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Mellow yellow: An experiment in amber

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ABSTRACT

Amber natron glasses were produced from at least the Hellenistic period and continued to be produced into the early second century CE. However, as with other strong colours used for Roman vessel production, this colour gradually declined in popularity as colourless and blue-green glass came to dominate. Whilst the colouring mechanisms for blue-green glasses are relatively well understood, the cause of the distinctive amber colour is more complex and can be attributed to the iron sulphur chromophore. This paper demonstrates, using analytical data and model glasses, that the amber colour develops during primary production, and that the sulphate-rich natron is key. The analytical data show that most natron amber glass was probably produced in the Levant alongside the more common blue-green glasses, however, its composition is different. Whilst many glass colours were made in a secondary stage, by adding colourants and opacifiers to a blue-green or colourless glass base, amber glass was not made this way since it required a slightly different set of raw materials and melting technologies. These findings suggest that the production of the glass required specialist knowledge, and particularly skilled furnace operation, in order to produce repeatable results. Skilled specialists would also be required to work amber glass whilst retaining the same clear amber hue, especially for complex wares, such as mosaic vessels, where the glass would be reheated more than once.

1. Amber glasses

Translucent natron amber glass (sometimes termed yellow/brown, golden-yellow or golden-brown) is found from at least the early Hellenistic period, in Hellenistic core-formed vessels (mid-4th-3rd century BCE) (Oikonomou et al., 2016) with opaque blue, white and yellow glass colours, and later in cast polychrome vessels. With the introduction of the mid-Hellenistic non-blown (cast) monochrome vessels from the mid-second century BCE, a specific type of vessel, the cast translucent monochrome grooved bowl appears, which is equally divided between ‘colourless’ and naturally coloured ‘golden-brown’ and yellowish-green (Grose, 1989, 193–4). These were followed in the first century BCE by ribbed bowls also found in similar colours, including ‘naturally coloured’ and amber (Grose, 1989, 244). Amber glasses are however, most common in the early Roman period in cast and blown forms, for monochrome tablewares but sometimes polychrome vessels, particularly on sites of Claudian and early Flavian date (Table 1). Although the majority of Roman glasses are transparent shades of light blue or green (Jackson and Paynter, 2015), in this early Imperial period strongly coloured glasses such as dark blue, purple and green were more commonplace, and amber glasses formed part of this brightly coloured repertoire. This colour continued to be produced in the early second century CE, but was relatively rare by the later second century in common with many other strong colours (Price and Cottam, 1998, 15).

The distribution of the mid-Hellenistic cast monochrome vessels led Grose (1989, 194) to suggest their origin was the Syro-Palestinian coastal zone, although there is no production evidence yet to support this. By the first century CE, amber vessels appear to have been formed at local centres through the empire as evidenced by the glass waste from workshops at Avanches (Amrein et al., 1995) and Montee de la Butte (Robin, 2012, 49).

Compositional studies to date show that, with rare exceptions, these natron amber glasses are high in silica (c. 68–70 wt%) soda (c. 18 wt%) and lime (c. 8 wt%) and contain relatively few impurities (Lemke, 1998; Jackson et al., 2009; Oikonomou et al., 2016; Reade and Privat, 2016). In this respect, amber glasses are almost identical to contemporary, but much more common, blue-green glass. These transparent blue, green and amber glasses are coloured from the impurities which are present in the raw materials used in their manufacture, in particular from the iron minerals in the sand. Whilst the colour mechanisms for blue-green glasses are relatively well understood (e.g. Bingham and Jackson, 2008), the cause of the distinctive amber colour eluded researchers for some time (Brill and Schreurs, 1988, 273, note

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Table 1
Amber glasses from sites in Italy (Oikonomou et al., 2016[1]; Gallo et al., 2013[2]), Syria (Reade and Privat, 2016[3]) UK (Freestone and Stapleton, 2015[4]), France (Lemke, 1998[5]) and Switzerland (Amrein et al., 1995[6]) compared to (a) manganese blue green glasses presumed to originate from the Levant (Gallo et al., 2013[2]) and (b) antimony colourless glasses presumed to originate from Egypt (Foy et al., 2004[7]). Standard deviation given in brackets. Est – estimated, b.d. – below detection, n.m. – not measured, *oxide wt%.

Wt%	Date	*n*	SiO_2	Na_2O	CaO	Al_2O_3	K_2O	MgO	Fe_2O_3	TiO2	MnO	Sb_2O_3	P_2O_5	SO_3	Cl	Cu	Pb	Ba
Satricum, Italy[1]	Mid-4th–3rd C BCE	4	69.7 (1.5)	18.1 (0.4)	7.38 (0.59)	1.77 (0.66)	0.50 (0.16)	0.56 (0.05)	0.30 ((0.05)	0.07 (0.01)	0.01 (< 0.01)	< 0.001	b.d.	0.33 (0.10)	1.45 (0.08)	23 (18)	18 (17)	183 (72)
Jebel Khalid, Syria[3]	3rd–1st C BCE	20	69.3 (2.2)	17.3 (1.2)	8.02 (1.55)	2.17 (0.20)	0.70 (0.15)	0.51 (0.14)	0.35 (0.06)	0.05 (0.01)	0.14 (0.20)	0.06 (0.01)	0.09 (0.02)	–	–	b.d.	n.m.	n.m.
Unknown, UK[4]	1st C BCE–1st C CE	4	69.3 (1.6)	17.5 (1.2)	7.28 (1.9)	2.44 (0.12)	0.74 (0.07)	0.44 (0.06)	0.27 (0.04)	< 0.1	< 0.1	< 0.4	< 0.1	0.39 (0.12)	1.11 (0.12)	< 0.1*	< 0.1*	n.m.
Adria, Italy[2]	1–2nd C CE	9	68.1 (1.2)	19.2 (0.9)	7.55 (0.33)	2.54 (0.09)	0.63 (0.09)	0.54 (0.06)	0.31 (0.03)	0.05 (0.03)	0.03 (0.01)	b.d.	0.11 (0.03)	0.23 (0.08)	1.67 (0.13)	14 (14)	11 (6)	211 (14)
Frejus, France[5]	1st–2nd C CE	28	68.1 (1.35)	18.1 (0.65	8.32 (0.87)	2.30 (0.17)	0.73 (0.08)	0.47 (0.06)	0.26 (0.04)	< 0.05 (0.01)	< 0.1 (0.06)	< 0.1	0.09 (0.03)	0.19 (0.06)	1.00 (0.13)	36 (115)	12 (7)	182 (19)
Avenches, Switzerland[6]	Mid-1st C	15	71.7 (2.1)	Est.	8.31 (1.56)	2.35 (0.33)	0.63 (0.16)	0.26 (0.12)	0.37 (0.05)	0.07 (0.01)	< 0.1	< 0.1	b.d.	0.12 (0.18)	0.70 (0.13)	0.05* (0.07)	0.20* (0.04)	0.04* (0.03)
Blue green Adria (a)[2]	1–2nd C CE	13	69.0 (1.43)	17.6 (1.0)	7.71 (0.50)	2.51 (0.08)	0.64 (0.14)	0.51 (0.06)	0.40 (0.08)	0.06 (0.01)	0.50 (0.21)	< 0.05	0.13 (0.04)	0.20 (0.08)	1.39 (0.21)	105 (145)	66 (72)	317 (220)
Antimony Col'less (b)[7]	1st–mid-3rd C CE	94	71.0 (0.10)	19.1 (1.0)	5.56 (0.53)	1.94 (0.17)	0.42 (0.08)	0.43 (0.09)	0.34 (0.10)	0.06 (0.02)	0.02 (0.06)	0.52 (0.17)	0.02 (0.03)	n.m.	n.m.	n.m.	n.m.	n.m.

22); although it is now attributed to an iron sulphur chromophore, which forms only under certain conditions (Schreurs and Brill, 1984).

However, amber glasses have a distinctive compositional feature which sets them apart. Most Roman glasses, whether colourless, transparent blue-green, or strongly coloured, contain some manganese or antimony oxide added as either a colourant, decolouriser or opacifier, or to modify their hue (Jackson et al., 2009; Paynter et al., 2015) but the concentrations of these additives in the amber glasses is negligible.

In this paper, we use compositional data for Roman amber glass to investigate the consistency of the composition over time, where the glass was made, and how it was used. In the second part of the paper, a series of experimental (model) glasses are made using Egyptian natron and laboratory reagents, to demonstrate how the colour was achieved, and how stable that colour is under different conditions. The results are used to revise our understanding of the production, trade and use of this enduring colour, which has been of longstanding fascination to archaeologists and glass technologists alike.

1.1. The technology of amber

The amber colour in ancient glass is produced by a combination of iron and sulphur, which form a ferri-sulphide chromophore in reducing conditions (Schreurs and Brill, 1984; Sanderson and Hutchings, 1987). The iron in Roman glasses is primarily derived from the sand and some from the walls of melting containers (Jackson and Paynter, 2015; Paynter, 2012). In previous experimental work (see Brill and Schreurs, 1988, 273), it has been found that only a small proportion of the iron and the sulphur present is involved in the generation of the chromophore and the colour is intense, so an amber colour can be produced with very small quantities of both (< 0.005 wt% sulphate quoted in Brill and Schreurs, 1988, 274). As both iron and sulphur are present in ancient glass in sufficient quantities, the key to amber glass production was ensuring an adequately reducing environment, but not overly so (Beerkens and Kahl, 2002). The desired chromophore incorporates Fe^{3+} and S^{2-} ions, so the amber colour will also be compromised if the Fe^{3+} ions are reduced to Fe^{2+}, therefore there is an ideal range necessary for stable amber glass. Sanderson and Hutchings (1987) demonstrated that the amber colour can be produced by altering the furnace conditions and/or the internal redox pairs. Soda lime silicate glass, such as natron glasses, are ideal for creating the amber colour as the Na^+ ions act to stabilise the ligand, balancing out any remaining charge (Beerkens and Kahl, 2002).

It was noted by Jackson et al. (2009) that the reducing environment, necessary for the formation of the ferri-sulphide complex, could have been achieved by adding carbon to the batch. Carbon, in the form of woodchips, coal and charcoal, amongst other things, has a long history of use in amber glass production. It is often mentioned in the large body of technical and patent literature concerning amber glass manufacture, particularly from the 19th century onwards, as amber glass was heavily used in bottle production since the colour protects the bottle contents from spoiling in sunlight (Moore and Scholes, 1943). In modern amber glass production, the iron- and sulphur-bearing components are deliberately introduced to the batch, for example as salt cake (sodium sulphate), iron pyrite or oxide, or blast furnace slag, as modern glass making materials contain very little iron or sulphur. Weyl (1981, 237–275) discusses the formation of amber coloured glass in detail, including experiments by Litzow (Weyl, 1981, 253), which suggest the optimum 'carbon amber' glasses were those made with a 1:1 ratio of carbon and sodium sulphate.

1.2. Composition of Hellenistic and early Roman amber glass

Table 1 shows the compositions of Hellenistic and Roman amber glasses, which demonstrates they are remarkably consistent over this time. This shared composition has only a very few exceptions: an early Hellenistic oinochoe (CAT104, mid-4th to 3rd centuries BCE) analysed by Oikonomou et al. (2016) with lower concentrations of aluminium and barium oxides, has been attributed to a group with a possible Egyptian origin. Amongst the Roman examples, 5 plain bowl fragments from Frejus (69, 64, 63, 62, 60) have increased calcium oxide (> 9 wt %), and to a lesser extent magnesium oxide (> 5 wt%), and slightly less silica (< 67 wt%) (Lemke, 1998). These slight deviations in composition are mirrored elsewhere, for example in some of the twenty analyses of amber glasses from the 3rd-mid 1st century BCE site at Jebel Khalid, Syria (Reade and Privat, 2016, JK09, JK25, JK389, JK44, JK45, JK48), and one example from 1st century CE site at Aquileia, Italy (AQ-FC-1/18-2). Otherwise the amber glass from sites in different parts of Europe and spanning the mid-4th century BCE to the 2nd century CE has a fairly consistent composition, suggesting that it was made on a large scale at a specialised centre(s) and distributed widely. This more common composition also remains relatively unchanged for several centuries indicating that production was long-lived.

The composition of the amber glass can be compared with contemporary blue-green glass, for example in the 1st/2nd century CE glass from Adria (Table 1, Gallo et al., 2013, analysed by XRF and EPMA; these data were obtained using the same techniques in the same study so can be compared with confidence). The main distinction between the two compositions is the absence of either antimony or manganese oxides in the amber glasses, but its constant presence in the blue-green glasses. Sayre (1963) described how the presence of manganese oxide (MnO) would promote oxidising conditions, which would inhibit the generation of the reduced amber colour (Sayre, 1963) and this would explain why the levels of manganese in amber glasses are consistently negligible (Gallo et al., 2013; Lemke, 1998).

Otherwise the glass compositions are almost identical. Both iron and sulphur, the elements needed to produce the amber colour, are present naturally in natron glasses, albeit in low amounts. The concentration of iron oxide in the amber glasses tends to be very slightly lower (c. 0.3 wt% vs 0.4 wt% (Gallo et al., 2013) Table 1), and it has been suggested this reflects the use of a particularly pure sand source (Jackson et al., 2009). The sulphate concentration in both amber and blue-green glasses is similar and is derived from the substantial amounts of sodium sulphate present in the natron, although only a small proportion of this dissolves in the glass (Jackson et al., 2016).

Freestone and Stapleton (2015) note that chlorine and sodium concentrations are very slightly higher in their amber glasses. Fig. 1 shows glasses from Adria (Gallo et al., 2013); some cobalt blue glass exhibits notably lower Cl than amber, however the amber have only marginally higher chlorine and sodium than the blue-green glasses. Freestone and Stapleton (2015) suggest the higher concentrations of soda and chlorine are because a slightly shorter duration and lower temperature was used to melt the amber glasses, to avoid spoiling the amber colour, and that amber glass was obtained directly from the primary glassmaking furnaces without an intermediate phase of melting. Jackson et al. (2016) suggest that there are many variables affecting the Cl content of natron glasses; possible factors, including production location, choice of raw materials and final glass composition, are explored later in this paper.

A range of questions arise from this review of Hellenistic and Roman amber glasses:

- Given the consistency in composition, where was most amber glass made?
- How was the amber colour produced in these natron glasses?
- Was the amber colour generated during the primary melting of the batch or could it be produced in a secondary stage using ready-made glass?
- How stable is the amber colour?

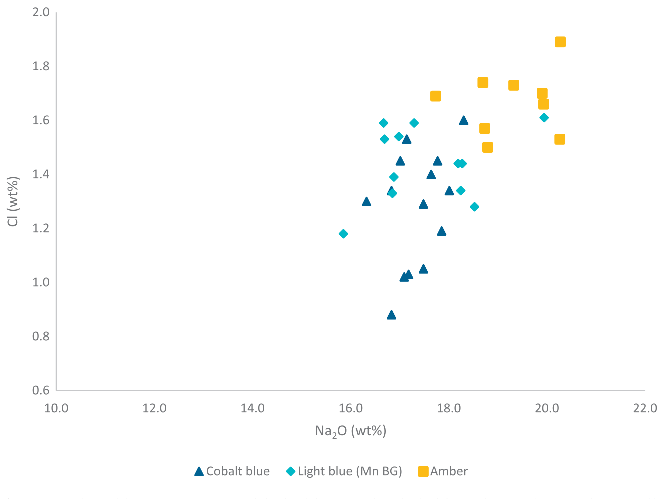

Fig. 1. Amber, blue-green (named light blue) and cobalt blue glasses from Adria (Gallo et al., 2013). Light blue is manganese blue-green (Mn-BG), the common raw blue-green composition in the eastern Mediterranean (Jackson and Paynter, 2015). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

2. Methods

To investigate the mechanisms by which an amber glass could be produced, a series of experiments were devised, making model glasses to mimic a basic Roman glass using laboratory reagents, silica sand, (Loch Aline L30A pure glassmaking sand with < 0.01 wt% iron (Table 2)), and laboratory grade calcium carbonate. The sodium was provided either by:

- Egyptian natron, sourced from El Barnugi in Egypt.
- A model natron (trona) made of sodium bicarbonate (40 wt%), sodium sulphate (30 wt%) and sodium chloride (30 wt%), in proportions based on the analysis of 'natron' from el Barnugi (Jackson et al., 2016).
- Sodium carbonate only (to compare the results in the absence of the sulphate component of the natron).

Molar calculations were performed to produce 10 g of glass which had the composition of 70 wt% SiO_2, 20 wt% Na_2O and 10 wt% CaO (see Jackson and Smedley (2004) and Jackson et al. (2016) and Supplementary data). Each starting batch of reagents weighed in the region of 22 g depending on the addition of variable amounts of iron oxide and carbon, described below. The reagents were mixed together and placed in a mullite crucible, but with the addition of different concentrations of carbon in the form of powdered charcoal. Small concentrations of iron oxide in the form of Fe_2O_3 (0.5 wt%) were added to some batches of the model glass to mimic a typical Roman composition. Crucibles were heated to 1100 °C or 1200 °C for specified times (12 to 36 h) in a slightly oxidising/neutral atmosphere, in an electric furnace, and cooled to room temperature.

The model natron glass (both with and without added charcoal) and the natron glass were analysed by SEM-EDS to determine whether the actual and predicted compositions were a good match and to test the influence of the charcoal on the final glass. The analytical procedures for SEM-EDS are given in Paynter et al. (2015). The trace elements for the Roman amber glasses were determined using LA-ICP-MS, using conditions outlined in Jackson and Nicholson (2010). The methodology, instrumentation and data validation are reproduced in full in the Supplementary data.

3. Experimental results

Analyses of the model glass batches showed that the actual and predicted compositions were a good match, and that the addition of 1.5 g of charcoal did not produce a significant difference in the composition of the final glass (Table 3); therefore the composition of the glasses was fairly consistent and only a subset were analysed. The interaction of the crucible with the glass is demonstrated by the variable aluminium, potassium and iron concentrations, discussed in more detail by Jackson et al. (2016). The concentrations of chlorine and sulphur in the glass made with real natron are lower than in the model glasses, as noted by Jackson et al. (2016), which may be due to combined water in the natron, although the exact reasons are unclear.

Many of the experiments successfully produced amber glasses (Table 4). Fig. 2 shows model natron batches, demonstrating a clear colourless glass without charcoal (Fig. 2b), and an amber glass resulting from an identical batch with added charcoal (Fig. 2a). The results also showed that there are several critical factors that affect the production of an amber hue, which are highlighted in turn below.

Table 2
Composition (oxide wt%) provided by Tilcon (1999, 1) for Lochaline sand (L30A) and the mullite crucible fabric and the El Barnugi natron used in the experiments; other elements reported in Jackson et al., 2016, nm = not measured.

Chemical Composition	SiO_2	Al_2O_3	Fe_2O_3	Na_2O	K_2O	CaO	MgO	SO_3	Loss on ignition
Lochaline sand	99.8	0.05	0.009	< 0.05	< 0.01	< 0.02	< 0.05	< 0.01	0.07
Mullite crucible	53.4	42.2	1.08	0.85	1.40	0.16	0.39	< 0.2	nm
El Barnugi natron	0.8	0.2	0.2	52.0	< 0.1	0.3	0.4	3.5	nm

3.1. Iron concentration

The Lochaline sand (L30A) contained < 0.01 wt% Fe_2O_3, such that melts with no added charcoal gave a completely colourless glass (e.g. exps. A0, B0 and D0, Fig. 2b). However, when the charcoal content reached a critical point (exps. B1.5, D1.5, Figs. 3–4) an amber colour resulted, confirming that in sufficiently reducing conditions the amber chromophore can be produced despite very low concentrations of iron, and that it is a very strong colourant (Fig. 3a, top). In some batches (exps. D1, A2.5) the glass was colourless towards the surface but amber towards the bottom, and plumes of amber could be seen rising from the crucible base when the glass was sectioned (Fig. 3b). Here diffusion of iron from the crucibles (Table 2) has raised the iron concentration sufficiently (in these small batches) to cause localised formation of the amber colour (Jackson et al., 2016). The colours of the model glasses were more variable because most iron was derived from the crucible in an unpredictable way.

When 0.5 wt% of Fe_2O_3 was deliberately added to the model glass batch, a deep brown colour resulted more consistently, and with less carbon (e.g. exps. C1.5 compared to B1.5, Fig. 3a). However, an olive-green colouration was sometimes seen rather than a bright amber (Fig. 3a, exp. C1.5), presumably due to a proportion of blue Fe^{2+} ions being present together with the amber chromophore, as suggested by Brill and Schreurs (1988). So, for a clean amber colour, a low iron concentration is ideal; too high and the colour becomes very dark or olive toned. Conversely if too little iron is present it is difficult to produce the amber colour, therefore there is an ideal range, between 0 and 0.5 wt%, which produces a clean colour.

Table 4 also shows that under the same parameters the real natron and model natron glasses produced different results, the natron glasses tending to produce a darker hue than the model natron glasses (Fig. 3d and e). Natron, as a natural material, contains small concentrations of iron (Jackson et al., 2016), but the laboratory grade sodium compounds used to produce the model natron glass do not. One interpretation is that the iron in the natron may derive from the underlying geology, and it is also utilised by microorganisms which live in the natron lakes, which produce the iron-containing pink blooms such as those seen at Wadi Natrun. These microorganisms need iron in order to thrive; the iron is then captured in the natron as it dries. These small concentrations or iron (and potentially any organic material) help the colour generation further.

3.2. Sulphate

Natron naturally contains large concentrations of sulphate (approximately 1/3 of the natron from el Barnugi, although this was found to be variable throughout the raw material (Jackson et al., 2016)). In all cases the real natron glasses produced an amber or brown glass (except the lowest charcoal batch) (Table 4, Fig. 4). In the model glasses which did not contain any iron, those without any added sulphate remained colourless (A1 to A2) until the maximum amount of charcoal was added, at which point amber plumes formed in the glass solely adjacent to the crucible fabric (A2.5) (Fig. 3d bottom, Table 2 (mullite composition)). This concurs with experiments conducted by Moore and Scholes (1943, 59) who also used a simple silica, soda and lime mixture to demonstrate that a strong amber colour can be made even when only traces of sulphur and iron are present. However, these experiments also show that an excess of reducing agent (carbon) is then necessary when compared to sulphate-rich batches, and that with negligible sulphate the colour is less strong and less stable. It can be argued that the unique combination of elements found in natron, most importantly the high sodium and sulphur and low concentrations of iron, allow a good amber colour to be developed.

3.3. Charcoal

The introduction of an organic reducing agent, in this case charcoal, proved to be crucial to producing amber glass, and the amount added had a considerable effect on the final colour (Fig. 4, Table 4). There is clearly a critical concentration of carbon necessary to produce a visible amber hue (Table 4, B1–1.5 and D1–2) for a given iron concentration. In natron glasses, increasing the concentration of charcoal increased the colour hue from only plumes of amber deriving from the crucible base where the iron concentration was higher (D1, Fig. 3b), to a deep brown glass when the charcoal concentration reached 15–20 wt% in the batch (D2, Table 4, D2.5 Fig. 3d).

3.4. Furnace parameters

The furnace atmosphere could not be altered in these experiments; it was consistently neutral to oxidising, as demonstrated by the golden colour of the melts with added iron (C0 and D0 with no charcoal), consistent with Fe^{3+} ions.

A small number of batches were repeated to test how temperature and duration of heating may affect the amber colour (Table 4). Whilst the results must be interpreted with caution because the batch sizes are very small (10 g equivalent of glass), and reaction with the crucible begins to substantially alter the glass composition with increases in the duration or temperature of firing (Table 4 (A2.5), Fig. 3c, and verified by analysis (see Jackson et al., 2016, Fig. 5a)), both variables influenced colour.

Two of the batches were melted at 12 h and then 36 h, and in both cases the amber colour diminished with increased firing time. D2 (with real natron) was a very dark brown after 12 h but a lighter colour after

Table 3
SEM-EDS analyses of model glasses (average of 3, normalised) made with model natron, with and without added charcoal, and real natron (n.b. the small concentrations of potassium and aluminium oxide derive from crucible contamination, and from the natron when this is used (together with small quantities of iron, magnesium and calcium (Jackson et al., 2016)).

	Batch	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	FeO
Model 'natron' glass	No charcoal	19.01	< 0.1	< 0.1	67.38	< 0.2	0.63	1.70	0.49	10.37	< 0.1	< 0.1
Model 'natron' glass	1.5 g charcoal	19.20	0.12	0.21	67.62	< 0.2	0.63	1.61	0.44	9.97	< 0.1	< 0.1
Natron glass	No charcoal	19.29	0.26	1.35	67.53	< 0.2	0.24	0.77	0.72	10.14	< 0.1	0.15

Table 4
Results of the experimental melts (referred to in the text as A0, A1, A1.5 etc. with the letter A–D indicating the table row, and the number 0–2.5 the amount of charcoal added), at 1100 °C and for 12 h unless stated otherwise.

	Flux	Variable/charcoal	0 g	1 g	1.5 g	2 g	2.5 g
A	Na_2CO_3		Colourless	Colourless	–	–	Amber plumes (@ 1200 °C colourless)
B	Model natron		Colourless	Colourless/very pale yellow	Amber	–	–
C	Model natron	+ 0.5 wt% Fe_2O_3	Very pale yellow	Blue/green	Brown/olive	Dark brown (@36 h mottled green amber)	–
D	Egyptian natron		Colourless	Amber plumes	Deep amber	Deep amber (@36 h light amber)	Dark brown/black

36 h, whereas C2 (model glass with added iron) melted for 12 h produced a deep brown but when melted for 36 h produced a mottled amber-green (Fig. 3c). Changing the temperature from 1100 °C to 1200 °C also altered glass colour in a similar way. For example, A2.5 showed amber plumes at the lower temperature but was colourless at the higher temperature (Table 4). At higher temperatures or longer durations these melts are more likely to oxidise, so the amber colour diminishes.

4. Analytical results and discussion

Table 1 shows that there is a fairly standard composition of amber glasses, indicating that the organisation of amber glass production follows the established pattern for many natron glass types. Large manufacturing centres produced raw glass on a large scale, sometimes in specific colours, and this production continued for a long duration.

4.1. *Where was amber glass made?*

The dominant production areas for natron glass throughout the Roman period and beyond, based on archaeological, historical and chemical evidence, are in Egypt and in the Levant. There is increasing and persuasive evidence that in the Roman period Egypt specialised in antimony colourless glass (so-called Alexandrian glass) and the Levant region specialised in manganese blue-green glass (so-called Judean glass) (see Foy et al., 2004; Jackson and Paynter, 2015, Nenna et al., 2005, Gliozzo, 2017, Paynter and Jackson, forthcoming and Schibille et al., 2016). As well as favouring different additives, the concentrations of lime and alumina are higher for the glasses from the Levant area compared to the Egyptian type (group 3 and group 4 of Foy et al., 2003 respectively, Table 1). Table 1 shows that the majority of amber glasses have virtually identical compositions to the manganese blue-green glasses from the Levant, suggesting both glass types may have used similar sand sources and therefore shared a common origin.

Table 5 shows new trace element data for a selection of amber glasses from Fréjus measured by LA-ICP-MS; the major element compositions are given by Lemke (1998) with averages shown in Table 1. Fig. 5 shows the average, trace element concentrations measured for these amber glasses compared with raw glass from Apollonia in the Syro-Palestine region (Freestone et al., 2000) and blue-green raw glass from the Mljet shipwreck (which demonstrates a typical blue-green Syro-Palestinian composition) (Radić and Jurišić, 1993). The similar trace element profile for these also supports a Levantine origin for the majority of amber glasses. The Mljet glass is used as a comparison here since it provides a larger suite of trace elements than that from Apollonia but shows the same normalised REE pattern. This high Sr peak is typical of the sands of the Syro-Palestinian region.

Although most amber glasses appear to have a Levantine origin, it has been noted in 1.3 that there are outliers in terms of major and minor element composition. The five calcium-rich compositions amongst the Fréjus glasses are included in Table 5 (highlighted in bold). Although they are slightly different in terms of their calcium, aluminium and

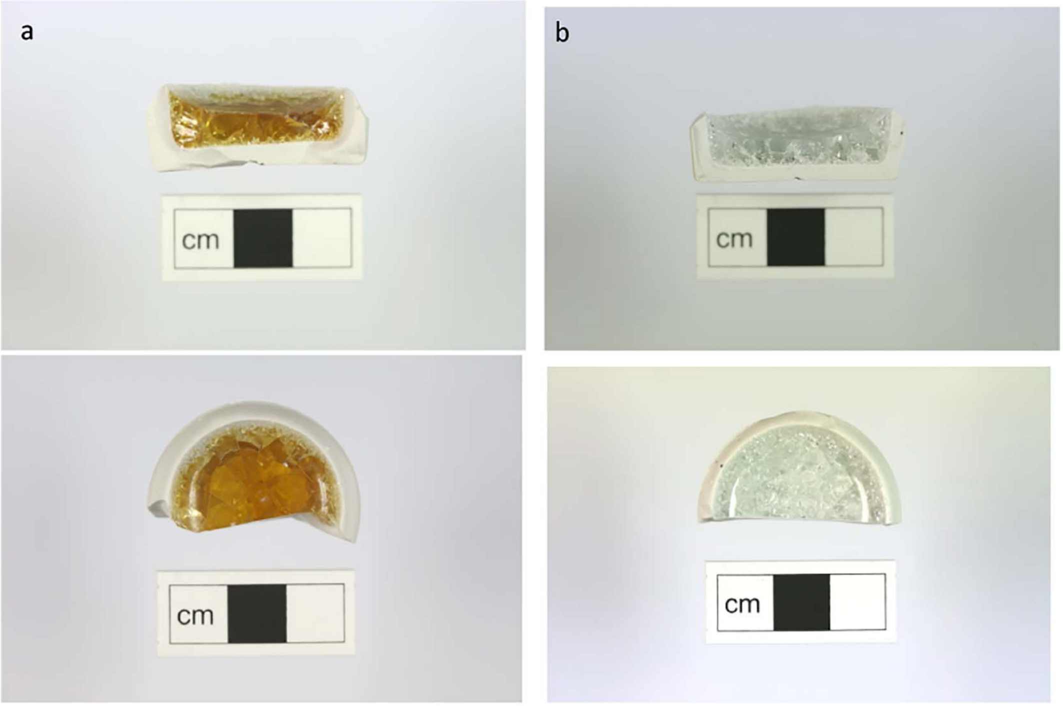

Fig. 2. Simple model soda-lime-silica glasses (20:10:70) (Table 4, glass B), 2a: an amber glass with the introduction of a small concentration of powdered charcoal, 2b: colourless glass is produced with the three component mixture without charcoal. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

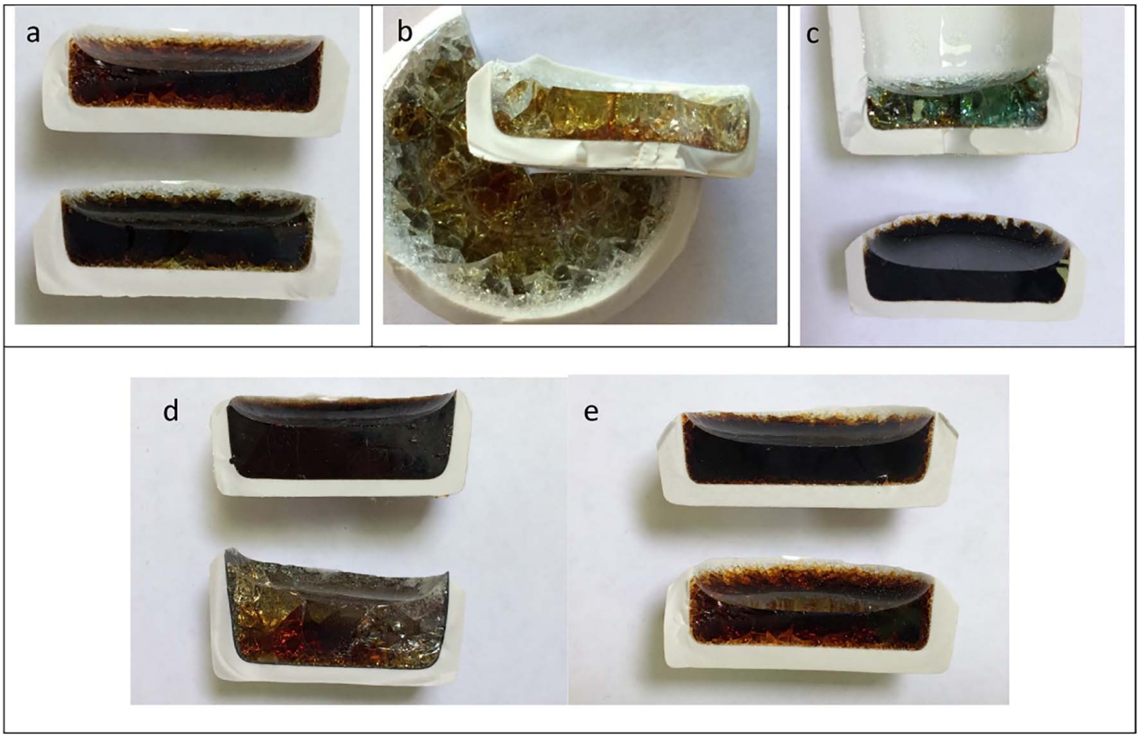

Fig. 3. a) Model glasses made with 1.5 g charcoal: top (B1.5 without iron) is amber, whereas bottom (C1.5 with 0.5 wt% added iron) is olive.
b) Glass batch D1, produced using natron, with 1 g added charcoal, without added iron. Note plumes of amber coloured glass arising from the base of the crucible and a yellowish-green base glass grading to colourless.
c) Model glasses with 0.5 wt% iron oxide and 2 g charcoal (C2) melted for differing times, 12 h (bottom) and 36 h (top), showing loss of amber colour probably due to oxidation and increasing melt/crucible interaction.
d) Glasses containing 2.5 g charcoal, made with natron (top; D2.5) versus sodium carbonate only (bottom; A2.5), showing the effect of sulphate on colour development.
e) Model natron glass B1.5 (bottom) versus real natron glass D1.5 (top), both with 1.5 g charcoal showing the deeper development of colour in the natron glass. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

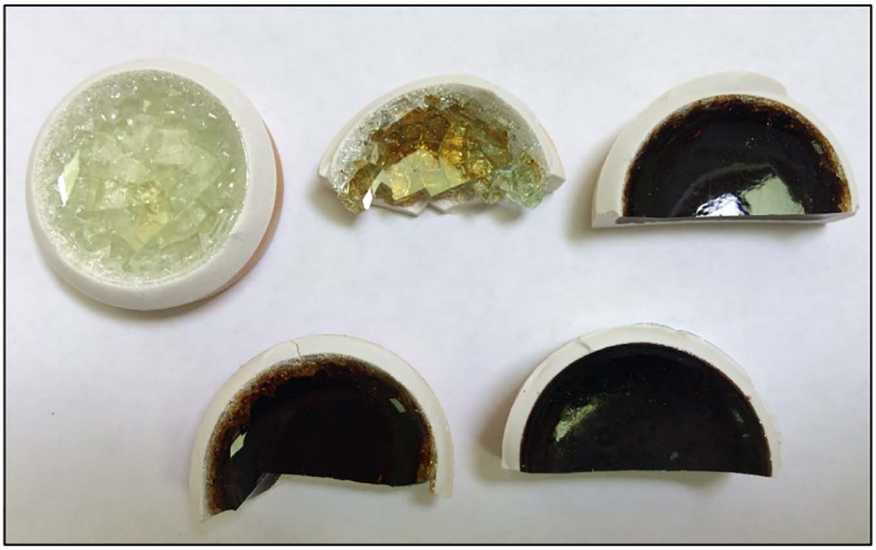

Fig. 4. Natron glasses, produced without added iron, with varying concentrations of charcoal (top left to bottom right (D0–D2.5)).

silica contents there is nothing in their trace elements which indicates a different provenance. Therefore explanations for these differences other than source location may include as unintentional variation in the raw materials as Levantine glasses have a tendency towards higher lime and calcium concentrations (Shibille et al., 2017), or even a chronological separation.

The trace element data for Roman amber glasses also show that the majority have probably not been recycled, since the concentrations of lead, copper and antimony detected in most examples are very low (Gallo et al., 2013, Tables 1 and 7 this study). There are only occasional exceptions, such as Frejus 65, which contains elevated levels of manganese (Lemke, 1998). This suggests that recycling of amber glasses may have been problematic.

4.2. Amber colour generation and control

The experiments show that amber glass can be successfully produced, even in an oxidising furnace atmosphere, by adding carbon in

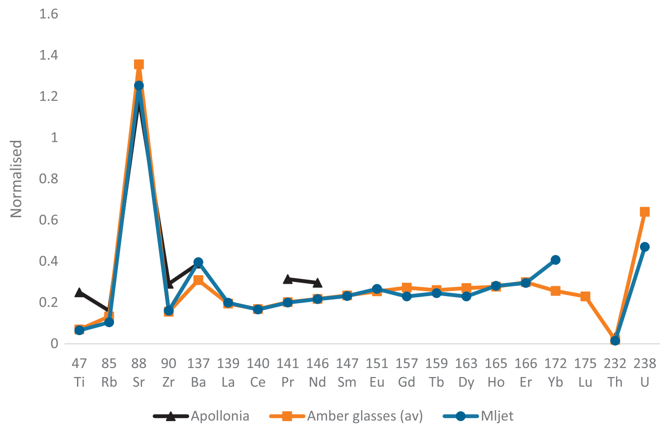

Fig. 5. Trace element compositions of amber glasses from Frejus (this study and Lemke, 1998), raw glass from Apollonia (Freestone et al., 2000), and raw glass block from the Mljet shipwreck (unpublished data), normalised to the crustal averages taken from Wedepohl, 1995.

the form of charcoal to an alkali glass batch containing very little iron or sulphur (Douglas and Zaman, 1969; Schreurs and Brill, 1984), but that the colour is often variable, ranging from colourless to dark green to amber. It is the fine balance of these variables that determine the hue.

4.2.1. *Charcoal*

In wood-fired reverberatory furnaces, the large surface area to volume ratio of a glass melting tank might otherwise result in a partially green or colourless batch due to oxidation at the surface, and temperature and atmosphere require skill to control. The addition of charcoal to the batch would have ensured that the glass nonetheless stayed slightly reduced (cf. Schreurs and Brill, 1984, 206), making the regulation of furnace atmosphere less crucial to control the redox state of the iron chromophore. The composition of charcoal varies depending on the species, but calcium compounds, followed by magnesium, are typically a major component (as well as carbon). However, most of the charcoal reacts and is lost as gases; only a very minor proportion contributes to the glass composition; the experiments indicate that approximately 10 to 20 wt% charcoal may have been sufficient to produce the colour, and that the incorporation of such small amounts of organic matter to archaeological amber glass may not leave any detectable chemical trace, as seen in the experimental batches.

4.2.2. *Natron*

A key advantage to the production of the amber colour was in the natron, which contained a significant proportion of sodium sulphate (Jackson et al., 2016). The model batches with no added sulphate described above, were more persistently colourless, only producing an amber/green colour at the crucible interface when an excess of charcoal was added. Batches containing sulphate turned amber more readily, because the sulphate provides the S^{2-} ions necessary for the formation of the iron sulphur chromophore.

The glassmaker must have also adjusted the proportions of natron and sand, adding slightly more natron than typical for contemporary blue-green glass. Concentrations of soda at around 19 wt% in these model glasses match the elevated levels observed in most Roman amber glass (Table 1). A high concentration of soda alkali is also known to enhance the amber colour (Moore and Scholes, 1943; Harding, 1972; Beerkens and Kahl, 2002). Weyl (1981, 253) reports that glasses with low soda produced 'greyish' shades and the precipitation of NaS. This was observed here when a calculation error led to a glass with much lower soda, which did not turn amber, but remained colourless even with > 20% added charcoal (not reported). Slightly higher sodium content might also have been beneficial in slowing oxidation or reoxidation of the glass, so spoiling the colour, by allowing the use of marginally lower temperatures or shorter firings.

4.2.3. *Low iron sands*

Low iron sands were specifically selected for the ancient amber glasses. These experiments show that the glass is more likely to turn olive brown with higher iron concentrations (Fig. 3a) due to the reduction of some iron to blue Fe^{2+} ions, supporting observations from archaeological material (Schreurs and Brill, 1984). Proportionately lower iron may also be a dilution effect as a result of increasing the proportion of natron in the batch.

4.2.4. *No additives added to the batch*

Most Roman Levantine glass is blue-green, which has a similar composition to the amber glasses but typically contains a small amount of manganese oxide (Jackson and Paynter, 2015; Vichy et al., 2003). In blue-green glass, the manganese is insufficient to decolourise the glass but its purpose may have been to regulate the colour, ensuring the glass remained slightly oxidised, becoming a consistent fresh blue-green throughout the depth of the tank. Without the manganese the results may have been more unpredictable, the glass may have become more green, dark green or even brown, like the amber glasses. Uniquely, antimony and manganese additives were omitted from amber glass to facilitate reduction of the iron. Most natron glass (other than amber) had either added antimony or manganese; the presence of these additives would also make secondary production of amber glass, using blue-green or any other primary glass, difficult.

These results indicate amber glasses were made with a natron-rich batch, the selection of low-iron sands, the addition of specific amounts

Table 5
Trace element concentrations (ppm) from a selection of amber glasses from Frejus measured by LA-ICP-MS (major elements given in Lemke, 1998), bold text is for subset with higher calcium contents.

Form	Cat no	47 Ti	85 Rb	88 Sr	90 Zr	137 Ba	139 La	140 Ce	141 Pr	146 Nd	147 Sm	151 Eu	157 Gd	159 Tb	163 Dy	165 Ho	166 Er	169 Tm	172 Yb	175 Lu	208 Pb	232 Th	238 U
Ribbed bowl	104	259	10.4	397	32.0	164	5.83	8.39	1.58	6.50	1.35	0.33	0.71	0.17	0.77	0.17	0.64	0.08	0.39	0.08	6.46	0.69	1.02
Ribbed bowl	103	280	10.9	419	32.0	171	5.99	9.24	1.56	6.32	1.50	0.32	1.33	0.12	0.79	0.25	0.46	0.10	0.35	0.10	6.20	0.68	0.56
Ribbed bowl	101	294	10.1	398	37.3	199	6.11	9.63	1.37	5.83	1.13	0.21	1.28	0.12	1.17	0.24	0.59	0.09	0.54	0.05	10.79	0.97	2.02
Plain bowl	**60**	**326**	**8.8**	**505**	**31.6**	**185**	**6.52**	**11.48**	**1.71**	**7.11**	**1.60**	**0.39**	**1.37**	**0.19**	**1.09**	**0.26**	**0.75**	**0.09**	**0.79**	**0.07**	**18.30**	**0.81**	**1.72**
Plain bowl	61	245	8.3	445	33.1	177	5.41	10.13	1.24	5.86	1.37	0.35	0.99	0.15	0.99	0.21	0.75	0.11	0.62	0.12	7.26	0.47	1.08
Plain bowl	**62**	**248**	**8.9**	**489**	**31.9**	**170**	**5.67**	**9.99**	**1.50**	**5.33**	**0.91**	**0.36**	**1.44**	**0.23**	**1.50**	**0.24**	**0.81**	**0.15**	**0.66**	**0.11**	**4.54**	**0.72**	**0.68**
Plain bowl	**63**	**288**	**11.3**	**527**	**28.8**	**194**	**6.22**	**11.20**	**1.23**	**5.49**	**1.16**	**0.25**	**1.14**	**0.17**	**0.92**	**0.22**	**0.64**	**0.07**	**0.48**	**0.08**	**12.80**	**0.62**	**1.58**
Plain bowl	**64**	**326**	**9.1**	**507**	**30.8**	**173**	**6.45**	**11.82**	**1.42**	**6.52**	**1.53**	**0.36**	**1.23**	**0.15**	**0.83**	**0.23**	**0.51**	**0.09**	**0.51**	**0.07**	**12.87**	**0.79**	**1.17**
Plain bowl	65	275	9.7	423	29.4	203	5.07	9.10	1.18	4.61	0.84	0.29	0.88	0.24	1.14	0.27	0.47	0.06	0.36	0.09	28.85	0.68	1.07
Plain bowl	66	252	11.9	412	29.6	168	5.18	9.62	1.11	4.97	1.03	0.26	0.82	0.13	1.17	0.23	0.62	0.06	0.38	0.07	10.67	0.63	0.62
Plain bowl	67	257	14.1	396	31.9	188	5.61	9.94	1.19	5.74	1.02	0.40	1.03	0.21	0.81	0.18	0.46	0.12	0.52	0.09	7.79	0.59	0.75
Plain bowl	68	292	9.8	397	31.6	171	5.59	9.50	1.19	5.48	1.32	0.31	0.72	0.15	0.92	0.15	0.75	0.09	0.48	0.05	20.77	0.68	0.90
Plain bowl	**69**	**261**	**10.8**	**552**	**29.9**	**185**	**6.33**	**11.30**	**1.32**	**6.56**	**1.39**	**0.45**	**1.23**	**0.17**	**1.26**	**0.23**	**0.69**	**0.73**	**0.59**	**0.08**	**13.57**	**0.64**	**0.98**

of organic material to control the redox and without the addition of manganese or antimony. Together, these are the key reasons why amber glass was coloured at the point of primary production, and not at a later stage during secondary production. The reported consistent, subtly unique composition of amber glass supports this (Table 1).

4.3. Furnace parameters

The colours of amber glasses vary widely from light honey shades to dark brown (Grose, 1989, 245), reflecting predominantly different concentrations of iron in the raw materials and changing redox conditions. In addition to the materials used in production, this also has implications for the furnace structures, melting temperatures and duration of heating of both the initial glass and upon reworking.

4.3.1. Primary production

Kock and Sode (1994) describe how wood-fired tank furnaces for primary glass production in India ran at substantial temperatures for several weeks, before being left to cool. Assuming Roman amber glasses were made in tank furnaces (as they have uniform compositions and were made on a large scale), then relatively large proportions of organic matter would probably have been needed to maintain the amber colour at high melting temperatures for a long period. However, long melting times or high temperatures would not have been beneficial to the generation and maintenance of the colour. Therefore, the addition of slightly higher proportions of natron to the batch would have minimised the melting times and temperatures, and might account for the marginally higher chlorine contents observed. Amber glasses therefore may have had slightly different melting regimes to the manganese blue-green glasses.

Studies of ancient pottery, and experimental pottery kilns, have also shown that pottery kiln firings often cycled through oxidising, reducing, then re-oxidising conditions (Dawson and Kent, 1999), influenced by stoking practices and fuel-type as well as furnace design. Special adaptions were required to ensure consistently oxidising conditions (muffle kilns) or reducing conditions (sealing the kiln during cooling). It is likely that reverberatory glass furnaces were subject to similar variation in atmosphere, and that glassmakers would need to control it, so for example the amber glass furnaces may have been largely sealed during cooling to preserve a more reducing environment.

4.3.2. Re-melting and secondary production

Secondary working furnaces did not need such high temperatures or long durations in order to simply re-melt the glass, compared to the primary glassmaking furnaces, which required both to melt and homogenise the batch. Taylor and Hill (2008) found that their wood-fired, updraught pot furnace for glass re-melting tended to be more oxidising than the gas furnace used to make the initial batch, with the result that the glass was blue-green from the gas furnace and a pale ‘apple’ green from the wood-fired furnace. Similarly, the Roman glass from the secondary melting site at Basinghall, London was bluer at the base, and greener (interpreted as more oxidising) towards the surface (Wardle et al., 2015). Nonetheless, glassworkers would have to carefully control the temperature and atmosphere as the glass was remelted so the glass did not oxidise, since high temperatures and long heating durations would cause the glass to lose the amber hue.

5. Conclusions and archaeological implications

The key findings relate to both the provenance and technology of the amber glasses, which are:

1. *The majority of amber glasses appear to have a Levantine origin*. Amber glass is compositionally related to blue-green glasses originating in that area; the glasses have a matching trace element profile and their major elements correspond. Those amber glasses with slightly different major element profiles (e.g. higher calcium) also have a similar trace element pattern, again indicating a similar provenance.
2. *The amber colour of the glass was produced during primary production*. Although related to blue-green glasses, amber glass was not produced using the same raw material batches or melting episodes, nor was it produced in a secondary stage by adding reducing agents to blue-green raw glass. Blue-green glasses contain manganese, whereas amber glasses do not.
3. *Unexpectedly, very low iron concentrations are best to produce a clean amber colour*. Glassmakers must have sourced sand with an especially low concentration of iron to ensure that the optimum ratio of Fe^{3+} and S^{2-} ions was achieved to make an amber, rather than olive, colour.
4. *Natron is key to colour development, as it is high in sodium and sulphur*. The sulphur in the amber glasses would derive from the natron and this, in conjunction with the high sodium concentration, would have facilitated amber colour development.
5. *The addition of an organic reducing agent is crucial*. Large volumes of organic material are needed to produce the amber hue by establishing and maintaining reducing conditions throughout firing.
6. *The production of amber glasses required skilled control of temperature and atmosphere to produce the desired result, as the amber colour diminishes if the atmosphere is not closely controlled, particularly at higher temperatures or longer durations*. The glassworkers may have used techniques similar to those used in pottery production, for example sealing the furnaces during cooling.
7. *Amber glasses can be remelted for secondary working, but are not suitable for extensive recycling*. The introduction of impurities into the glasses through recycling, particularly the decolourisers antimony or manganese present in most Roman glass, would spoil the amber colour, as would gradual oxidation through continual remelting.

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Appendix A. Supplementary data

Supplementary data to this article, on experimental methods, can be found online at https://doi.org/10.1016/j.jasrep.2017.11.038.

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