periods of their use. In Britain and NW Germany, Foy's Série 3.2 glass is reported from early 4th c. AD contexts (see Rehren and Brüggler 2020: 12 for a discussion), and in Bulgaria, it is linked to assemblages from the late 4th to early 6th c. AD (Cholakova and Rehren 2018: 46), while Foy's Série 2.1 falls mostly into the 6th c. AD (Cholakova et al. 2016). The PA II glass from Armant (Rosenow and Rehren 2018) is only very

broadly dated to around the 5th c. AD. The Levantine I glass, linked to the 6th century furnaces in Apollonia (Freestone et al. 2000; Tal et al. 2004), occurs in Palestine still throughout the 7th century (Phelps et al. 2015: 63), and is therefore the most recent of the glass compositions present at the Rethymno assemblage. Thus, the occurrence of several samples of Série 3.2 glass is consistent with the suspected 4th/5th c. glass kiln in Argyroupoli, while the samples of Série 2.1 and Levantine I glass match the period immediately prior to the destruction layer of the 7th century AD.

Taking into consideration the fact that there is no complete glass object in the Rethymno assemblage and only a very small number of fragments can be assembled, we might assume that the fragments were collected elsewhere and brought to the workshop intended for remelting/recycling. Interestingly, the glassworking debris in each of the compositional groups have on average about 10 to 20% higher amounts of the typical fuel ash oxides phosphate and potash (Table 1; Fig. 9) compared to the corresponding objects. The increase of these two oxides with each additional working step (recycling/remelting, longer exposed to the furnace atmosphere etc.) due to contamination from fuel ashes has been first shown experimentally by Paynter (2008) and since observed in several assemblages (e.g. Rehren et al. 2010, 2016). Therefore, it seems that the glass working debris are one step further in the number of recycling events for the glass, and therefore less likely to be imported cullet from which new vessels were made. Instead, we believe that the broken vessel fragments were brought to the site as cullet to be worked locally, with the working debris evidence for this activity and preserving the increased concentrations of fuel ash components as the result of the additional melting time involved. This interpretation, and the seemingly unbroken spread of compositions in the potashphosphate diagram (Figure 9) raises the question whether the PA II glass is indeed based on plant ash as the main flux, as proposed by Rosenow and Rehren (2018), or whether we see here another natronbased glass group that is just more heavily contaminated by fuel ash than other compositions. This does not necessarily have to be due to repeated recycling, but could, for instance, be a reflection of a different furnace design, firing regime or fuel employed in the making of PA II, compared to the practices used for making glass of the undisputed natron-based compositions.

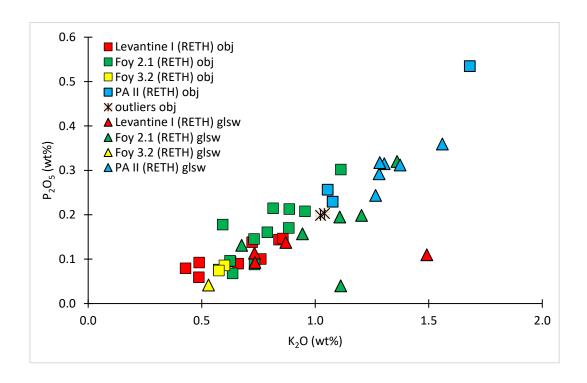


Figure 9. The strong positive correlation between potash (K_2O) and phosphorus oxide (P_2O_5), and their on average higher levels in the working debris compared to the object fragments of the same compositional group, might be an indication that both occur as contamination from fuel ashes after a series of recycling events.

Furthermore, among the glassworking debris there are in total seven chunks of glass, covering all four groups of glass (Table 1). These chunks can be either the remaining glass left in the kiln after working/recycling is finished, or fresh glass from the primary tank furnaces in the Levant or/and Egypt. The fact that five out of seven samples have adhering furnace wall material points to the idea that they probably are the remains of the glassworking activity, possibly residual glass at the bottom of the melting installation which could not be further exploited. Interestingly, these five samples belong to PA II (3 samples) and Série 2.1 (2 samples) groups, which then would indicate that these two groups were indeed worked in the workshop. Additionally, the fact that these chunks have higher amounts of indicative trace elements than the corresponding glass objects (Figure 9) may well be a result of additional rounds of recycling events. The other two chunks do not show any adhering furnace wall material and belong to Levantine I and Série 3.2 group, respectively. The Levantine I chunk most likely is the product of recycled glass, since it has also higher values of the corresponding trace elements as in the case of PA II and Série 2.1 samples (Figure 9). The opposite is true for the Série 3.2 chunk which has lower trace elements than the objects, and we may assume it was imported to produce a limited amount of glass objects. In the investigated assemblage there are only two fragments of glass vessels belonging to this group. However, we should also take into consideration the uncontrolled sampling and also the low proportion of samples we analysed compared to the total of more than 1500 available fragments.

The fact that PA II was more frequently worked than the other two main groups is also reflected in the total number of glassworking debris within the analysed assemblage. PA II group contains 9 samples of working waste and only 6 object fragments. In contrast, 3 and 5 times as many object fragments than working waste were analysed for Série 2.1 and Levantine I, respectively. It seems that the prevailing group at Lappa is PA II, the glass composition very similar to the one Rosenow and Rehren (2018) have recently identified in Upper Egypt. To our knowledge, this is the first time this compositional group is reported outside Egypt, and remarkable that it occurs here in substantive quantities. However, we should take into consideration that the sampling procedure can be biased since we sampled only a small proportion of the total glass fragments. The PA II group, both objects and glassworking debris, shows a consistent colour which is slightly greenish compared to Levantine group (more aqua blue) and the Série 2.1 group (more yellow and darker green).

4.1. Recycling indicators

The level of recycling in both objects and glassmaking debris was tested by investigating specific elements which act as recycling markers. In particular, elements used as colourants (Co, Cu) or decolourants (Sb, Mn) can show the potential degree of recycling (Jackson 1997; Smirniou and Rehren 2013; Freestone 2015). Rehren and Brüggler (2015: 174, and references therein) identified threshold concentration values of elements, such as Cu, Pb, Sn and Sb, above which these are indications of recycling; these values differ significantly for different base glass compositions and are much higher for HIMT (typically multiple tens of ppm) than for Levantine glass (mostly single ppm). Another indication of recycling could be the simultaneous presence of both manganese (Mn) and antimony (Sb) in the low fraction of a percent level, which is likely the result of mixing Mn-decolourised glass with Sb-decolourised glass during remelting (Jackson 1997; Freestone 2015).

These conditions were tested in the Rethymno assemblage and it appears that there is not extensive recycling. In Figure 10, we test the correlation between Mn and Sb and there is some recycling mostly in the Série 2.1 samples and to a lesser extent in the PA II samples. The same is noticed also in Figure 11, where Cu and Pb are investigated (four samples, three vessels and one glassworking debris, were excluded because they are intentionally coloured blue and have significant high values of Cu and Pb). Interestingly, according to both graphs (Figure 10, 11) the Levantine glass shows almost no evidence of recycling, while the "outliers" samples were most likely recycled. A similar behaviour is noticed when testing the mean values of these elements for the corresponding groups (Figure 12). According to this graph, Série 2.1 and the "outliers" samples show the highest values of these indicative elements, suggesting a higher degree of recycling, while the later glass (Levantine I) shows little evidence for recycling, as would be expected for a compositional group newly emerging towards the end of the life cycle of the site.

Overall, it is not immediately clear whether the fragmented glass objects represent the products of the workshop, or cullet collected for remelting to produce new objects. It is possible that at least some of the glassworking debris was among the cullet collected and brought to the furnace from elsewhere; however, as argued earlier in this paper we believe that most of the glassworking debris represents local working, while the broken objects are more likely cullet for remelting. Lastly, the workshop could have operated at any one of a number of different scales and socio-economic settings. It could have been a small workshop dependent on opportunistic glass supplies procured from the surrounding settlements, or from the occasional merchant ship carrying cullet as part of its cargo; such shipments

are known e.g. from the earlier *Iulia Felix* in the Adriatic Sea (Silvestri et al. 2008; Freestone 2015), and from the later *Serçe Limani* on the Turkish coast (Bass et al. 2009). Alternatively, it could have been a more established workshop, able to order or organise glass shipments, either of chunks fresh from some primary furnaces or recycled cullet.

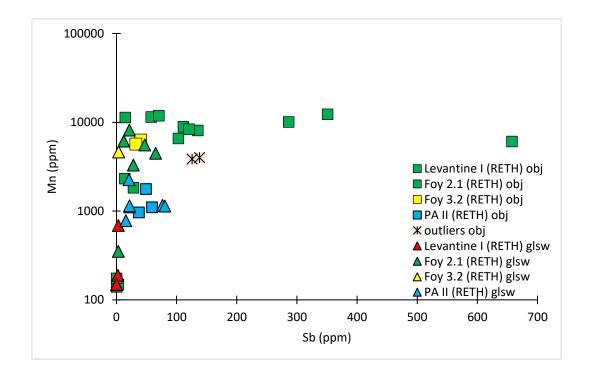


Figure 10. The majority of Série 2.1 and PA II samples show elevated amount of Sb which is a sign of recycling.

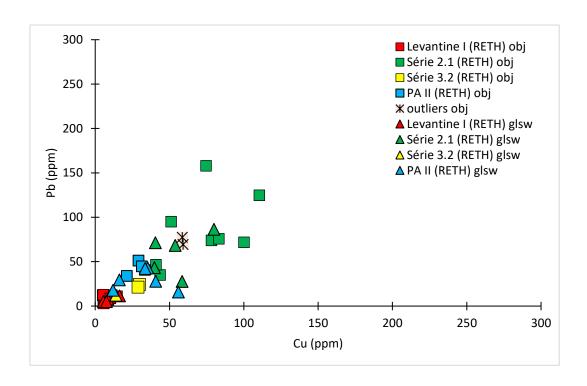


Figure 11. The same is noticed in this graph; Série 2.1 and PA II samples have elevated levels of both Cu and Pb compared to Levantine I which, in both graphs, forms a homogeneous group with minimum recycling.

Whatever the case is, the use of different glass compositions highlights the versatility and longevity of the workshop at ancient Lappa, and possibly indicate that the glassmakers were dependent on erratic supplies of glass, accordingly producing objects with different chemical compositions. It is rather remarkable that there is little mixing between these four groups. We do not notice any mixed compositions of the groups and there is little overlap between them in specific biplots. Each group is rather homogeneous and "clean" in terms of its chemical composition. This has been regularly seen in other workshop assemblages where multiple compositional groups were present (e.g. Rehren and Brüggler 2018). Some of this may be due to the difference in chronological 'currency' of the different compositions, rendering them less likely to be mixed if contemporaneous cullet is being recycled; another reason may be that different glass compositions have different hues, enabling the workmen to keep them separate when re-melting cullet. In any case, the evidence here indicates a level of professionalism on a par with other workshops of the time.

The variability in glass used, including glass chunks as well as likely cullet shows that the workshop in Argyroupolis used multiple sources for its glass. The glass originates in two different broad geographic regions, with the early glass coming from Egyptian production sites and the later from the Levantine coast. However, we have to take into consideration the fact that cullet used for recycling/remelting potentially can originate from a much broader area in the Mediterranean where glass was being used, and not necessarily come directly from the two geographically limited major sources of raw glass. In general, these two broad regions of origin for the Rethymno glass and the potential even wider origin of fragmented glass objects reflect the significant and well-connected position of Crete in trading activities during the period under study, as well as the changing compositions of glass available at any one time.

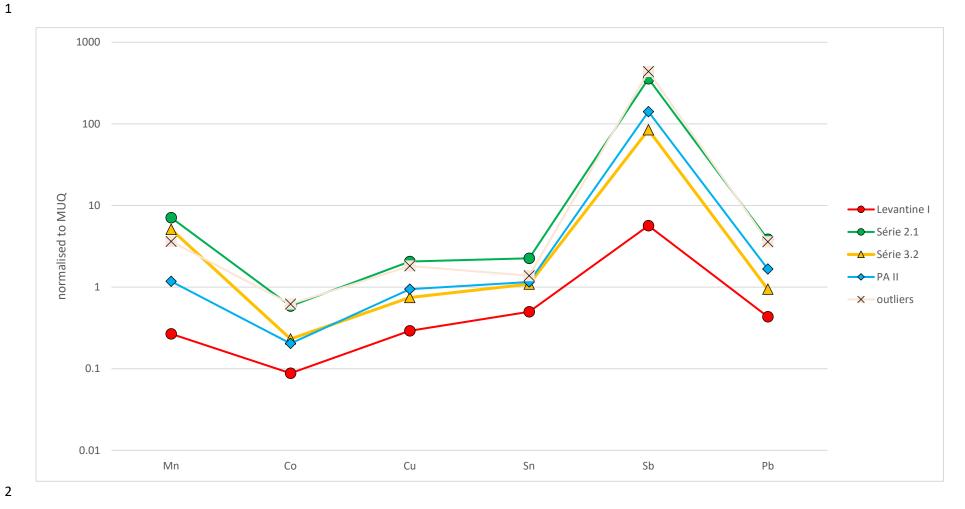


Figure 12. The mean value of the trace elements which are commonly used as indicators for recycling for the five corresponding groups. The Série 2.1 group and the "outliers" show the highest values of the corresponding elements, indicating a higher degree of recycling. The data are normalised to the upper continental crust (MUQ, Kamber et al. 2005). Sb is normalized using the data from Wedepohl (1995). The four coloured samples are excluded from this graph.

Conclusions

The available analytical data gives an insight into the longevity and likely importance of Argyroupolis' workshop during the early Byzantine period in Crete. The assemblage shows interesting features in terms of its chemical composition and can be characterised as a rather complex material. Four different glass compositions were identified, namely Levantine I, Foy Série 2.1, Foy Série 3.2, and PA II. All four compositions were assigned to both broken objects and glassworking debris, indicating that glassworking continued throughout the period under study, even though remains of a potential glass furnace were only found in the earlier layers. We note the shift in geographical origin from Egypt for the earlier glass to the Levant for the latest finds; a similar trend is also seen in the glass supply of Cyprus, where the proportion of Levantine glass increases over time (Ceglia et al. 2015: fig. 5). The authors link this in part to the geographic position of the three sites in question, but favour a broader chronological trend across the eastern Mediterranean as a more plausible reason (Ceglia et al. 2015: 220-1).

There are signs of recycling in the glass assemblage. Systematic subtle differences in composition between glassworking debris and objects might indicate that the majority of glass fragments found in Argyroupolis were intended for remelting/recycling, acting as cullet, and were not the product of the workshop. This idea is also reinforced by the existence of chunks with adhering furnace wall material which probably were remains of the glassworking activity rather than imported material for remelting. In addition, the trace element fingerprint and the intercomparison between objects and glassworking debris suggests this pattern.

The fact that the primary glass comes both from the Levantine coast and Egypt underlines the importance and good integration of Crete in trade activities in the SE Mediterranean, and especially highlights the chronologically enduring role of Argyroupolis in glass distribution. A similar supply of glass from multiple large production centres has been already noticed in Cyprus during the early Byzantine period (Ceglia et al. 2015; Cosyns and Ceglia 2018), with the same shift from earlier Egyptian glass to later Levantine glass.

Beyond this initial assessment, comparative data from ongoing research from both consumption sites (Gortyna, Eleutherna) and contemporary workshops (Knossos, Eleutherna) in Crete is expected to provide further details of the distribution of glass on the island, and the position of Argyroupolis in the glassmaking tradition of Crete.

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Supplementary material

Table I. Table of analysis in excel

Table II. Catalogue of samples ($\alpha.\epsilon.$: inventory number)

A/A	Sample	Description	Photograph
1	RETH001 (α.ε. 1074)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base. Bicoloured.	1 cm
2	RETH002 (α.ε. 1074)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	1 cm
3-5	RETH003 (α.ε. 1072)	a-c) Rim fragments.	a b c
6	RETH004 (α.ε. 1072)	Rim fragment	1 cm
7	RETH005 (α.ε. 1072)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	1 cm

8	RETH006 (α.ε. 1035)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	1 cm
9	RETH007 (α.ε. 1035)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	1 cm
10	RETH008 (α.ε. 1035)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	1 cm
11-13	RETH009 (α.ε. 1035)	a-b) Rim fragments. c) Foot base fragment.	a b c
14	RETH010 (α.ε. 1035)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	I cm
15	RETH011 (α.ε)	Glass waste. Deformed glass mass.	1 cm

16	RETH012 (α.ε. 1078)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	1 cm
17	RETH013 (α.ε. 1078)	Body vessel fragment.	1 cm
18	RETH014 (α.ε)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base. Bicoloured.	1 cm
19	RETH015 (α.ε)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	1 cm
20-23	RETH016 (α.ε)	a-d) Rim fragments.	a b c
24-28	RETH017 (α.ε)	a-e) Rim fragments.	a b e c d

29-32	RETH018 (α.ε)	a-d) Glass wastes. Deformed glass masses.	a b C d
33	RETH019 (α.ε)	Cullet.	1 cm
34-36	RETH020 (α.ε)	a-c) Rim fragments.	a b
37	RETH021 (α.ε)	Cullet with ceramic attached to it. Possibly from the floor or wall of the furnace.	1 cm
38-41	RETH022 (α.ε. 857)	 a) Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base. b) Fragment of conical, with knob or pointed base, lamp. c-d) Rim fragments. 	a b d
42-46	RETH023 (α.ε. 857)	a-e) Rim fragments.	a d d e

47-49	RETH024 (α.ε. 857)	a-b) Cullets c) Glass waste. Deformed glass mass.	a b c
50	RETH025 (α.ε. 857)	Cullet.	i cm
51-52	RETH026 (α.ε. 859)	a-b) Rim fragments.	a b
53-54	RETH027 (α.ε. 859)	a) Cullet. b) Glass waste. Deformed glass mass.	a b
55	RETH028 (α.ε)	a) Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	â lan
56	RETH029 (α.ε)	Rim fragment.	Len

57-58	RETH030 (α.ε. 903)	a) Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base. b) Rim fragment.	a b
59-61	RETH031 (α.ε. 882)	a) Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base. b-c) Rim fragments.	b a a c
62	RETH032 (α.ε. 912)	Stem of cylindrical or inverted conical, in the type of beaker with a foot and disk-shaped base.	i cm
63-66	RETH033 (α.ε. 912)	a-d) Rim fragments.	a b C d
67-71	RETH034 (α.ε. 857)	a-e) Glass waste. Test drops.	a b e c d
72	RETH035 (α.ε. 862)	Cullet with ceramic attached to it. Possibly from the floor or wall of the furnace.	1 cm

73	RETH036 (α.ε. 997)	Glass fragments attached to a clay-sediment mass. Possibly from the floor or wall of the furnace.	1 cm
74	RETH037 (α.ε)	Glass fragments attached to a clay-sediment mass. Possibly from the floor or wall of the furnace.	1 m