

# First evidence in Italian mainland of Pantelleria obsidian: highlights from WD-XRF and SEM-EDS characterization of Neolithic artefacts from Galliano necropolis (Taranto, Southern Italy)

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## Abstract

In the Late Neolithic necropolis of Galliano (Taranto, Southern Italy) 52 samples of obsidian artefacts were found. Their characterization by absolutely non-destructive techniques like WD-XRF and SEM-EDS, for almost all of the samples, recognized the Lipari source for the raw material. Two samples show a different origin area: one from Palmarola, the southernmost signalling in Puglia for this source, and one from Pantelleria (sub-source of Salto la Vecchia and Balata dei Turchi) as first signalling on the continental Italy. Both source area, Palmarola and Pantelleria, have also been safely ascertained through quantitative compositional analyses of the microphenocrysts (pyroxenes and feldspars) present in the glass of 27 and 42 obsidian samples. In particular, the discovery of obsidian from Pantelleria in the Late Neolithic necropolis of Galliano, which can be traced back to the first centuries of the second half of the fifth millennium BC, significantly expanded its distribution area towards the east and the north, in a chronologically well-defined context.

**Keywords:** obsidian provenance, Late Neolithic Galliano necropolis, Southern Italy, WD-XRF, SEM-EDS, non-destructive analyses, Pantelleria.

## 1. Introduction

For several years a research program dedicated to the provenance study of obsidian artefacts found in different archaeological sites of Mediterranean area has been conducted by the Department of Earth Sciences and Geo-environmental of the “Aldo Moro” University of Bari (Italy) in close collaboration with the territorially competent Superintendence. The use of a fast and absolutely non-destructive analytical protocol is proved suitable for the treatment of a great number of samples (Acquafredda et al., 2018).

The main source sites signalled in the central-eastern Mediterranean are those of the islands of Lipari, Pantelleria, Sardinia, Palmarola, Melos, Antiparos and Gyalì (Acquafredda et al., 1999, 2006, 2019; Fig. 1).

Provenance studies regarding numerous Italian and Mediterranean sites have shown the different trade of sources and sub-sources of the outcrops of Lipari, Monte Arci (Sardinia), Palmarola and Pantelleria. The analysis of obsidian artefacts can contribute to a more detailed reconstruction of the sourcing and trade of the raw obsidian and their distribution network and related socio-economic aspects in the past (Ammerman and Polglase, 1993; Robb and Farr, 2005; Pessina and Radi, 2006; Vaquer, 2007; Freund, 2013; Tykot, 2017a).

In particular, for the Apulia, an overall picture of the attested sources and their frequencies in the different phases of the Neolithic has been outlined in adequate detail (Acquafredda et al., 2017 and the references therein): Lipari source in absolute prevalence, sometimes exclusive, during all Neolithic phases (Freund, 2018); Palmarola source, in very limited quantities, especially in the sites from the Apulia Tavoliere plain and Murge plateau, referable from an advanced phase of the Early Neolithic to the Middle Neolithic; Monte Arci source (Perdas Urias) reported in an isolated case at

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the Pulo di Molfetta doline among the artefacts dated back to the middle/final Neolithic (Acquafredda, Muntoni 2008).

With the aim of enriching the reference database under construction, new archeological obsidians coming from the Late Neolithic necropolis of Galliano (40°32'54.4"N, 17°02'40.1"E; Palagianò, Taranto province, Southern Italy), excavated by the Soprintendenza Archeologia della Puglia (Radina 2017; Venturo, D'Onghia 2017) between 2009 and 2012 (Fig. 1), were analysed, for a total of 52 finds. In particular obsidian artefacts are documented in the fillings of eight different funerary structures (Tombs 3-8 and 10-11), with a particular frequency of finds in T5 (n = 8), T7 (n = 9) and T4 (n = 15). There is no evidence of deposition of obsidians artefact as grave goods of burials. The collection consisted of bladelets, flakes and chips; no cores have been found (Fig. 2).

## 2. Analytical methods

After a washing in distilled water, the samples were chemically characterized in a non-destructive way by means of a Wavelength Dispersion X-ray Fluorescence spectrometer (WD-XRF). The intensity ratios of some trace elements such as Rb, Sr, Y, Zr and Nb were measured as suggested in Acquafredda et al. (2018). The WD-XRF spectrometer used is a Panalytical, AXIOS-Advanced apparatus, equipped with a rhodium super sharp end-window X-ray tube (Fig. 1). The obsidian samples were placed in the centre of a sample holder, which had previously been closed at the bottom with a thin polyester film (Mylar ©). Analytical operating conditions were X-ray tube set at 60 kV and 66 mA; LiF 220 diffracting crystal; scintillator detector for X-rays collection (Acquafredda et al., 2018).

For the two samples Gal 27 and Gal 42 it was necessary to strengthen constrain the obsidian provenance and therefore major elements of the glass and the rare microphenocrysts included in it were analysed by SEM-EDS techniques. The two samples were submerged in cold acetic acid for 6 hours in order to remove any carbonate encrustations (Acquafredda et al., 2018). Each sample was then fixed on an aluminium support (stub) using plasticine (Fig. 1) so that one of the surface of the artefact would have turned out to be perpendicular to the SEM electron beam. Subsequently the surface of the samples was covered by a 30 nm carbon film using an Edwards Auto 306 thermal evaporator.

SEM-EDS analyses were performed with a LEO-EVO50XVP coupled with an X-max (80 mm<sup>2</sup>) Silicon drift Oxford detector equipped with a Super Atmosphere Thin Window© (Fig. 1). Quantitative analyses were obtained using the following operating conditions: 15 kV accelerating potential, 500 pA probe current, about 25 kcps as output count rate on the whole spectrum, counting time 50 s and 8.5 mm working distance. Oxides in wt% were obtained converting the X-ray intensities by XPP correction method developed by Pouchou and Pichoir (1988, 1991), granted as quantitative software support by Oxford-Link Analytical (U.K.). The accuracy of the analytical data was checked using standard minerals manufactured by Micro-Analysis Consultants Ltd. (U.K.), for mineral phases taking into particular account the crystal chemical formula.

## 3. Provenance data

The X-ray intensity ratios of the trace elements, determined for the 52 obsidian samples from the Galliano necropolis, were compared with those already determined for the main obsidian source areas of the central-eastern Mediterranean basin (Acquafredda et al., 2018, 2019).

In fact various trace elements X-ray intensity ratios can be used to determine obsidian source areas (De Francesco et al. 2011) which are very successful in giving indications on obsidian geological outcrops (De Francesco et al. 2011; Acquafredda et al. 2018); in particular “the use of a log-log plot, that minimizes non-significant differences” (Acquafredda et al. 2018) very quickly and effectively allows to attribute to an obsidian artefacts its source area (Fig. 4).

The WD-XRF data (Table 1), using a Nb/Sr vs Rb/Sr log-log plot (Fig. 4), clearly indicate, for most of the samples (50 artefacts) found in Galliano, provenance from the island of Lipari. Only two samples show different source areas: in particular the sample 27 from the island of Pantelleria and the sample 42 from the island of Palmarola (Fig. 4). All provenance data are confirmed using the

Rb/Sr vs Zr/Y semi-log plot that allow to better define also the sub-source “Salto la Vecchia and Balata dei Turchi” of Pantelleria for the sample 27 (Fig. 4).

The identification of a sample of Palmarola represents the southernmost find in Puglia for this Mediterranean source; even more interesting is the documentation of a Pantelleria sample in the Galliano site, representing the first evidence of this source in the continental Italy.

The contextual identification of three different sources in the studied Late Neolithic necropolis, two of which attested by a single obsidian sample, was possible through a comparison with the systematic analyses conducted on the entire body of Mediterranean finds by the effective analytical protocol developed by Acquafredda et al. (2018). Confirmation of some provenance data, for samples whose source area was significantly important to attest new exchange trades (Gal 27 and Gal 42), was validated through glass and microphenocrysts analyses at SEM-EDS (Figg. 4, 5; Tab. 2, 3).

Regarding the attribution of the Gal 27 sample to Pantelleria source, the SEM-EDS analysis of its glass, showing a silica content of 72.7 wt%, already represent a stringent constraint (Fig. 4). In addition, quantitative analyses performed on feldspar microphenocrysts (anorthoclase and sanidine) in the same sample (Figg. 4, 5; Tab. 3) constitute a further proof indicating a clear provenance from Pantelleria.

In the case of the Gal 42 sample, on the other hand, SEM-EDS analysis of the glass alone was not sufficient to allow a certain attribution to Palmarola source. However, SEM-EDS microanalysis of small augitic pyroxene crystals (Fig. 4; Tab. 3) confirms the sure provenance from Palmarola. The strongly corroded surface of Gal 42 obsidian, on which the alteration highlighted the presence of microphenocrysts of feldspar also, for the first time allowed their chemical characterisation as anorthoclase (Fig. 4; Tab. 3), like as signalled in the Monte Arci source area, as reported in Acquafredda and Paglionico (2004).

#### **4. Discussion of the results**

The absolute prevalence even among the Galliano samples from the Lipari source is absolutely in agreement with what already known about the dynamics of obsidian diffusion in central-southern Puglia (Acquafredda et al., 2017), especially in middle and final Neolithic. This datum was also further confirmed by provenance study on obsidians from Serra Cicora (n = 4), Carpignano Salentino (n = 7) and Supersano (n = 4) (Calcagnile et al., 2011; Quarta et al., 2011), and from the most recent analyses from Grotta dei Cervi in Porto Badisco (n = 33; Aprile et al., 2017) and from Grotta delle Veneri (n = 10; Calcagnile et al., 2020). Unfortunately, the origin of the samples from Grotta Zinzulusa (n=16) still remain unknown (Tykot 2017b, p 1778, table 2).

On the contrary, the attestation of the two other sources, such as Palmarola, for Galliano 42 sample, and Pantelleria, for Galliano 27 sample, in two of the Late Neolithic burials with a particular frequency of obsidian finds (respectively n = 15 and n = 8), significantly expanded its distribution area towards the east and the north, in a chronologically well-defined context.

In the most recent studies, a significant presence of obsidian from Palmarola has already been highlighted in various Neolithic villages of the Puglia Tavoliere, object of archaeological excavations, such as Monte Aquilone, Passo di Corvo and Masseria Candelaro (phase II), and the Altopiano delle Murge in Balsignano (Acquafredda et al. 2017), to which recent data from the Early Neolithic village of Ripa Tetta (n = 8 of which 4 from Palmarola; Acquafredda et al., 2018) and from surface sites also in the Tavoliere (n = 111 of which 12 from Palmarola; Brown et al., 2018) are been added. This source is documented, albeit in very limited quantities, in contexts that can be framed from an advanced phase of the Early Neolithic to an early Middle Neolithic. At Tremiti archipelago, in Prato Don Michele site, obsidian from both the Lipari and Ponza sources were just recognized, although in the past (Cornaggia Castiglioni et al., 1963; Fussi, 1965). The eastward diffusion of obsidian from Palmarola is now recently confirmed by its presence at the inland site of Baselice in Campania (Langella et al. 2003) and, albeit in a very small sample, on the Croatian

island of Sušac (2 finds out of a total of 58 analysed samples; Tykot, 2014), part of the bridge of islands which connect with Tremiti the two shores of the Adriatic (Forenbaher, 2009). As regards the southernmost part of Puglia, the data of Galliano necropolis are added to those from the settlement of Oria – Sant’Anna (BR) in which the main provenance remain Lipari (n = 36; Ingravallo, 1995; Bigazzi and Radi, 1998; Negrino and Radi, 2006), with a single find from Palmarola (De Francesco et al., 2011), even if its stratigraphic context, from the lower middle Neolithic levels, with painted and scratched ceramic, or from the upper final Neolithic levels with Diana ceramic, is not reported.

Finally, completely unexpected was the identification of obsidian from Pantelleria Island. Pantelleria obsidian, documented only in a restricted area of the central Mediterranean as western Sicily, Malta and the coast of Tunisia (Mulazzani et al., 2010; Tufano et al., 2012; Tykot et al., 2013; Freund et al., 2017; Foresta Martin et al., 2020), hadn’t yet finding in mainland Italy (Bigazzi and Radi, 2003). The data presented here by T5 of Galliano, which can be dated to the first centuries of the second half of the fifth millennium BC (on female human bone, LTL15556A:  $5628 \pm 45$  BP,  $\delta^{13}\text{C} -23.3 \pm 0.2$ , 4550-4350 cal.  $2\sigma$  BC; Radina et al., 2020: 117, table III), with the identification of the Salto la Vecchia and Balata dei Turchi sub-source, significantly expand its distribution area towards the east, in a chronologically well-defined context. A possible large-scale diffusion of Pantelleria obsidian in a wide-range exchange circuits in the Mediterranean in an advanced phase of the Neolithic has already been supposed due to the discovery of three artefacts from Pantelleria in southern France in the dolmen 2 of San Sebastien at Plan-de-la-Tour et Sainte-Maxime in a context datable to the final local pre-bell-shaped Neolithic (Thorpe et al., 1984; Binder and Courtin, 1994). A further comparison with Galliano data could be made with those of Fossacesia village (CH) (De Francesco et al., 2006: 540, fig. 3; Negrino and Radi, 2006: 557). On a total of 44 samples analysed by XRF, the main origin is confirmed to be Lipari, with five samples from Palmarola, as well as a single one from Pantelleria of which, however, only a preliminary report is available at this moment (Pessina and Radi, 2006: 438-439, note 8; De Francesco et al., 2011: table 5.3) and the related analytical data have not been published, nor has the related sub-source been identified.

## 5. Conclusions

The analyses of 52 obsidian samples find as bladelets and flakes artefacts in the Galliano necropolis (Taranto) confirm that during the Neolithic in Southern Italy the Lipari island represented the main source for the supply and the trade of raw obsidian. Significant was the discovery of two samples, one from Palmarola and the other from Pantelleria, testifying to a greater diffusion of obsidian from both source areas. The sample from Palmarola is the southernmost signalled in Puglia, confirming sea- and land-based exchange networks of Pontine obsidian covering long distances, from the Tyrrhenian coast to inland Irpinia and thence to Apulia. The sample from Pantelleria significantly expanded its distribution area towards the east and the north of the Mediterranean area, during the second half of the fifth millennium BC, respect the previous noted restricted area of Western Sicily, Malta and northern coast of Tunisia (Tufano et al., 2012).

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## Figure and table captions

Figure 1: Central-eastern Mediterranean obsidian source areas; with a star is indicate the location of the Galliano necropolis and in the inset (from Radina 2017, modified) a particular of the tomb 5.

Figure 2: Some selected obsidian artefacts of the Galliano Neolithic obsidian assemblage.

Figure 3: on the left WD-XRF spectrometer with the sample holder detail, closed at the bottom with a Mylar© film. On the right the SEM-EDS equipment used for glass and microphenocrysts analyses of the obsidian samples; in the bordered inset the SEM sample holder on which numerous obsidian artefacts are mounted with plasticine.

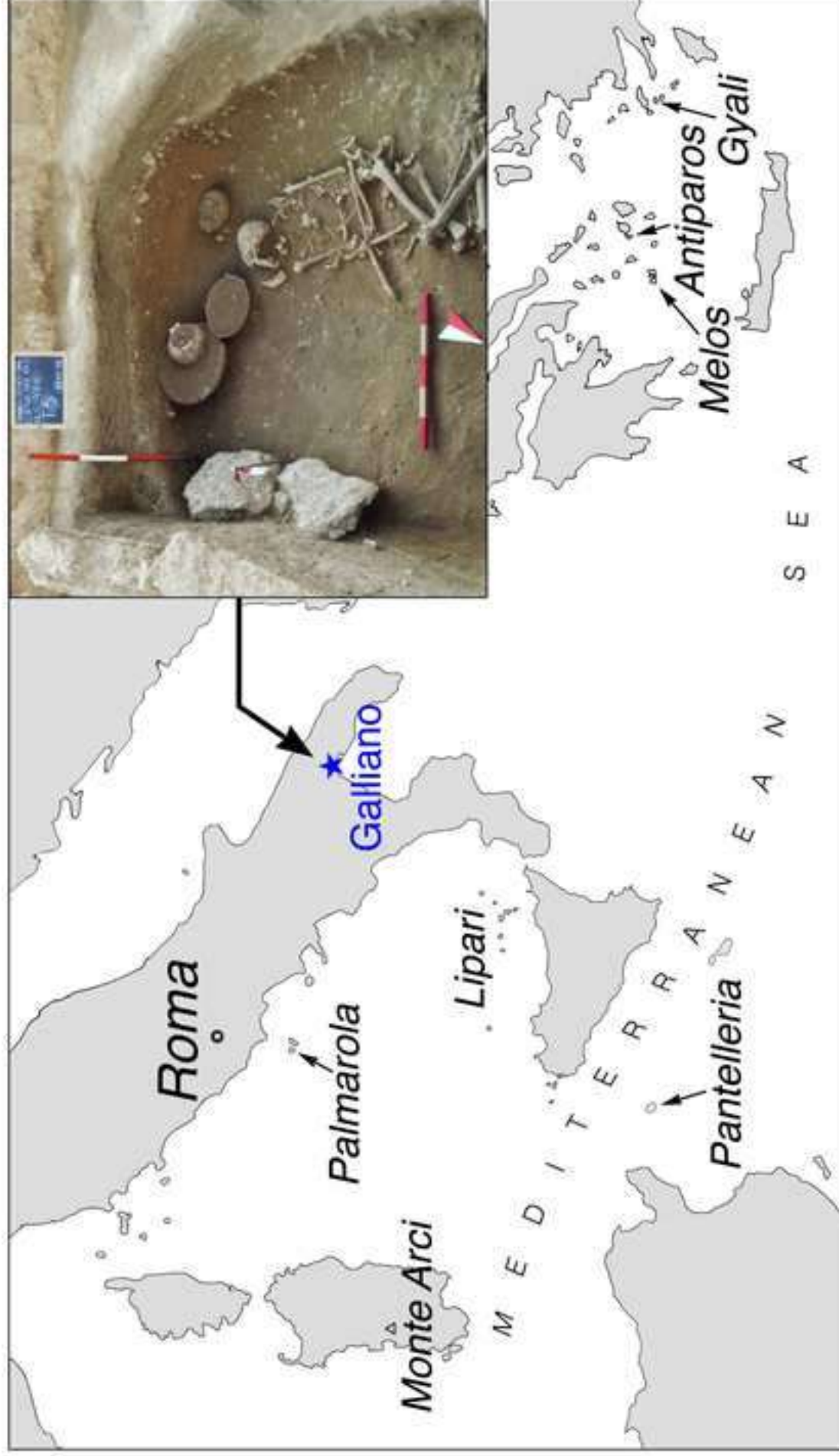
Figure 4: Plots at the top: WD-XRF data plots of obsidian from Galliano site compared with the data from other source areas in the Mediterranean (data from Acquafredda et al., 1999; Acquafredda et al., 2019; sub-sources outcrops: Monte Arci, S.C. = Perdas Urias; S.A. = Conca Cannas, Canale Perdera and Riu Solacera. Pantelleria, B.V. = Bagno di Venere, S.V. = Saltò la Vecchia and Balata dei Turchi. Melos, D. = Demenegaki; N.A.S. = Nihia, Adamas and Sarakiniko). Plot at the center: SEM-EDS microanalyses of the major obsidian source areas in the Mediterranean basin (data from Acquafredda et al., 1999, 2019); obsidian sample 27 and 42 from Galliano site (glass analyses, representing the mean value of five microanalyses) are plotted as black circles. Plots at the bottom: at the left compositions of pyroxenes according to the Morimoto et al. (1989) nomenclature for the major obsidian source areas in the Mediterranean (Acquafredda and Paglionico, 2004; Acquafredda et al., 2019); at the right, composition of the feldspars from the major obsidian source areas in the Mediterranean (Acquafredda and Paglionico, 2004; Acquafredda et al., 2019) plotted in the Ab-An-Or diagram. Pyroxenes and feldspar of Galliano 27 and 42 samples are plotted as solid symbols. En: enstatite; Fs: ferrosilite; Wo: wollastonite; Aug: augite; Ab: albite; An: anorthite; Or: orthoclase; Sa: sanidine; Anrt: anorthoclase; Olig: oligoclase; And: andesine.

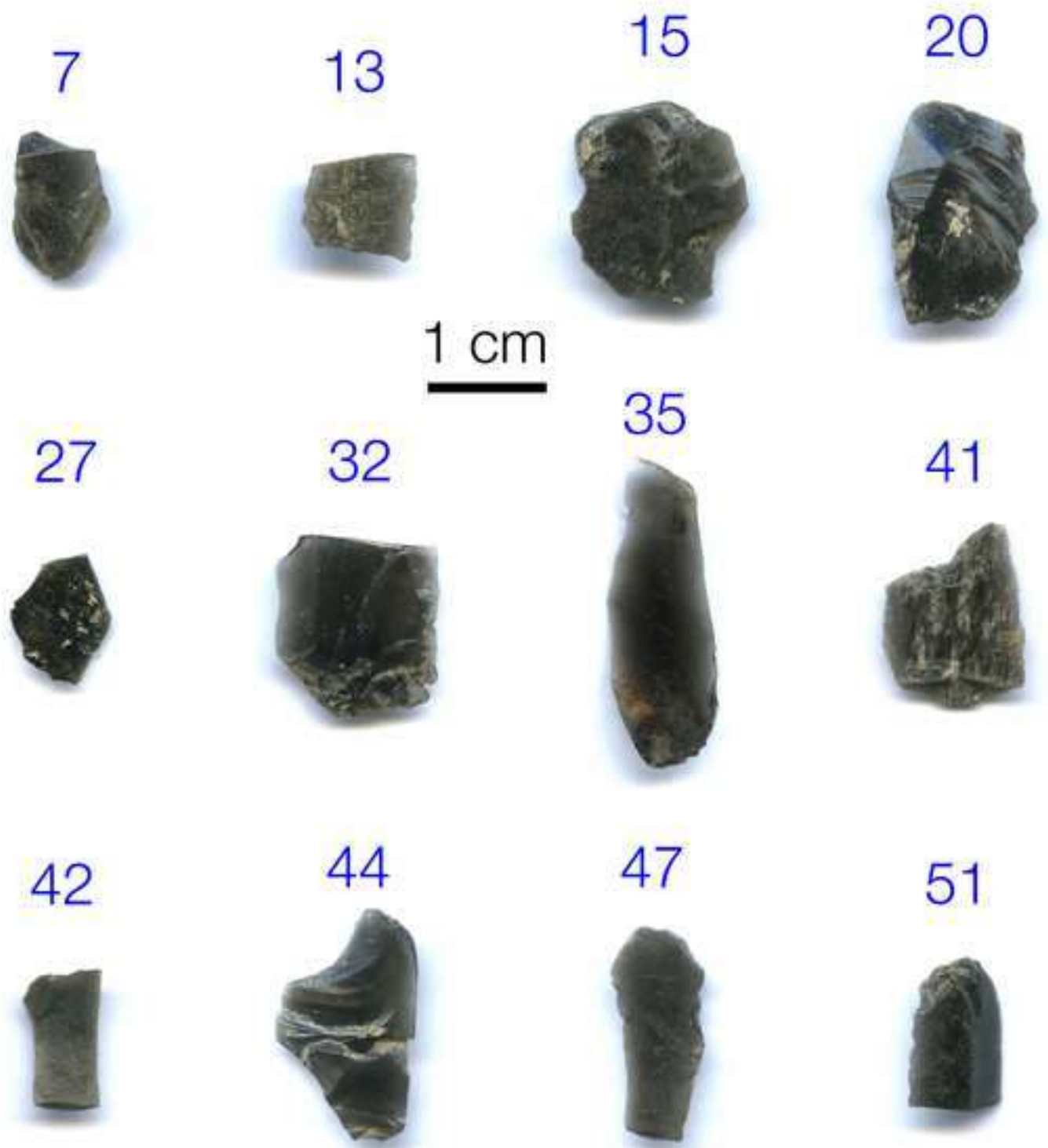
Figure 5: SEM BSE image of the Galliano 27 and 42 obsidians. In the Galliano 27 sample (top of the figure) are clearly recognizable two sanidine crystals (the bigger also Carlsbad-twinned). The Galliano 42 sample (bottom of the figure) show a strongly corroded surface on which the alteration highlighted the presence of microphenocrysts: very little augitic pyroxenes (Aug), idiomorphic anorthoclase feldspars (Anrt), showing faces according to prismatic elongation (right crystal) and or faces perpendicular to the prismatic elongation (left crystal), cubic magnetite crystal (Mag) and secondary minerals that grew during burial period in the fractures and cavities.

Table 1: X-ray analyses of some trace elements of selected samples of Galliano obsidians. The WD-XRF X-rays intensity values, expressed as counts per second, are background and interference free; some characterizing ratios are also reported.

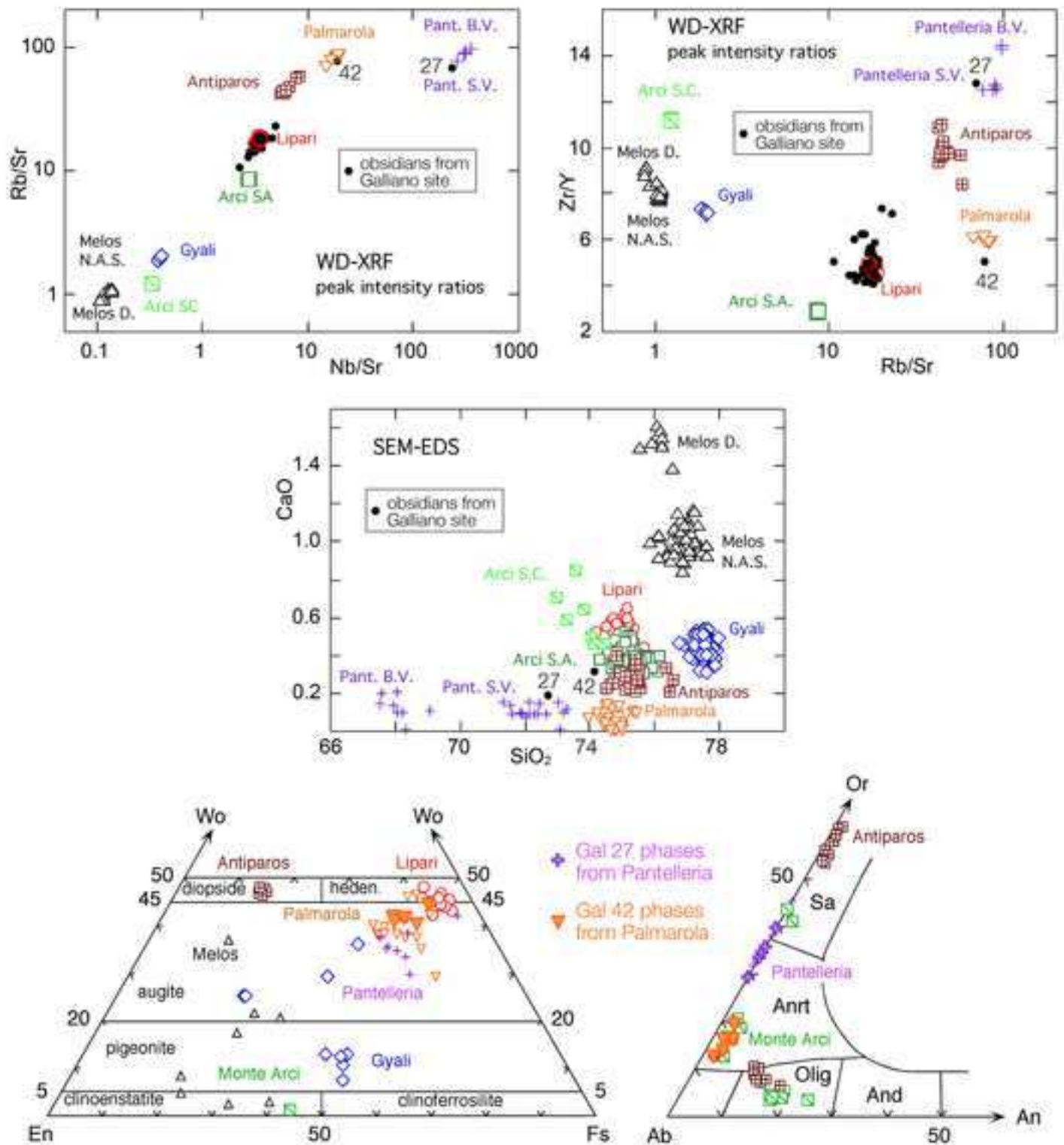
Table 2: SEM-EDS microanalyses of the glass of the Galliano 27 and 42 obsidians; each value reported in the table represents the mean of five different determinations.

Table 3: Pyroxene (formula on the basis of 6 oxygens) and feldspar (formula on the basis of 8 oxygens) selected microanalyses. En: enstatite; Fs: ferrosilite; Wo: wollastonite; Aug: augite; Ab: albite; An: anorthite; Or: orthoclase; Sa: sanidine; Anrt: anorthoclase; Olig: oligoclase; And: andesine.









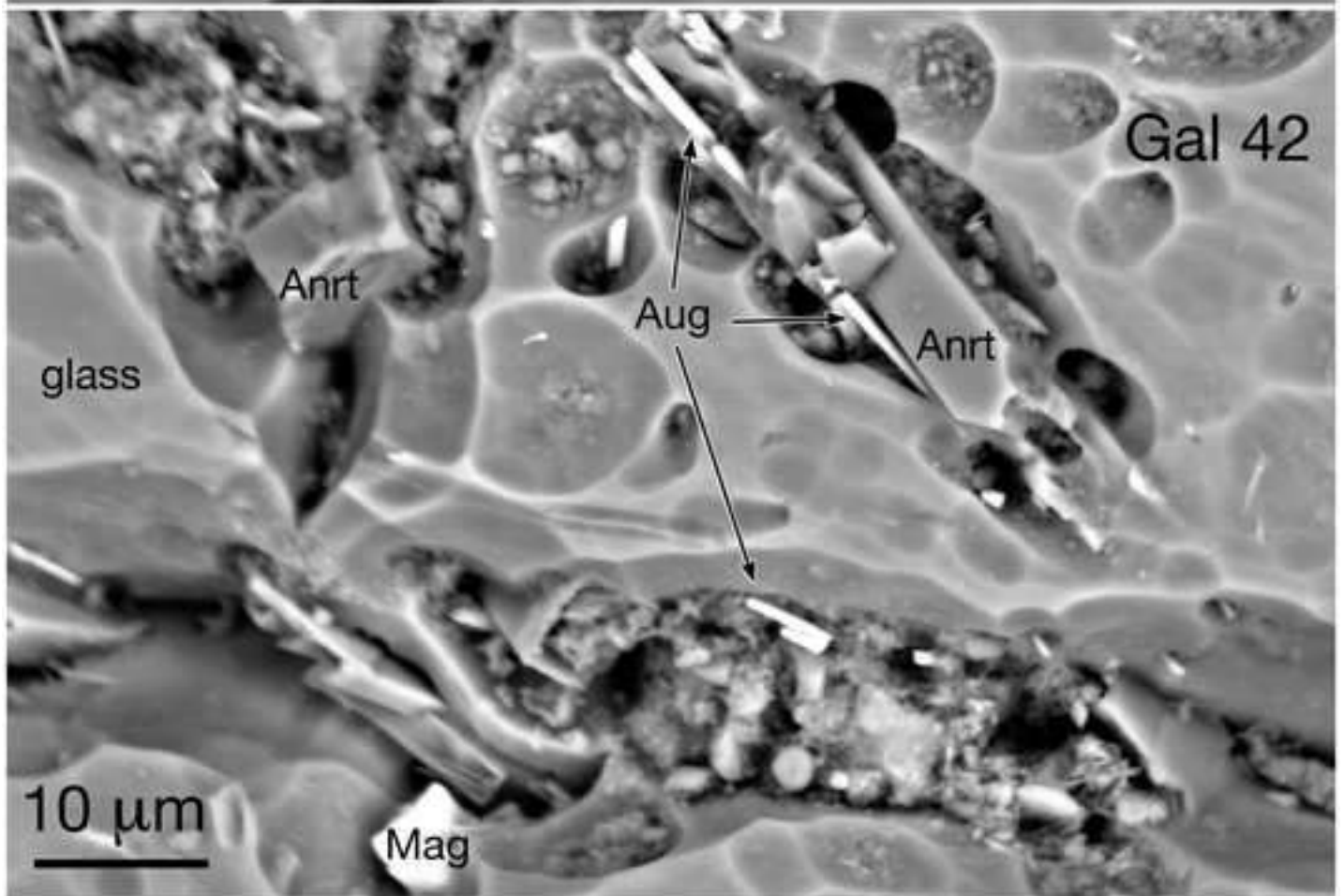
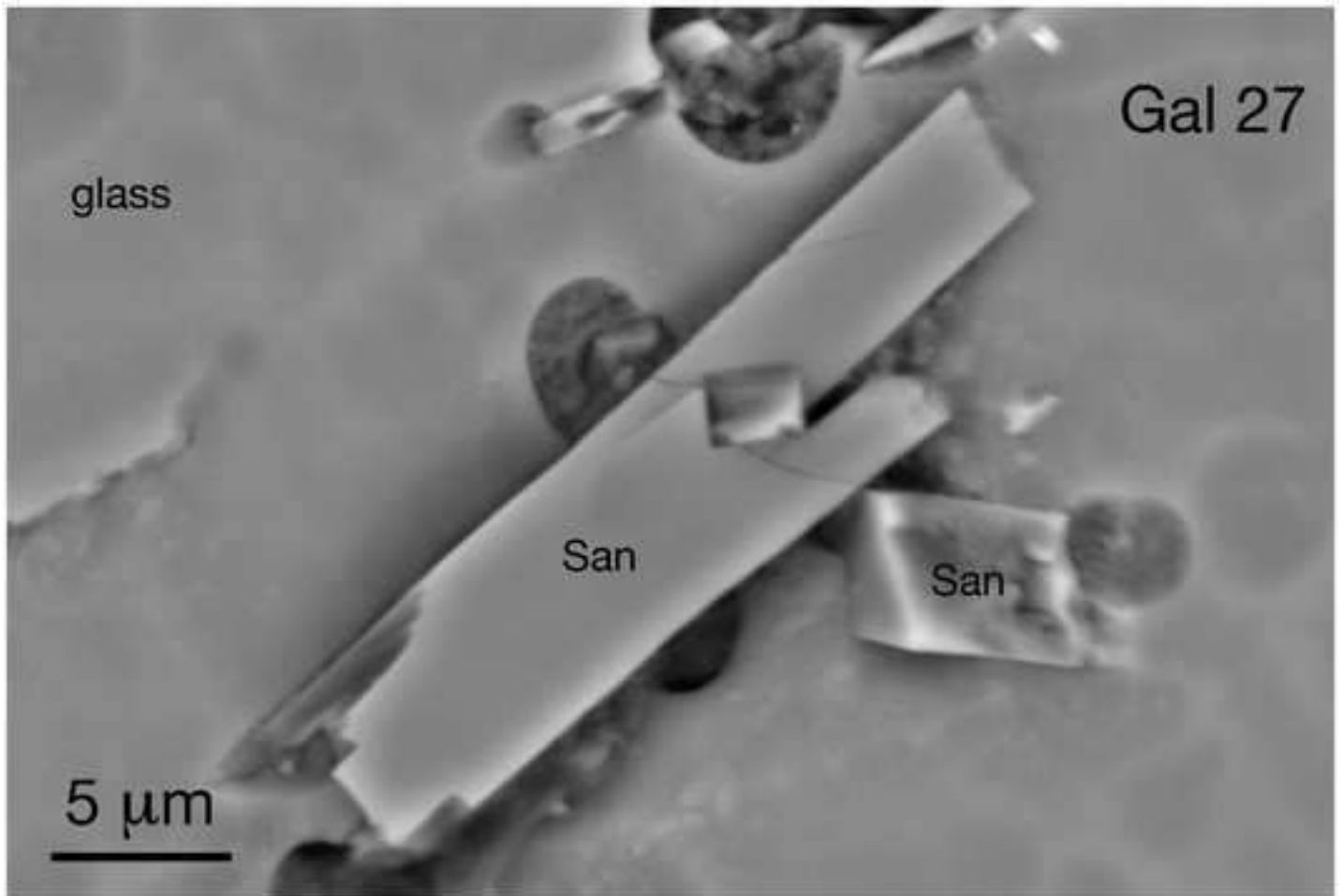


Table 1: X-ray analyses of some trace elements of selected samples of Galliano obsidians. The WD-EDS X-rays intensity values, expressed as counts per second, are background and interference free; some characterizing ratio are also reported.

sample	Rb	Sr	Y	Zr	Nb	Nb/Sr	Rb/Sr	Zr/Y
Gal 01	1217.1	61.5	171.5	1260.0	236.5	3.84	19.8	7.35
Gal 04	5835.8	356.6	1212.8	5704.2	1266.8	3.55	16.4	4.70
Gal 09	3220.5	185.0	615.0	3162.4	665.9	3.60	17.4	5.14
Gal 12	1782.0	104.3	300.6	1696.7	329.8	3.16	17.1	5.64
Gal 17	1423.6	62.2	193.2	1375.3	308.3	4.96	22.9	7.12
Gal 18	4403.1	269.0	881.6	4246.7	918.2	3.41	16.4	4.82
Gal 22	7018.5	416.3	1474.7	6483.4	1412.5	3.39	16.9	4.40
Gal 27	1670.3	24.4	1982.7	25390.3	5661.9	231.99	68.4	12.81
Gal 28	2273.8	126.8	405.5	2170.1	432.4	3.41	17.9	5.35
Gal 36	7195.3	498.3	1624.0	7063.5	1557.1	3.12	14.4	4.35
Gal 41	4722.5	444.2	972.4	4929.4	1021.0	2.30	10.6	5.07
Gal 42	4929.7	64.2	942.9	4785.4	1205.7	18.79	76.8	5.08
Gal 45	3582.3	186.8	700.1	3033.9	637.8	3.41	19.2	4.33
Gal 46	1279.4	92.4	213.9	1293.9	261.1	2.82	13.8	6.05
Gal 49	1750.5	95.2	306.6	1798.5	371.0	3.90	18.4	5.87

Table 2: SEM-EDS microanalyses of the glass of the Galliano 27 and 42 obsidians; each value reported in the table represents the mean of five different determinations.

sample	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	FeO	MnO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O
Gal 27	72.72	0.20	7.59	7.61	0.34	0.19	6.97	4.38
Gal 42	74.14	0.00	13.49	1.33	0.00	0.32	5.82	4.91

**Table 3: Pyroxene (formula on the basis of 6 oxygens) and feldspar (formula on the basis of 8 oxygens) selected microanalyses. En: enstatite; Fs: ferrosillite; Wo: wollastonite; Aug: augite; Ab: albite; An: anorthite; Or: orthoclase; Sa: sanidine; Anrt: anorthoclase; Olig: oligoclase; And: andesine.**

Sample mineral	37 Gal42 Aug	38 Gal42 Aug	53 Gal42 Aug	39 Gal42 Anrt	40 Gal42 Anrt	44 Gal42 Anrt	53 Gal27 San	54 Gal27 Anrt	59 Gal27 Anrt
SiO <sub>2</sub>	50.25	49.46	50.37	67.46	67.66	68.54	69.39	69.65	68.56
TiO <sub>2</sub>	0.38	0.21	0.28	18.69	18.33	18.71	16.80	17.37	17.33
Al <sub>2</sub> O <sub>3</sub>	3.22	3.80	4.40	0.53	0.46	0.77	0.00	0.00	0.00
FeO	23.58	23.39	18.56	10.66	10.06	9.36	7.01	7.32	8.49
MnO	2.80	2.32	1.47	2.38	3.10	2.95	7.01	6.20	5.35
MgO	3.45	2.18	3.87	99.41	99.61	100.33	100.21	100.54	99.73
CaO	15.67	17.38	13.86						
Na <sub>2</sub> O	1.10	1.33	2.71	2.99	3.01	3.01	3.09	3.08	3.06
Total	100.45	100.18	100.54	0.98	0.96	0.97	0.77	0.88	0.91
Si	1.99	1.97	2.06	0.03	0.02	0.04	0.00	0.00	0.00
Ti	0.01	0.01	0.01	0.92	0.87	0.80	0.61	0.63	0.73
Al	0.15	0.18	0.24	0.13	0.18	0.17	0.40	0.35	0.30
Fe	0.78	0.78	0.59	5.05	5.03	4.98	4.97	4.96	5.01
Mn	0.09	0.08	0.05						
Mg	0.20	0.13	0.22	85.2	81.5	79.8	60.3	64.2	70.7
Ca	0.66	0.74	0.56	2.3	2.0	3.6	0.0	0.0	0.0
Na	0.08	0.10	0.20	12.5	16.5	16.6	39.7	35.8	29.3
Σ cat.	3.97	3.99	3.92						
En	12.3	7.9	16.0						
Fs	47.3	47.3	42.9						
Wo	40.3	44.8	41.1						

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