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*Original Citation:*

*Availability:*

This version is available <http://hdl.handle.net/2318/1883902> since 2025-02-08T20:46:12Z

*Published version:*

DOI:10.1016/j.jasrep.2022.103763

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# RING-EYE BLUE BEADS IN IRON AGE CENTRAL ITALY – PRELIMINARY DISCUSSION OF TECHNOLOGY AND POSSIBLE TRADE CONNECTIONS

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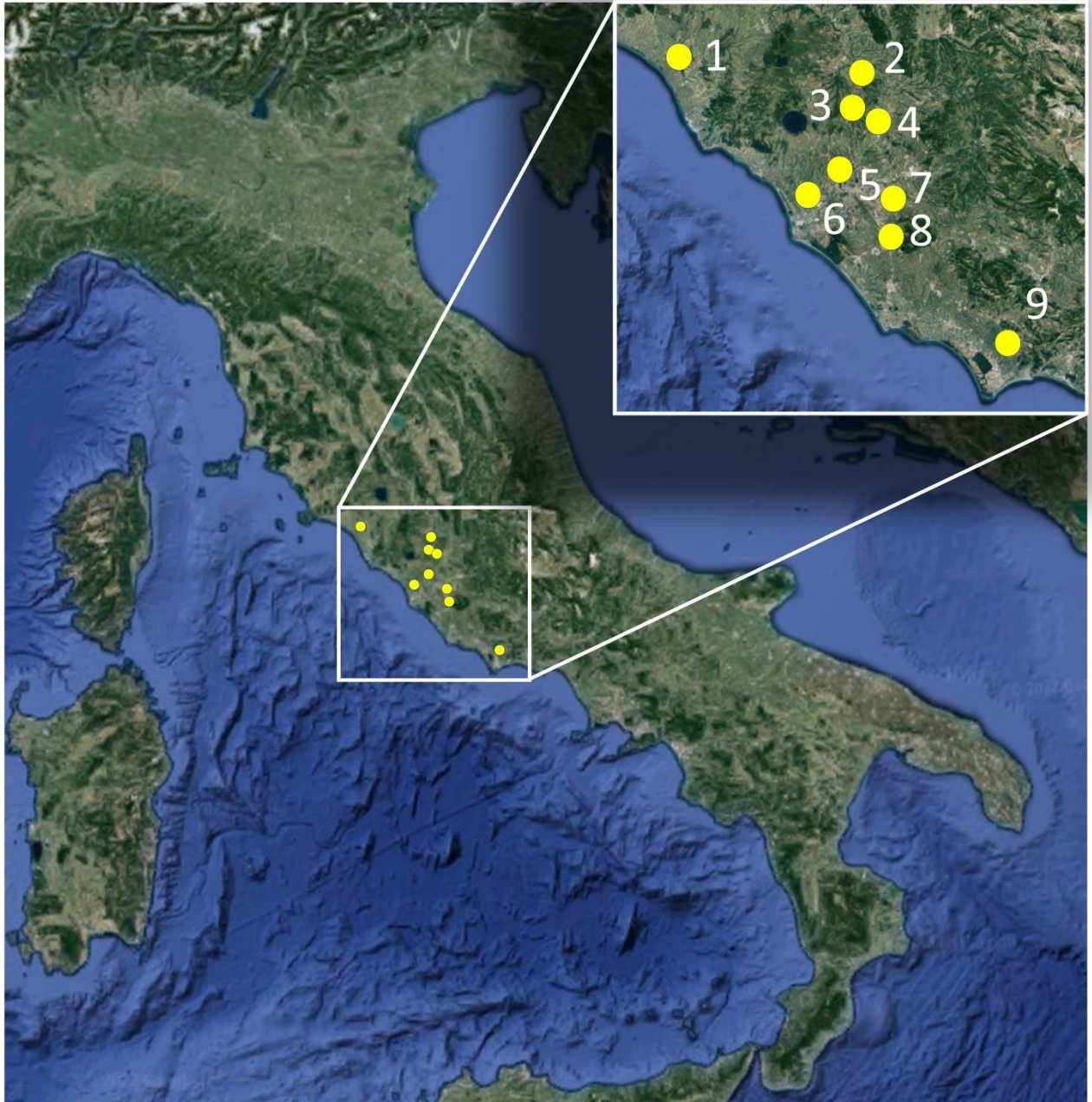
**ABSTRACT.** The Iron Age was a remarkable period in glass technology development and its spread across the Mediterranean. Communities that populated what is nowadays Central Italy underwent profound changes during this period forming more complex societies, developing proto-urban and urban centres, and incorporating into a wide trade network of the Mediterranean Sea and beyond. Glass objects in that small region are frequently found in burial sites dated to the first half of the first millennium BCE, with small blue beads with simple ring eyes being among the most abundant types. Fifty-six objects of this type (both whole beads and fragments) were studied with a non-invasive approach by means of Optical Microscopy, Fibre Optics Reflectance Spectroscopy, and portable X-Ray Fluorescence spectroscopy. The analyses were conducted *in situ* at the Museo Nazionale Etrusco di Villa Giulia and at the Museo delle Civiltà (both in Rome, Italy). Five samples from the main set were also analysed with a Scanning Electron Microscope coupled to an Energy Dispersive Spectrometer. The data gave preliminary information on the raw materials used to prepare the glass, the manufacturing techniques, and offered some hints to (tentatively) locate the region of provenance. In particular, the analyses established that the beads are soda-lime-silica glass and the source of cobalt, used as the blue colorant, could be an ore from Egypt. Within this general frame, a smaller group showed a different compositional pattern. These preliminary results contribute new knowledge for tracing exchange routes within the Mediterranean during the Iron Age.

**KEYWORDS:** glass, Iron Age, beads, FORS, p-XRF.

## 1. INTRODUCTION.

This study is the first systematic investigation - performed mainly through a non-invasive approach - of ring-eye blue beads, which are among the earliest glass bead types from the Early Iron Age (EIA) contexts in the Italian peninsula. Because such beads are frequently found, the archaeological inferences can take great advantage from the determination of the chemical composition of the glass. Compositional homogeneity - or heterogeneity - within a set of beads can, in fact, give clues to define at least the number of glass-making sites

47 that were covering the demand of such items. Moreover, in some instances, compositional features may give  
48 useful information to locate the production sites (Shortland et al. 2007; Conte et al. 2016; Oikonomou et al.  
49 2018; Costa et al. 2021). The beads considered in this work were found in nine archaeological sites in the  
50 present-day Lazio region of Italy. These sites belong to two different historic regions, commonly named  
51 Southern Etruria (Capena, Cerveteri, Falerii, Narce, Veio and Vulci) and Latium (Marino, Osteria dell'Osa  
52 and Sermoneta) in the IA archaeological scholarship. The position of the sites is reported in Figure 1, which  
53 shows a map of Italian peninsula.  
54



55  
56 *Figure 1. Location of the archaeological sites on the map of Italian peninsula. 1 – Vulci; 2 – Falerii; 3 – Narce; 4 –*  
57 *Capena; 5 – Veio; 6 – Cerveteri; 7 – Osteria Dell’Osa; 8 – Marino; 9 – Sermoneta.*

58  
59 With more details, the beads were found in twenty-one inhumations and three cremation burials (see  
60 Supplementary information, Table SII). Many graves also contained faience and amber beads in addition to  
61 the glass ones, testifying developed trade connections, as such materials were not available locally (D’Ercole,  
62 2017). In accordance with the burial tradition of that time, most of the graves also contained local pottery and  
63 bronze objects, although the occurrence of imported ceramics (predominantly Greek or Greek-type ware) was  
64 also frequent in this cultural milieu.

65 In the Mid-Tyrrhenian area, and especially in the region of Etruria, the 8<sup>th</sup> and 7<sup>th</sup> centuries BCE were  
66 times of profound social and economic changes. Settlements evolved to proto-urban and urban centres, crafts  
67 took new heights in development (Bietti Sestieri, 1997, Pacciarelli 2017) and long-distance trades resumed  
68 after the Bronze Age Collapse (Sherratt and Sherratt, 1993; Collis, 2003). This can be viewed as part of a wider  
69 ‘globalising’ phenomenon encompassing the entire Mediterranean area (Hodos 2020).

70 The deep blue colour of glass was achieved starting from the Bronze Age Egypt by the addition of cobalt  
71 to the glass batch. It was highly valued by ancient societies up to the restriction of its production and use  
72 (Barag, 2006; Hodgkinson, 2019; Schenkel, 2019). Glass makers knew several sources of cobalt. How and  
73 where these sources were exploited for the preparation of blue glass were points of debate among scholars for  
74 several decades (Sayre and Smith 1973; Henderson, 1985; Rehren, 2001; Tite and Shortland, 2003; Reade et  
75 al. 2005; Gratuze and Picon, 2005; Nikita and Henderson, 2006; Abe et al., 2012; Smirniou and Rehren 2013;  
76 Oikonomou et al. 2018; Hodgkinson et al., 2019; Costa et al., 2021 and references within). This extensive  
77 debate gives robust basis for the interpretation of compositional data for blue glass.

78 In this work, a non-invasive, in situ approach was adopted to highlight compositional and technological  
79 heterogeneity (if any) of ring-eye blue beads from IA Central Italy. The goal was to obtain as much information  
80 as possible directly in the museum, with a focus on technology of production (investigated through Optical  
81 Microscopy - OM), colouring agents (detected by Fibre Optics Reflectance Spectroscopy - FORS) and  
82 elemental composition (determined by portable X-Ray Fluorescence spectroscopy – p-XRF).

83 All the beads of this type (i. e. hundreds of specimens) preserved in the Museo Nazionale Etrusco di  
84 Villa Giulia and in the Museo delle Civiltà (both in Rome, Italy) were observed under the OM. Then, fifty-six  
85 beads were selected in order to represent this large assemblage. It is worth stressing that the investigated set  
86 of beads represents all the ring-eye blue beads found in a large set of the presently known IA archaeological  
87 sites in South Etruria and Latium, covering the entire timespan that yielded such beads. In order to integrate  
88 the results of the preliminary non-invasive screening, a selected subset of five beads was analysed in the  
89 laboratory with Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM-EDS).

90

## 91 2. MATERIALS AND METHODS.

92 **2.1. Glass beads.** The graves in South Etruria and Latium in which ring-eye blue beads were found  
93 belong mostly to the Early Iron Age II period (EIA II), therefore forty-five beads (i.e. the majority of the fifty-  
94 six beads considered in this work) originate from tombs of this period. Most scholars conventionally date EIA  
95 II to 800-720 BCE (see Bartoloni and Delpino 2005 for an overview on periodisation and absolute dating, with  
96 related discussions on these topics). One bead of the considered sample set dates to the Early Iron Age I (EIA  
97 I: ca 950 – 800 BCE), nine beads were found in tombs of the Orientalising period (most of them from the Early  
98 Orientalising period, which is approximately dated to 720-670 BCE). Finally, one bead is dated to the Late  
99 Archaic period (530-400 BCE).

100 In the selection of the samples, attention was given also to include all colour and/or translucency  
101 variations. Typical representatives of this bead type are shown in Figure 2: they are compressed spheres of 5-  
102 9 mm in diameter (dimension perpendicular to the aperture) and 3-6 mm in height (dimension along the  
103 aperture). The diameter of the aperture for the whole assemblage is 2-3 mm. All of them are semi-translucent,  
104 blue with various degrees of saturation. Most of them are decorated with 3 or, in fewer cases, 2 ring-eyes of

105 opaque white glass. In some of the beads, the decoration was detached and lost. Moreover, some of the beads  
106 keep integrity, while others were available as fragments.



107 *Figure 2. Glass beads from Capena, Early Orientalising period, Museo delle Civiltà, Rome. Four beads from this set*  
108 *were studied with the non-invasive spectroscopic approach reported in the text. © Museo delle Civiltà, Rome.*

109  
110 Remains of white glass droplets instead of rings are visible on the blue-green body of sample PG112,  
111 suggesting that this bead may belong to a different type, known from the Late Bronze Age through the finds  
112 from Frattesina (Italy) (Bellintani and Angelini 2020).

113  
114 **2.2. Optical Microscopy (OM).** A Dino-Lite AM4815ZT – Edge digital microscope was used for  
115 visual examination and documentation of the beads. All the ring-eye blue beads preserved in the two museums  
116 were observed and some images at 20× and 100× magnification were acquired. This wide set of similar beads  
117 explored under the OM highlighted some features relevant for the interpretation of the bead-forming process.

118 **2.3. Fibre Optics Reflection Spectroscopy (FORS).** An Ocean Insight HL-2000-HP-FHSA 20W  
119 Tungsten halogen light source was used to transmit light through a 2m-long reflection/backscatter fibre optics  
120 probe with 400 µm core size. The angle of the incident light beam was set around 45°, with adjustments to  
121 better capture the characteristic features of the diffuse light spectrum. Diffuse light was then transmitted to the  
122 Ocean Insight QEPro CCD detector with HC1 grating. Operating range was 248 – 1038 nm with optical  
123 resolution 6.78 nm FWHM. The instrument was calibrated using a high reflectivity Spectralon reference. The  
124 integration time was from 0.019 to 0.029 s, no less than 40 scans were averaged for a single spectrum  
125 acquisition and several spectra were acquired in different parts of each bead, including the white decorations.  
126 For these latter, the spectra did not show any characteristic band, except for those of the blue glass. Therefore,  
127 spectra of white decorations were not taken into consideration in the discussion of the data. All the spectra  
128 were normalized to 100%.

129 **2.4. Portable X-Ray Fluorescence spectroscopy (p-XRF).** Three p-XRF units were used for the  
130 analyses. One was an ELIO spectrometer (XGLab S.R.L. Milan, Italy), equipped with Rh anode source with a  
131 focusing beam spot size of 1.2 mm and a 25 mm<sup>2</sup> Silicon Drift Detector (SDD). A second unit was an Unisantis  
132 XMF-104 spectrometer (Geneva, Switzerland) equipped with Mo anode source, polycapillary optics focusing  
133 the beam on a spot of 80 µm and a Si-PIN detector of 7 mm<sup>2</sup> area. The third unit, called Frankie, was developed  
134 in house by the Italian National Institute of Nuclear Physics-LNF (INFN-LNF Frascati, Italy) and has a W  
135 anode source, polycapillary optics focusing the beam onto a spot of 300 µm and a SDD detector with an active  
136 area of 20 mm<sup>2</sup>. Acquisition settings of these instruments were as follows (integration time, tube current,  
137 voltage):

- 138
- 139 • ELIO: 90 s; 40 µA; 40 kV.
  - 140 • Unisantis: 150 s; 300 µA; 50 kV.
  - 141 • Frankie: 200 s; 80 µA; 40 kV.

142 For each bead, both the blue glass and the white decoration were analysed in three different parts of  
the same bead. Nevertheless, as signals from the blue parts are likely to be included in the spectra obtained

143 from the white decorations due to the low thickness and small surface of these parts, elemental data for the  
144 white glass can include, at least partially, the composition of the blue glass.

145 Spectra were elaborated by PymCA software (Solé et al. 2007) and elemental concentrations were  
146 calculated based on the algorithm of the Fundamental Parameters method calibrated using Corning Glass  
147 references (A, B, and D) and several archaeological glasses with known composition. The complete description  
148 of the procedures, as well as the assessment of the accuracy of the quantitative data, is given in Yatsuk et al.  
149 2022. In this work, limits of quantification were cautiously risen for some elements after critical evaluation of  
150 the obtained spectra and are indicated in the Table 1.

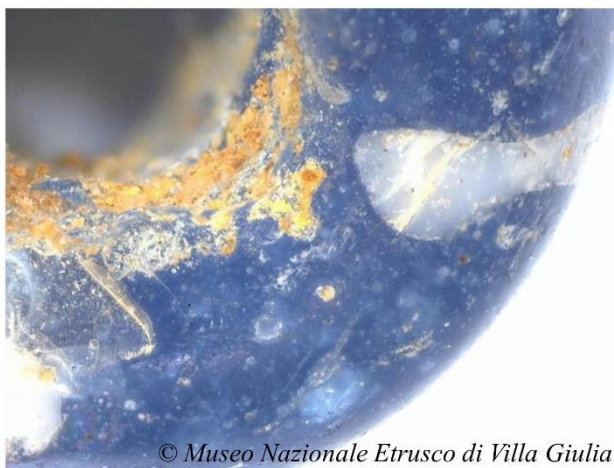
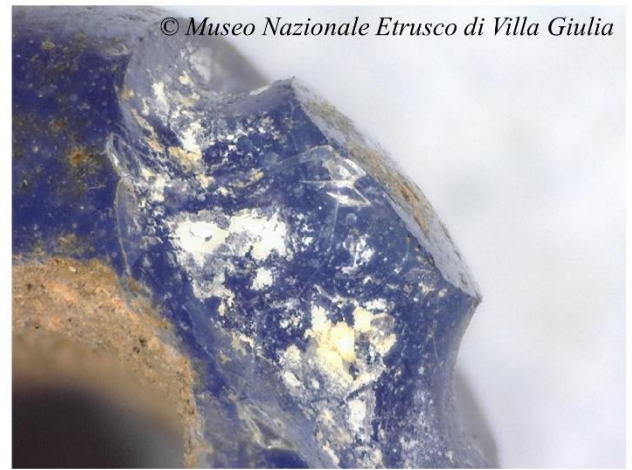
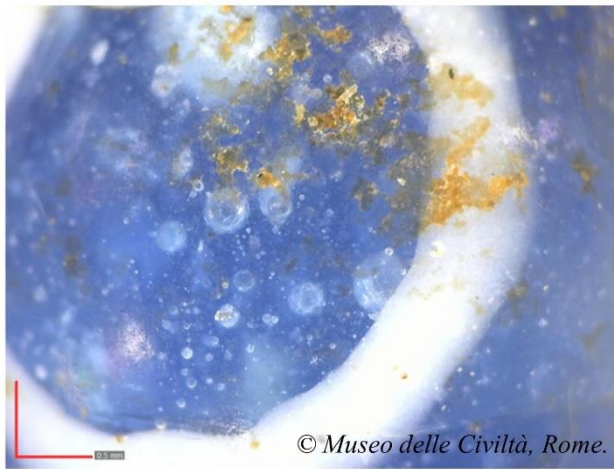
### 151 **2.5. Scanning Electron Microscopy coupled with Energy Dispersive Spectrometry (SEM-EDS).**

152 Five samples were analysed using this technique. Among them, three samples (namely, PG110\_1, PG110\_2  
153 and PG139) were embedded in epoxy resin and prepared as polished cross sections. Two samples (namely  
154 PG156 and VG109) were analysed without any preparation at low vacuum conditions – 50 Pa (VP-SEM-EDS).  
155 The microscope was a JEOL JSM-IT300LV coupled to an EDS with SDD detector (Oxford Instruments).  
156 Operating conditions were as follows: acceleration voltage: 15 kV; acquisition time: 40 s; working distance:  
157 10 mm. The compositional data were obtained as mean values of the compositions collected on five squares  
158 of about 10  $\mu\text{m}^2$  at 5000 $\times$  magnification in the high vacuum mode and at variable magnifications in the variable  
159 pressure mode.

160

## 161 **3. RESULTS.**

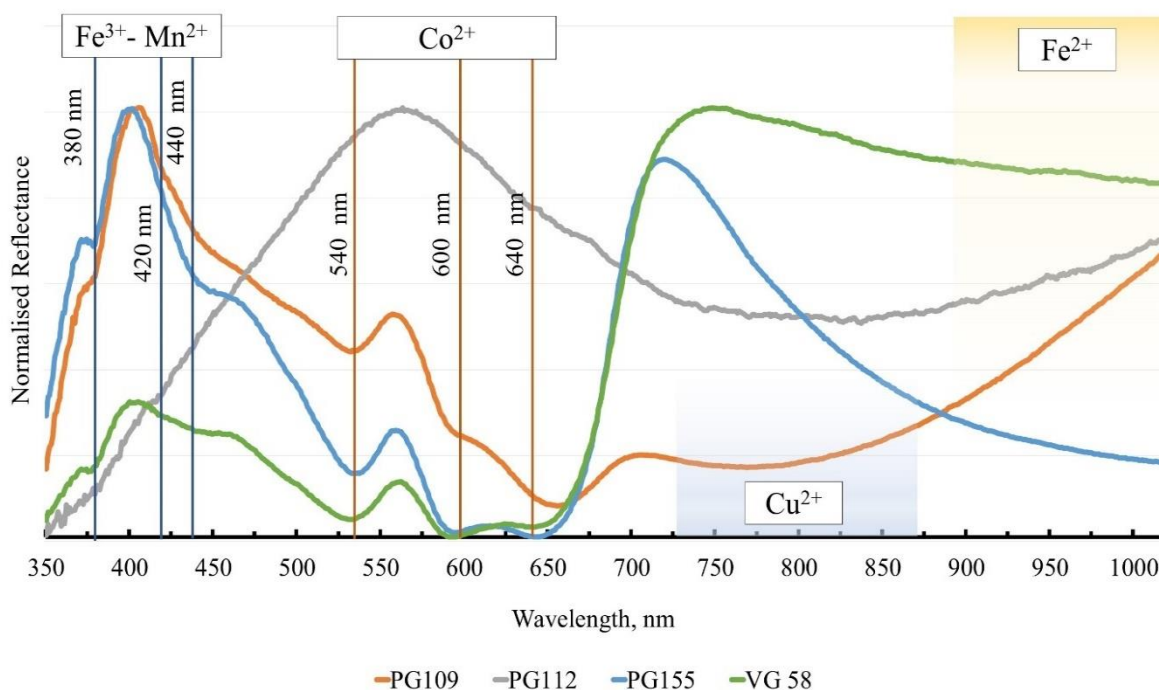
162 **3.1. OM.** The visual examination through OM gave some hints on the bead-making processes. OM  
163 images (Figure 3) show that the white decorations were applied as soft threads of hot glass inlaid in the  
164 surface of the still hot blue bead, producing a thin and narrow decoration (Figure 3 top left image). In many  
165 cases, the white glass is partially or completely detached from the main body leaving a groove (Figure 3, top  
166 right). The white thread forms rounds or, more frequently, irregular ovals, and sometimes the ends of the white  
167 tread do not join (Figure 3, bottom left). The eye spot is constituted by the body of the bead itself (Figure 3,  
168 top right and bottom left), thus allowing to classify the type as simple ring-eyes beads. The blue bulk of the  
169 bead was made by winding the hot glass around a mandrel. The technique is suggested by the shape of the  
170 bead and by the slight asymmetry in the direction parallel to the aperture. Also, winding tips near one of the  
171 poles, i.e. finishing points of the glass mass that was wound around the mandrel, are visible in some specimens  
172 (Figure 3, bottom right) (Koch, 2001; Bellintani, 2011; Sprague, 1985).



173  
 174 *Figure 3. Microscope images of selected beads. Top left – PG44, top right – VG55, bottom left – VG35, , bottom right –*  
 175 *VG20. The scale bar, same for all the images, is 0.5mm.*

176  
 177 **3.2. FORS.** The diffuse reflectance spectra were employed to detect the colouring chemical species in  
 178 the glass (values compared to ones in Micheletti et al., 2020, and references therein). The blue bulk of the  
 179 beads mostly produced similar spectra, with some noticeable exceptions. Representative spectra from the  
 180 whole sample set are reported in Figure 4. Most of the samples show bands similar to those reported for PG155  
 181 and VG58: in the UV-Visible region they feature the bands of  $\text{Fe}^{3+}$  at 380, 420 and 440 nm, which can be also  
 182 tentatively attributed to  $\text{Mn}^{2+}$ , whereas the pronounced set of  $\text{Co}^{2+}$  triple bands at approximately 540, 600, and  
 183 640 nm, expected for soda-lime glass (Fornacelli et al. 2018), is detected in the visible region. In the NIR  
 184 region, the trend of the spectra may vary within this large group of samples, due to the variable presence of  
 185  $\text{Fe}^{2+}$ , which mirrors the variability of the redox conditions in the furnace for different glass batches. Yet,  
 186 reducing environment would be preferable to produce Co-blue glass (Arletti et al. 2013; Hunault et al. 2016).

187 For a few samples (namely PG109, 110\_1, 111, 112, 138, 151, and VG72) the wide band centred at  
 188 approximately 775 nm revealing  $\text{Cu}^{2+}$  is present, suggesting that this small group is Co-Cu coloured. On the  
 189 other hand, PG112 does not show the  $\text{Co}^{2+}$  bands, and  $\text{Cu}^{2+}$  plays therefore the main role in producing the  
 190 colour of this bead. The different colouring technique highlighted through FORS can be expected for this bead,  
 191 as a different type was already suggested for this bead by the slightly different decoration.



192  
 193 *Figure 4. Representative FORS spectra for the blue part of the beads, with indicated the characteristic bands ( $\text{Fe}^{3+}$ - $\text{Mn}^{2+}$   
 194 bands – blue vertical lines;  $\text{Co}^{2+}$  bands – brown vertical lines,  $\text{Cu}^{2+}$  - blue area,  $\text{Fe}^{2+}$  - yellow area).*

195  
 196 **3.3. p-XRF.** The results of the p-XRF analyses are reported in Table 1. Concentrations for K, Ca, Ti,  
 197 Mn, Fe, Co, Ni, Cu, Zn, Sr, and Sb were measured for all the samples. For some spots on a same bead, also  
 198 Cr, Rb, Zr, Sn, and Pb were detected. The Limits of Quantification (LOQ) for these elements were established  
 199 as follows (the highest among three instruments): Cr – 273ppm, Rb – 128ppm, Zr – 162ppm, Sn – 1417ppm,  
 200 Pb – 510ppm. As the concentrations of these elements were close to the LOQs of the method, they were not  
 201 considered in the discussion of the results. For the majority of the samples,  $\text{K}_2\text{O}$  concentration below 1.2%  
 202 suggests the use of an evaporitic source of flux (Henderson 1985), though the use of soda-rich plant ash can  
 203 be suggested for VG21 and VG113 (Rehren, 2001).

204  
 205  
 206  
 207 *Table 1. Compositional data obtained with p-XRF (blue bases) with their respective standard deviation values. All values  
 208 are represented as oxide %. Character “<” is followed by the Limit Of Quantification, which is specific for each element  
 209 and p-XRF unit.*

Sample name	$\text{K}_2\text{O}$	$\text{CaO}$	$\text{TiO}_2$	$\text{MnO}$	$\text{Fe}_2\text{O}_3$	$\text{CoO}$	$\text{NiO}$	$\text{CuO}$	$\text{ZnO}$	$\text{SrO}$	$\text{Sb}_2\text{O}_5$
PG44	<1.2	4.2	0.131	0.27	1.08	0.08	0.084	<0.4	0.047	0.032	0.16
st. dev.	-	0.6	0.002	0.02	0.02	0.02	0.007	-	0.007	0.002	0.02
PG45	<1.2	4.52	0.139	0.29	1.12	0.085	0.08	<0.4	0.053	0.033	0.18
st. dev.	-	0.09	0.009	0.03	0.01	0.006	0.02	-	0.001	0.003	0.01
PG46	<1.2	4.16	<0.12	0.31	0.68	0.079	0.063	<0.4	0.058	0.034	0.28
st. dev.	-	0.02	-	0.01	0.02	0.008	0.008	-	0.001	0.001	0.03



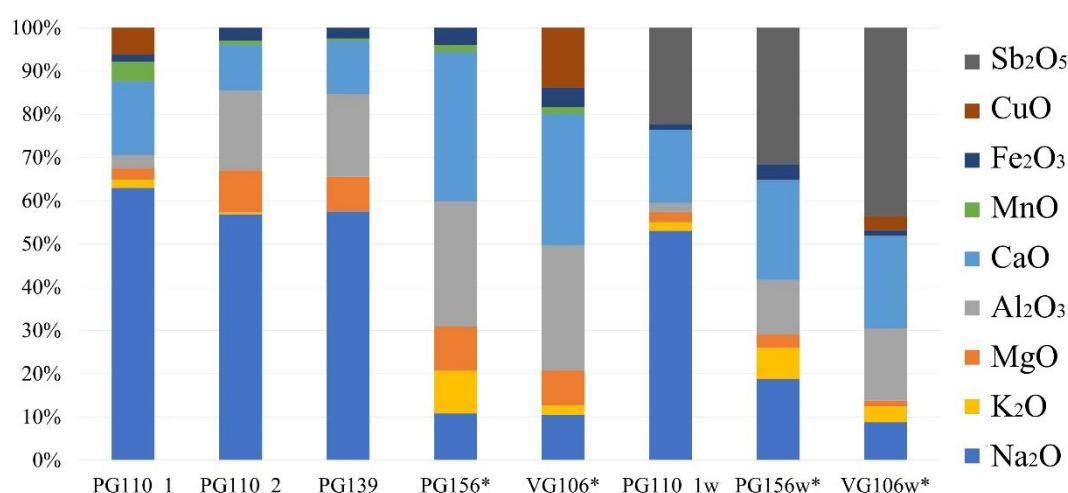
<b>PG47</b>	<1.2	5.7	0.13	0.45	0.99	0.111	0.079	<0.4	0.076	0.037	0.3
<b>st. dev.</b>	-	0.6	0.04	0.03	0.03	0.002	0.001	-	0.005	0.004	0.1
<b>PG73</b>	<1.2	3.7	0.22	0.32	1.1	0.09	0.089	<0.4	0.056	0.024	<0.087
<b>st. dev.</b>	-	1.7	0.01	0.09	0.3	0.03	0.008	-	0.021	0.011	-
<b>PG77</b>	<1.2	3.6	0.12	0.099	0.77	<0.042	0.044	<0.4	0.027	0.026	0.17
<b>st. dev.</b>	-	0.2	0.01	0.004	0.07	-	0.005	-	0.003	0.007	0.04
<b>PG78</b>	<1.2	3.6	<0.12	0.26	0.96	0.083	0.077	<0.4	0.043	0.029	<0.087
<b>st. dev.</b>	-	0.3	-	0.02	0.07	0.007	0.005	-	0.004	0.003	-
<b>PG79</b>	<1.2	4.9	<0.12	0.25	0.9	0.07	0.08	<0.4	0.049	0.033	<0.087
<b>st. dev.</b>	-	0.8	-	0.07	0.2	0.02	0.02	-	0.009	0.007	-
<b>PG80</b>	<1.2	3.7	<0.12	0.19	0.8	0.08	0.056	<0.4	0.052	0.029	<0.087
<b>st. dev.</b>	-	0.6	-	0.04	0.1	0.02	0.005	-	0.008	0.001	-
<b>PG82</b>	<1.2	4.1	<0.12	0.38	1.2	0.108	0.11	<0.4	0.056	0.033	<0.087
<b>st. dev.</b>	-	0.6	-	0.05	0.1	0.007	0.02	-	0.008	0.005	-
<b>PG85</b>	<1.2	5.9	0.144	0.25	0.9	0.068	0.07	<0.4	0.037	0.032	0.160
<b>st. dev.</b>	-	0.8	0.008	0.05	0.1	0.009	0.01	-	0.005	0.007	0.002
<b>PG86</b>	<1.2	6.1	0.18	0.39	2.2	0.11	0.11	<0.4	0.083	0.032	<0.087
<b>st. dev.</b>	-	0.6	0.03	0.05	0.2	0.02	0.01	-	0.006	0.001	-
<b>PG91</b>	<1.2	4.1	<0.12	0.22	0.7	0.07	0.07	<0.4	0.07	0.034	0.31
<b>st. dev.</b>	-	1.5	-	0.06	0.2	0.02	0.02	-	0.02	0.009	0.02
<b>PG92</b>	<1.2	4.7	0.28	0.41	1.5	0.10	0.11	0.41	0.08	0.033	<0.087
<b>st. dev.</b>	-	0.7	0.05	0.04	0.2	0.02	0.02	0.09	0.01	0.004	-
<b>PG93</b>	<1.2	4.9	0.15	0.3	0.9	0.09	0.09	<0.4	0.088	0.04	0.4
<b>st. dev.</b>	-	2.6	0.04	0.1	0.4	0.04	0.03	-	0.038	0.02	0.1
<b>PG94</b>	<1.2	4.1	0.18	0.3	1.2	0.07	0.09	0.4	0.05	0.04	0.28
<b>st. dev.</b>	-	1.8	0.05	0.1	0.4	0.03	0.03	0.2	0.02	0.01	0.05
<b>PG100</b>	<1.2	4.5	0.13	0.3	1.1	0.09	0.10	<0.4	0.12	0.03	0.24
<b>st. dev.</b>	-	1.3	0.04	0.1	0.5	0.04	0.04	-	0.05	0.01	0.02
<b>PG102</b>	<1.2	7.3	<0.12	0.18	0.72	0.07	0.05	0.45	0.051	0.028	0.6
<b>st. dev.</b>	-	1.0	-	0.03	0.04	0.01	0.01	0.07	0.005	0.002	0.4
<b>PG103</b>	<1.2	4.5	<0.12	0.26	0.8	0.07	0.08	<0.4	0.05	0.032	0.21
<b>st. dev.</b>	-	1.6	-	0.06	0.2	0.02	0.02	-	0.01	0.006	0.02
<b>PG107</b>	<1.2	1.8	0.12	0.15	0.9	<0.042	0.05	0.8	0.04	<0.010	<0.087
<b>st. dev.</b>	-	0.5	0.04	0.07	0.2	-	0.03	0.6	0.01	-	-
<b>PG108</b>	<1.2	3.3	0.18	0.20	1.2	0.06	0.05	<0.4	0.03	0.024	0.176
<b>st. dev.</b>	-	1.1	0.03	0.07	0.2	0.02	0.01	-	0.01	0.005	0.030
<b>PG109</b>	<1.2	5.1	0.12	0.59	0.44	<0.042	<0.025	0.54	<0.019	0.028	0.4
<b>st. dev.</b>	-	1.4	0.06	0.07	0.06	-	-	0.02	-	0.004	0.1
<b>PG110_1</b>	<1.2	4.7	0.14	1.1	0.5	<0.042	<0.025	0.9	<0.019	0.030	0.4
<b>st. dev.</b>	-	1.1	0.04	0.3	0.1	-	-	0.2	-	0.007	0.1
<b>PG111</b>	<1.2	4.2	<0.12	0.61	0.85	<0.042	0.06	0.53	0.038	0.026	0.73
<b>st. dev.</b>	-	0.3	-	0.09	0.08	-	0.01	0.06	0.007	0.003	0.05
<b>PG112</b>	<1.2	3.6	0.123	0.9	0.52	<0.042	<0.025	0.7	<0.019	0.028	<0.087
<b>st. dev.</b>	-	0.5	0.003	0.1	0.07	-	-	0.1	-	0.004	-
<b>PG134</b>	<1.2	2.7	0.19	0.19	0.9	0.06	0.05	<0.4	0.039	0.028	<0.087
<b>st. dev.</b>	-	0.8	0.06	0.06	0.3	0.02	0.01	-	0.009	0.008	-
<b>PG138</b>	<1.2	3.9	0.21	1.5	0.4	<0.042	<0.025	0.9	<0.019	0.04	0.3
<b>st. dev.</b>	-	1.3	0.06	0.6	0.1	-	-	0.4	-	0.01	0.2
<b>PG139</b>	<1.2	4.9	<0.12	0.3	0.5	0.09	0.08	<0.4	0.07	0.03	0.27

<b>st. dev.</b>	-	3.4	-	0.2	0.4	0.06	0.05	-	0.05	0.02	0.06
<b>PG149</b>	<1.2	3.8	0.15	0.26	0.64	0.121	0.103	<0.4	0.086	0.030	0.16
<b>st. dev.</b>	-	0.3	0.04	0.03	0.03	0.006	0.006	-	0.002	0.003	0.07
<b>PG150</b>	<1.2	4.4	0.12	0.53	1.1	0.17	0.14	<0.4	0.11	0.035	0.3
<b>st. dev.</b>	-	0.6	0.02	0.05	0.2	0.02	0.02	-	0.01	0.004	0.1
<b>PG151</b>	<1.2	4.7	0.25	0.49	1.5	0.10	0.081	<0.4	0.080	0.030	<0.087
<b>st. dev.</b>	-	0.3	0.02	0.06	0.1	0.01	0.007	-	0.005	0.003	-
<b>PG152</b>	<1.2	3.2	<0.12	0.25	0.8	0.09	0.07	<0.4	0.06	0.030	<0.087
<b>st. dev.</b>	-	1.1	-	0.08	0.3	0.03	0.03	-	0.02	0.007	-
<b>PG155</b>	1.2	3.8	0.16	0.25	1.3	0.10	0.12	<0.4	0.14	0.028	<0.087
<b>st. dev.</b>	0.5	0.9	0.06	0.05	0.4	0.03	0.03	-	0.04	0.007	-
<b>PG156</b>	<1.2	5.3	0.16	0.22	0.94	0.088	0.08	<0.4	0.076	0.035	<0.087
<b>st. dev.</b>	-	0.5	0.02	0.02	0.09	0.009	0.01	-	0.008	0.002	-
<b>VG20</b>	0.8	3.8	<0.087	0.20	0.5	0.06	0.05	0.3	0.05	0.023	<0.376
<b>st. dev.</b>	0.3	1.3	-	0.06	0.2	0.02	0.02	0.4	0.01	0.004	-
<b>VG21</b>	2.4	3.6	<0.087	0.4	1.6	0.09	0.11	0.4	0.09	0.014	<0.376
<b>st. dev.</b>	0.9	0.9	-	0.1	0.3	0.03	0.03	0.2	0.02	0.001	-
<b>VG35</b>	<0.398	3.2	<0.087	0.10	1.0	0.045	0.07	<0.015	0.08	0.015	<0.376
<b>st. dev.</b>	-	0.6	-	0.03	0.3	0.008	0.01	-	0.02	0.002	-
<b>VG55</b>	<0.398	3.4	<0.087	0.13	0.52	0.06	0.06	<0.015	0.045	0.018	<0.376
<b>st. dev.</b>	-	0.5	-	0.03	0.09	0.01	0.01	-	0.006	0.001	-
<b>VG58</b>	<0.398	3.8	<0.087	0.27	0.9	0.06	0.04	0.31	0.055	0.022	<0.376
<b>st. dev.</b>	-	0.3	-	0.04	0.2	0.01	0.01	0.18	0.005	0.003	-
<b>VG60</b>	<0.398	3.2	<0.087	0.3	1.9	0.10	0.09	0.3	0.12	0.019	<0.376
<b>st. dev.</b>	-	0.1	-	0.1	0.1	0.04	0.02	0.1	0.02	0.003	-
<b>VG73</b>	0.4	4.1	0.09	0.24	1.1	0.03	0.03	0.04	0.03	0.024	0.7
<b>st. dev.</b>	0.2	0.9	0.02	0.18	0.5	0.02	0.02	0.02	0.02	0.002	0.5
<b>VG74</b>	0.59	3.5	0.13	0.24	1.4	0.07	0.06	0.017	0.10	0.023	0.27
<b>st. dev.</b>	0.09	1.5	0.06	0.04	0.1	0.02	0.02	0.002	0.03	0.004	0.06
<b>VG75</b>	0.6	2.9	0.2	0.1	1.4	0.04	0.03	0.06	0.05	0.02	0.2
<b>st. dev.</b>	0.6	3.1	0.2	0.1	1.6	0.03	0.03	0.07	0.05	0.02	0.2
<b>VG95</b>	<0.398	4.2	<0.087	0.3	0.9	0.09	0.11	<0.015	0.09	0.022	<0.376
<b>st. dev.</b>	-	1.8	-	0.1	0.3	0.03	0.04	-	0.03	0.006	-
<b>VG96</b>	<0.398	5.5	<0.087	0.5	1.7	0.080	0.16	<0.015	0.074	0.021	<0.376
<b>st. dev.</b>	-	1.8	-	0.1	0.3	0.003	0.02	-	0.007	0.001	-
<b>VG97</b>	<0.398	3.3	<0.087	0.4	1.4	0.13	0.10	0.3	0.10	0.012	<0.376
<b>st. dev.</b>	-	1.1	-	0.2	0.4	0.04	0.03	0.1	0.03	0.004	-
<b>VG100</b>	<0.398	4.9	<0.087	0.5	1.6	0.08	0.16	<0.015	0.08	0.026	<0.376
<b>st. dev.</b>	-	1.5	-	0.1	0.5	0.03	0.05	-	0.02	0.005	-
<b>VG106</b>	0.5	4.8	0.07	0.7	0.9	0.09	0.06	0.4	0.08	0.031	0.46
<b>st. dev.</b>	0.1	1.1	0.02	0.2	0.2	0.02	0.01	0.2	0.02	0.002	0.02
<b>VG107</b>	0.48	4.5	0.10	0.30	1.1	0.04	0.04	0.09	0.036	0.022	0.28
<b>st. dev.</b>	0.05	0.2	0.04	0.07	0.2	0.01	0.01	0.05	0.003	0.003	0.02
<b>VG108</b>	0.5	4.1	0.05	0.34	0.8	0.07	0.05	0.14	0.06	0.023	0.28
<b>st. dev.</b>	0.1	0.9	0.01	0.08	0.2	0.02	0.01	0.01	0.01	0.001	0.03
<b>VG109</b>	0.6	4.3	0.2	0.3	1.2	0.05	0.04	0.07	0.08	0.026	0.2
<b>st. dev.</b>	0.2	1.3	0.1	0.1	0.6	0.02	0.02	0.04	0.02	0.006	0.1
<b>VG112</b>	0.8	2.8	0.08	0.4	1.3	0.10	0.07	0.047	0.10	<0.017	0.46
<b>st. dev.</b>	0.1	0.6	0.02	0.1	0.3	0.03	0.02	0.007	0.02	-	0.03

<b>VG113</b>	1.5	7.2	0.047	0.21	0.9	0.05	0.035	0.038	0.05	0.025	0.174
<b>st. dev.</b>	0.2	1.7	0.009	0.07	0.2	0.01	0.008	0.005	0.01	0.004	0.004

211  
212 **3.4. SEM-EDS.** The results of SEM-EDS analyses for the samples mounted as cross sections  
213 (PG110\_1, PG110\_2 and PG139) and for those analysed without pre-treatment (PG156 and VG106) are  
214 reported in Figure 5. It presents the values of major (excluding silica) and minor oxides that reflect the content  
215 of network modifiers, stabilisers and some colourants. Their values are normalised to 100% in order to  
216 highlight the relative compositional differences among samples. Data for the samples prepared as cross  
217 sections support the hypothesis that a Na-rich flux, such as soda-rich evaporites, was used for the glass.  
218 Unfortunately, VP-SEM-EDS analyses on PG156 and VG106 highlighted sodium depletion of the surface,  
219 therefore data for these samples are not fully representative of the composition of the pristine glass.

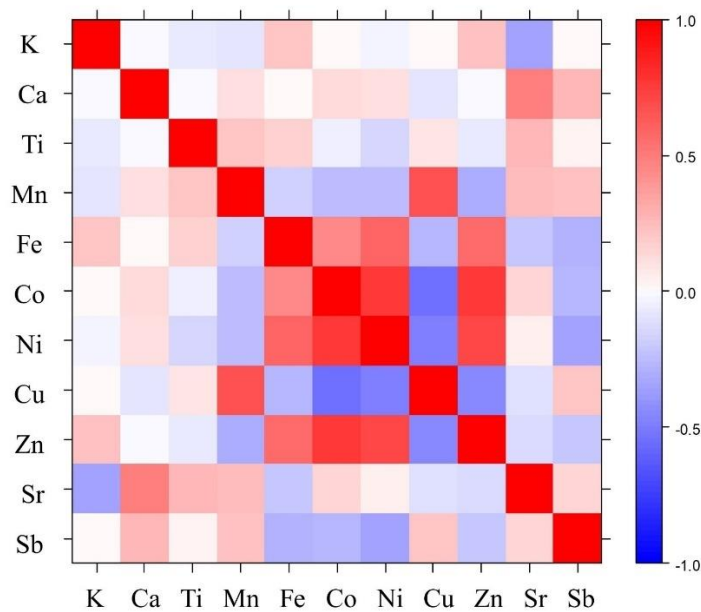
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223 *Figure 5. SEM-EDS data for some major and minor oxides normalised to 100%. \* Samples analysed without pre-*  
224 *treatment in variable pressure mode (data are therefore not fully representative of the composition of the pristine glass);*  
225 *“w” indicates white decoration.*

226  
227 **4. DISCUSSION.**

228 The presence of Co, detected by FORS as Co<sup>2+</sup> in tetrahedral coordination, which is responsible for  
229 the intense blue colour of the glass, was quantified by p-XRF in the range of 0.05 – 0.17 CoO %. This element  
230 was therefore added to achieve the blue colour of the beads in a range of concentration which is frequently  
231 reported in the literature for blue glass (Shortland and Tite 2000; Abe et al. 2012; Conte et al. 2016). The  
232 correlation matrix calculated for p-XRF data (Figure 6) revealed a strong positive correlation (r-values in the  
233 range from 0.71 to 0.77) within Co, Ni, and Zn, suggesting that these elements entered the batch through the  
234 same raw material. As for Fe<sub>2</sub>O<sub>3</sub>, the amount is consistently within the range of 0.5-1.5 %, which is enough to  
235 produce colour variations. Iron is weakly correlated with Co, Zn and Ni, therefore it is reasonable to assume  
236 that it could have entered the batch, at least partially, as an impurity of the cobalt-containing ore, although  
237 some contribution from the silica source is also expected.



238 *Figure 6. Correlation matrix of the data from the Table 1.*

239

240

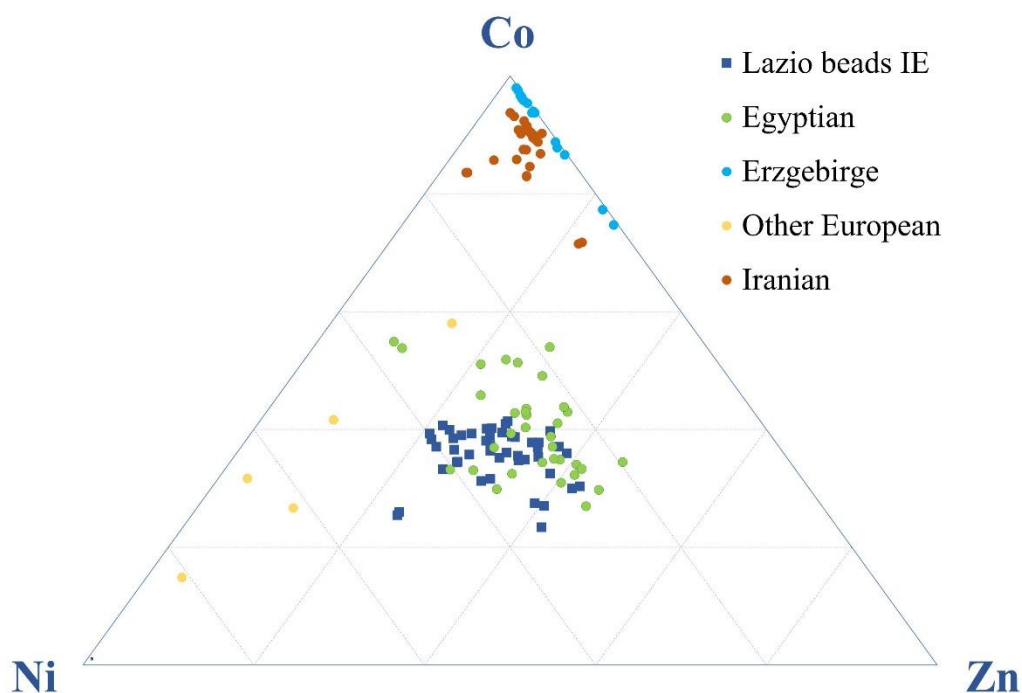
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The chemical evidence linking Co, Zn and Ni can lead to further information if we compare the compositional data obtained here with those from the literature (Figure 7). Chemical composition is in fact available for blue glass from different contexts (mostly Iron Age Mediterranean), made by using cobalt ores from several sources, namely Iran, the Erzgebirge/Krušnohoří mining region, an unlocated (probably European) source and Egypt.



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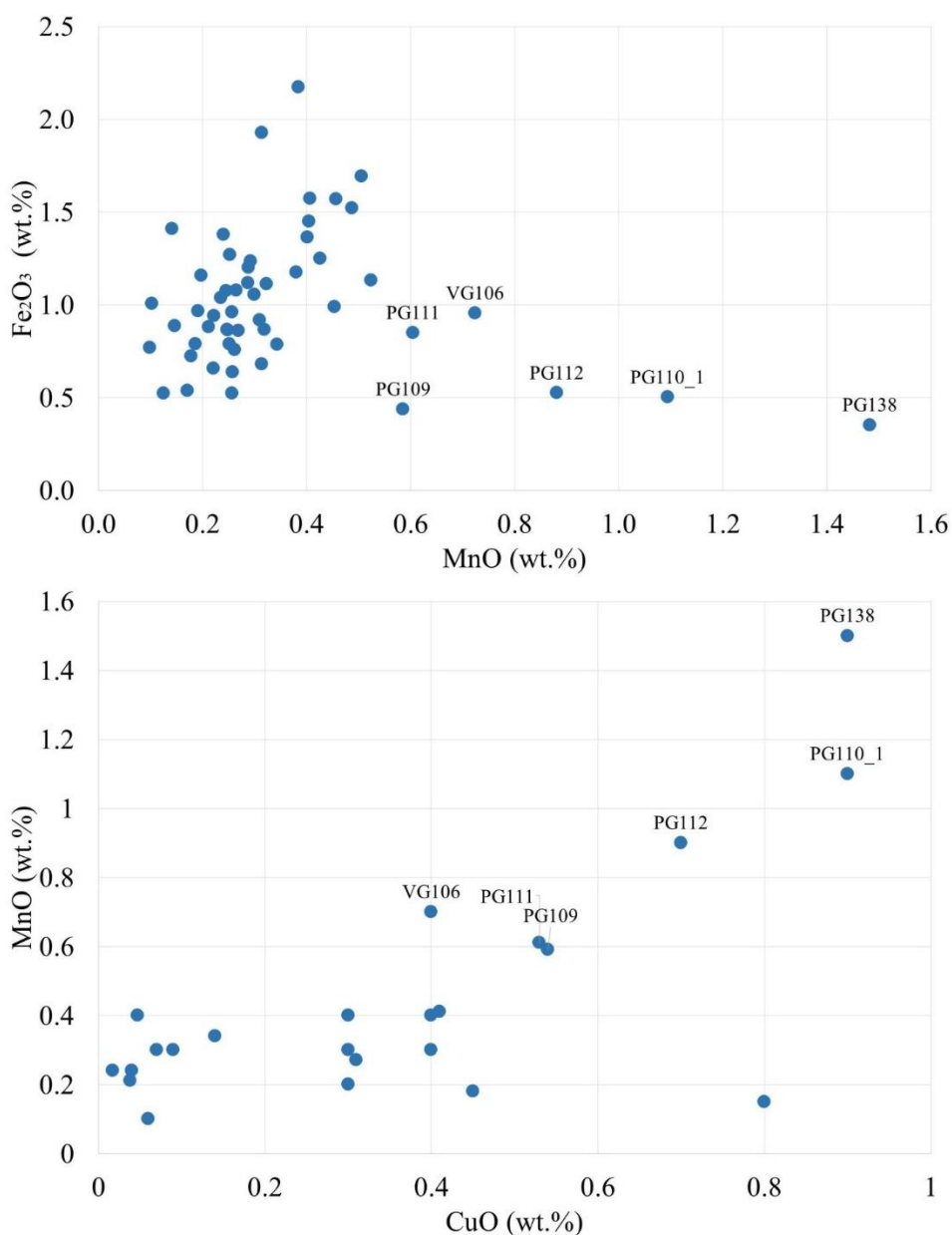
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251

*Figure 7. Ternary scatter plot of Co-Ni-Zn content in the blue parts of the beads of the present study (Lazio beads EI) and data sets of Co-coloured glasses from the literature, each representing a different source of cobalt: Egyptian from Gratuze and Picon 2005 (averaged composition), Conte et al. 2016 and Reade 2021; Erzgebirge from Costa et al. 2021; Other European sources from Towle et al. 2001 and Gratuze and Picon 2005 (averaged composition); Iranian from Oikonomou et al. 2018.*

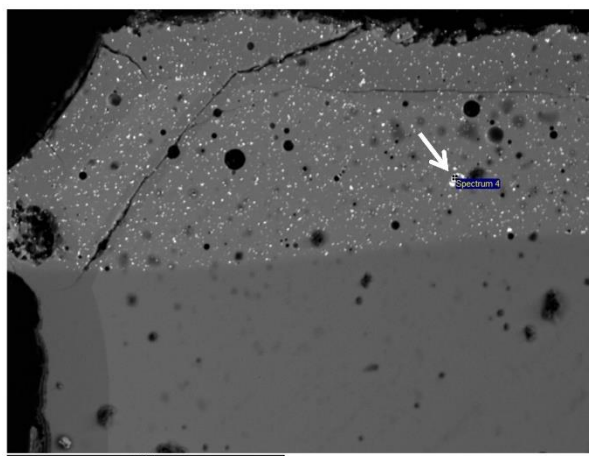
252 The content of Co, Zn and Ni determined in this work for the considered set of blue-eye beads resemble  
 253 the composition of the blue glass coloured by using Egyptian source of cobalt, where this element was obtained  
 254 from cobaltiferous alum and used since the Bronze Age (Abe, 2012). SEM-EDS data (Figure 5) also indicate  
 255 that Mg and Al are abundant in the samples analysed with this technique (namely: PG110\_2, PG139, PG156,  
 256 and VG106). This information further substantiates the hypothesis of the use of Co-rich alum for the production  
 257 process of the beads considered in the present study. The beads analysed in this work are attributed to a time-  
 258 span in which the production of Co glasses in Egypt temporarily ceased (Kaczmarczyk and Hedges, 1983).  
 259 Starting from the 9<sup>th</sup> century BCE, in fact, Co blue glass was produced in Nimrud (Iraq) where craftsmen  
 260 apparently used evaporitic soda-rich deposits as the source of flux and Co-bearing alum as a colorant, both  
 261 imported from Egypt (Reade et al., 2005).

262 MnO is associated with Fe<sub>2</sub>O<sub>3</sub> in the majority of the samples (Figure 8, top), but it also shows another  
 263 independent source for a separate group of samples. p-XRF data would suggest that the beads PG 109, 110\_1,  
 264 111, 112, 138 and VG106 can be considered as a separate group of beads coloured by Co-Cu (and with  
 265 substantial MnO content). This result is in good agreement with the FORS spectra.  
 266



267  
 268 *Figure 8. MnO/Fe<sub>2</sub>O<sub>3</sub> (top), and CuO/MnO (bottom) binary plots of the samples analysed with p-XRF (blue bases' values*  
 269 *only).*

270  
271 In these samples, Co levels were below the LOQ for p-XRF (but the samples still exhibit  $\text{Co}^{2+}$  bands  
272 when analysed with FORS). On the other hand, CuO levels of these samples are higher (0.4 – 0.9%) in  
273 comparison with the main group. Moreover, SEM-EDS data (Figure 5) for the one representative of this group  
274 analysed with the techniques, namely PG110\_1, shows lower concentration of MgO and  $\text{Al}_2\text{O}_3$  when compared  
275 with other samples of the main group analysed with the same technique. Finally, for this subset of beads, MnO  
276 concentrations show a strong positive correlation with CuO (Figure 8, bottom) which is not a commonly  
277 observed pattern. This allows us to state that the final colour of this group of samples was influenced by three,  
278 or even four elements (Mn, Fe, Co, and Cu) coming probably as separate components into the batch. This  
279 group of beads (henceforth Co-Cu coloured) encompasses beads from various archaeological periods and from  
280 different archaeological contexts, therefore, no evidence emerged based on distribution in space and time of  
281 this small heterogeneous set. The bead PG112 stands out in this picture, as Co was not detectable both with p-  
282 XRF and FORS.



283 *Figure 9. PG110\_1 cross section BSE image; white part (top) and blue part (bottom); the point of the analysis*  
284 *in the white part featured some 11% of  $\text{Sb}_2\text{O}_5$*

285  
286 Calcium, apparently, performs two functions in the samples. It is most of all a stabilising component  
287 of the batch, with CaO and SrO weakly positively correlated. Ratio of these oxides is higher for white parts  
288 and r value is also higher (about 0.59). The excess of Ca in white parts can be explained by the use of Ca  
289 antimonate as a colouring (and opacifying) agent, as in these parts the antimony levels detected by p-XRF are  
290 higher and the correlation coefficient between Ca and Sb is equal to 0.49. It is worth noting that Sb was  
291 frequently detected also in the blue parts of the beads, but the very small dimension of the beads does not allow  
292 us to speculate on the significance of this element in the blue body of the bead. Figure 9 demonstrates the  
293 presence of inclusions rich in Sb and Ca, that confirms the presence of calcium antimonate, a quite widespread  
294 agent for making white opaque glass (Lahlil et al. 2008).

## 295 296 5. CONCLUSION.

297 The analytical techniques combined in this study to investigate a set of white-eye blue beads selected  
298 to represent the entire *corpus* of presently known beads of this type found in South Etruria and Latium - and  
299 covering the entire timespan that yielded such beads - gave complementary information that allowed to discuss,  
300 at least preliminarily, some compositional feature of the glass batch and of the beads themselves.

301 OM observation of these beads indicates that they were obtained by winding hot glass around a  
302 mandrel, then the decoration was made by inlaying coils of white glass into the soft blue base.

303 It was established that cobalt was the major colorant, as emerged from both FORS and p-XRF data,  
304 and that calcium antimonate crystals were used to obtain the white opaque glass for the decorations, as  
305 highlighted by p-XRF and SEM-EDS data. The compositional features associate a larger part of the  
306 investigated beads with Egyptian raw materials, but the attested lack of production of Co blue glasses and  
307 faience in Egypt during the 10<sup>th</sup>-7<sup>th</sup> centuries BCE suggests Nimrud as the possible place of production, albeit

308 with Egypt still playing a role for the supply of the flux and the cobalt ore. This seems to be an initial point of  
309 a trade network that spanned the entire Mediterranean. Lands of west-Central Italy were incorporated into this  
310 network, apparently, from the 9<sup>th</sup> century BCE, but the traders (probably Phoenicians or Levantine) still need  
311 to be identified.

312 A smaller sub-group of beads appears to be Co-Cu coloured according to FORS, p-XRF and SEM-  
313 EDS data, demonstrating the presence of alternative tradition of glass colouring, possibly attributed to local  
314 imitation, or mirroring a change in the supply chains.

315 This preliminary investigation, mainly based on a non-invasive approach, gives for the first time a  
316 compositional overview on a representative set of the (apparently) homogeneous *corpus* of white-eye blue  
317 beads from IA Central Italy, suggesting a possible provenance for the majority of the beads and highlighting  
318 compositional heterogeneities that need further attention in future archaeometric investigations.

319  
320 ACKNOWLEDGEMENTS. Authors express their gratitude to the administration of the Museo  
321 Nazionale Etrusco di Villa Giulia and the Museo delle Civiltà for granting permissions for the analyses. This  
322 project has received funding from the European Union's Horizon 2020 research and innovation programme  
323 under the Marie Skłodowska-Curie grant agreement No 754511. The contents of this paper are the sole  
324 responsibility of the authors and do not necessarily reflect the opinion of the European Union.

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