



On the making, mixing and trading of glass from the Roman military fort at Oudenburg (Belgium)

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Abstract

This paper presents the analysis of decoloured and naturally coloured glass from well-dated contexts in the southwest corner of the Roman fort at Oudenburg (Belgium) ranging from the late second to the early fifth century AD. The aim is three-fold. First, provide comparative material in the study of glass consumption from the northwestern provinces of the Roman Empire. Secondly, evaluate possible diachronic shifts in the applied decolourizing agent to produce colourless glass as to assess potential correlations between glass production recipes, provenance and chrono-typology. Finally, provide an added value to the research of glass recycling and mixing in the Roman imperial period. Nine subgroups are distinguished based on their chemical composition determined by LA-ICP-MS: Sb-only, two groups of Mn-only, four groups of mixed Mn-Sb, HIMT and one glass without any decolouring agent. The Sb-decoloured glass is used in the earliest phases and can be attributed to an Egyptian provenance. The two subgroups of Mn-glass likely come from different provenances: one from Egypt and the other later one from the Levant. Most of the glass shows high marks of mixing based on high trace elements concentrations and the simultaneous presence of antimony and manganese. Inhomogeneous mixing of manganese and antimony was also detected through μ XRF. One Mn-Sb subgroup likely comes from mixing antimony glass with HIMT. The obtained results help better recognise the shifts in applied glass recipes throughout the Roman imperial period and improve our understanding about the mixing and recycling of glass to supply a Roman military camp.

Keywords Roman glass · Oudenburg · Belgium · Recycling · LA-ICP-MS · μ XRF

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Introduction

During the last decades, material science methods were extensively applied in archaeological glass research. Involving a varied geographical range, recent research focuses on regions scarcely explored so far (Cholakova et al. 2016; Coutinho et al. 2017; de Juan Ares and Schibille 2017). The military camp of Oudenburg, Belgium, has five neatly dated successive building phases between the end of the second and the beginning of the fifth century AD (Vanhouette 2007). This is an added value as it helps refining the diachronic overview on glass production and use throughout the vast Roman Empire. A further interest to analyse the glass consumption from this military fort concerns the site location at the North Sea coast. Acting as a crossroad of trade and communication routes on the Channel relating Britain and the Continent (Millet 1990), the North Sea and river Rhine must have played an important role in introducing glass within the Roman military networks (Stern 1999).

From the perspective of material culture, glass from the military camp at Oudenburg also provides an insight in the daily use of windowpanes, vessels and jewellery at a regional level. The supply of glass consumers' goods in the northwestern provinces was the result of a long-lasting trade of raw glass from primary workshops in the eastern part of the Mediterranean towards secondary workshops distributed all over the Empire (Freestone 2005a; Silvestri 2008). Based on the archaeological finds of large tank furnaces in the south-eastern Mediterranean in Levant (Gorin-Rosen 2000) as well as in Egypt (Nenna et al. 2003) and the high number of shipwrecks with large loads of raw glass (Nenna 2007), a 'centralised' model of production is generally accepted (Freestone et al. 2000; Gorin-Rosen 2000; Nenna et al. 2000; Tal et al. 2004). This model implies the distribution of raw glass which was re-melted, (de)coloured and shaped into objects in secondary glass workshops, which, in general, resulted in a relatively homogeneous chemical composition of the glass matrix throughout the Roman Empire (Freestone et al. 2002; Janssens 2013). Glass from Roman times has a typical soda-lime chemical composition with 0.4 wt% of iron oxide on average, a sand impurity which causes glass to have a blue-green hue (Freestone 2015). Roman glassmakers were able to neutralise this 'natural' hue by adding decolouring agents that oxidise the reduced iron to its oxidised form, giving the glass a yellowish tinged colourless appearance (Gliozzo 2017; Jackson 2005). As Gliozzo (2017) highlights, colour description in ancient glass is complex and there is a wide range of hues related to the generic term 'colourless'. The decolouring agents added to the glass are either manganese oxide or antimony oxide; the chemical composition ranges of different decoloured glass subgroups were first given by Foster and Jackson (2010). Trace elements and chemical composition comparisons indicate that Roman manganese (Mn)-decoloured glass was produced on the Syro-Palestinian coast (Foy 2000; Jackson et al. 2015) and already used in the first millennium BC, whereas antimony (Sb)-decoloured glass seems to have been made with sand from Egypt characterised by lower concentrations of lime, alumina and titanium compared to the Levantine glass. This was first proposed by Schibille et al. (2016a, b) and then reported as well by others (Gliozzo 2017). Although the technology of decolouring glass was already known from early periods, the popularity of decoloured glass increased during the Roman period reaching very high production volumes in the late first and second century AD and declining from the late third century AD (Gliozzo 2017). In general, the use of manganese and antimony follows opposite trends: while antimony was predominant in the first century AD, it was used less and less afterwards giving manganese more importance as a decolouring agent (Gliozzo et al. 2015; Sayre 1963). Another glass group, HIMT, is characterised by high amounts of iron, manganese and titanium, the term was coined by Freestone in 1994

(Freestone 1994) but first documented by Mirti et al. (1993). The high concentration of manganese oxidises turns iron into its yellowish form preventing the glass from becoming deeply coloured due to the high concentration of iron and the reducing conditions typical of Roman furnaces (Ceglia et al. 2014). This group is supposed to have originated in Egypt and was increasingly common from the mid-fourth century AD (Foy et al. 2003; Freestone et al. 2005; Nenna 2014). More recently, HIMT has been divided into further subgroups based on chemical composition (Ceglia et al. 2015; Foster and Jackson 2009; Foy et al. 2003) and based on both chemical composition and isotopic data as was done in San Giusto (Gliozzo et al. 2017).

Manganese and antimony have been found together in Roman glass (Gliozzo et al. 2015; Jackson et al. 2015). As their simultaneous presence is not necessary to decolour glass, this has been considered as a proof for mixing and recycling of different glass types (Braun 1983; Freestone 2015; Jackson et al. 2015; Nenna et al. 1997; Paynter and Jackson 2018). The study of glass recycling has gained importance in the last years as it is a crucial element to understand glass production patterns as well as ancient technology (Ceglia et al. 2017; Freestone 2015; Jackson et al. 2015; Paynter and Jackson 2018).

To better understand ancient glass production, provenance and distribution, trace elements analysis can be particularly useful. More information about provenance is obtainable by analysing smaller variations of the chemical composition through trace elements (Ceglia et al. 2017; Dussubieux et al. 2016) and isotopes (Brems et al. 2013a, b), because certain elements (e.g. Al, Zr, Sr, Ti, Nb, REE) are characteristic of the geology of the sand used (Brems and Degryse 2014).

The purpose of this study is three-fold:

1. The evaluation of the glass consumption at Oudenburg aims to provide comparative material in the study of glass consumption from the northwestern provinces of the Roman Empire. Indeed, Gliozzo (2017) highlighted that 70% of the glass collection analysed stems from Egypt, Italy and the UK thus raising the necessity to expand the number of regions with analysed Roman glass. Aside from earlier extensive research on the topic from British sites (Foster et al. 2010; Jackson 2005; Jackson et al. 2015), glass from the continental north-western provinces has scarcely been covered by archaeometry. Relatively close to Oudenburg, the glass of the elite tumulus grave at Bocholtz (Netherlands) dating to the late second to early third century has been extensively analysed (Huisman et al. 2009). This assemblage can be compared with that of Oudenburg as both are from the same region and coeval periods (Bocholtz being contemporary to the second phase of Oudenburg) but keeping in mind that the status of the two sites is different (civilian elite tomb versus military camp).

2. The chemical composition and trace element patterns are used to determine the provenance of the material and to discuss glass production recipes. Provenance and recipes are put in parallel with chrono-typology in order to analyse how they evolved from the second to the fourth century AD, to investigate the extent to which they are correlated and to contextualise technological changes through time.
3. The antimony-manganese recycled glass of Oudenburg provides a suitable corpus to assess the rate of homogeneous mixing and recycling of glass and to explore which glass types could have been mixed.

Materials and methods

Archaeological context and analysed samples

The Roman castellum of Oudenburg is situated at the Belgian coast. Presently located 8 km inland, the site was in Roman times directly connected to the sea and to inland road networks linked to other military camps. Around the mid-first century AD, a civil settlement was founded at the site location, whereas the fort was constructed by the end of the second century AD (Ganio et al. 2012; Vanhoutte 2007). Extensive archaeological investigations in the southwestern corner of the site defined five distinct building phases of the military camp. The pre-fort phase (circa 50–180/200 AD) was followed by three wooden fort phases (circa 180/200–280 AD) and two stone fort phases (circa fourth century AD–410). The site was not used in permanent continuity; at some periods, the fort was abandoned for several decades (Ganio et al. 2012; Vanhoutte 2007). Since Roman military networks were particularly useful in introducing glass and glass working techniques (Huisman et al. 2009; Stern 1999), the glass consumption at Oudenburg has a lot of potential to picture the general glass consumption patterns within the wider region. Figure 1 locates Oudenburg and other contemporaneous sites where glass has been analysed and which is compared to the dataset studied here.

The complete set of samples comprises 90 colourless/naturally coloured glass fragments described in Fig. 2. Samples were chosen after a detailed study to include as many diagnostic decolourised or naturally coloured pieces as possible from the different archaeological sequences; they were dated based on their typology. In this paper, we examine whether the analysed colourless and naturally coloured glass is subject to changes in the relatively short-lived trends, which also allows for direct comparison with the extensive literature (Baxter et al. 2005; Gliozzo 2017; Gratuze forthcoming; Jackson 2005; Maltoni et al. 2016; Paynter 2006; Silvestri et al. 2008). As some fort phases remained briefly in use and succeeded each other quickly, such as the wooden fort phases,

some short time archaeological sequences were merged into longer-lasting chronological groups, more appropriate from a material culture point of view. Four larger time periods were thus assigned:

- (1) First period: pre-fort phase: second half first (for certain samples last quarter first) to first half second century AD;
- (2) Second period: early fort phases: second half second to first half third century AD;
- (3) Third period: later fort phases: second half third to first half fourth century AD;
- (4) Fourth period: last fort phase: second half fourth century AD up to about 410 AD.

The majority of the glass is dated to the second half of the third century—first half of the fourth century. This can be directly related to the history of Oudenburg as troops re-installed the Roman fortress in the mid-fourth century bringing with them fresh equipment, food supplies as well as commodities (Vanhoutte 2007).

Analytical methods

The chemical composition (major, minor oxides as well as trace elements) was obtained using laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) at the Ernest-Babelon laboratory, IRAMAT—CNRS, Orléans (Dussubieux et al. 2016; Gratuze 2014) on small fragments mounted in epoxy blocks and polished. This technique has many advantages and can be used to obtain the accurate and precise chemical composition of ancient vitreous materials (Gratuze 2014, 2016). The instrument used was an Element XR mass spectrometer from Thermo Fisher Instrument combined with a Resonetics M50E excimer laser working at 193 nm. The excimer laser was operated at 5 mJ with a repetition rate of 10 Hz. The beam diameter was adjusted between 60 µm for the HIMT glasses to 100 µm for most of the other colourless glasses to avoid saturation from manganese and sometimes from antimony. The depth of the ablation crater is around 150 µm but can vary according to the ablation duration. A pre-ablation time of 15 s is set to allow the signal to reach the mass spectrometer and to eliminate the transient part. An argon/helium gas flow (1 l/min Ar + 0.65 l/min He) carries the ablated aerosol to the injector inlet of the plasma torch. The signal is then acquired for 30 s corresponding to 11 mass scans from lithium to uranium (the signal in count/s is measured in low-resolution mode for 58 different isotopes).

The mass spectrometer offers the advantage of being equipped with a three-stage detector system: a conventional high-performance discrete dynode (a dual mode secondary electron multiplier, SEM) with a linear dynamic range of over nine orders of magnitude associated with a single Faraday

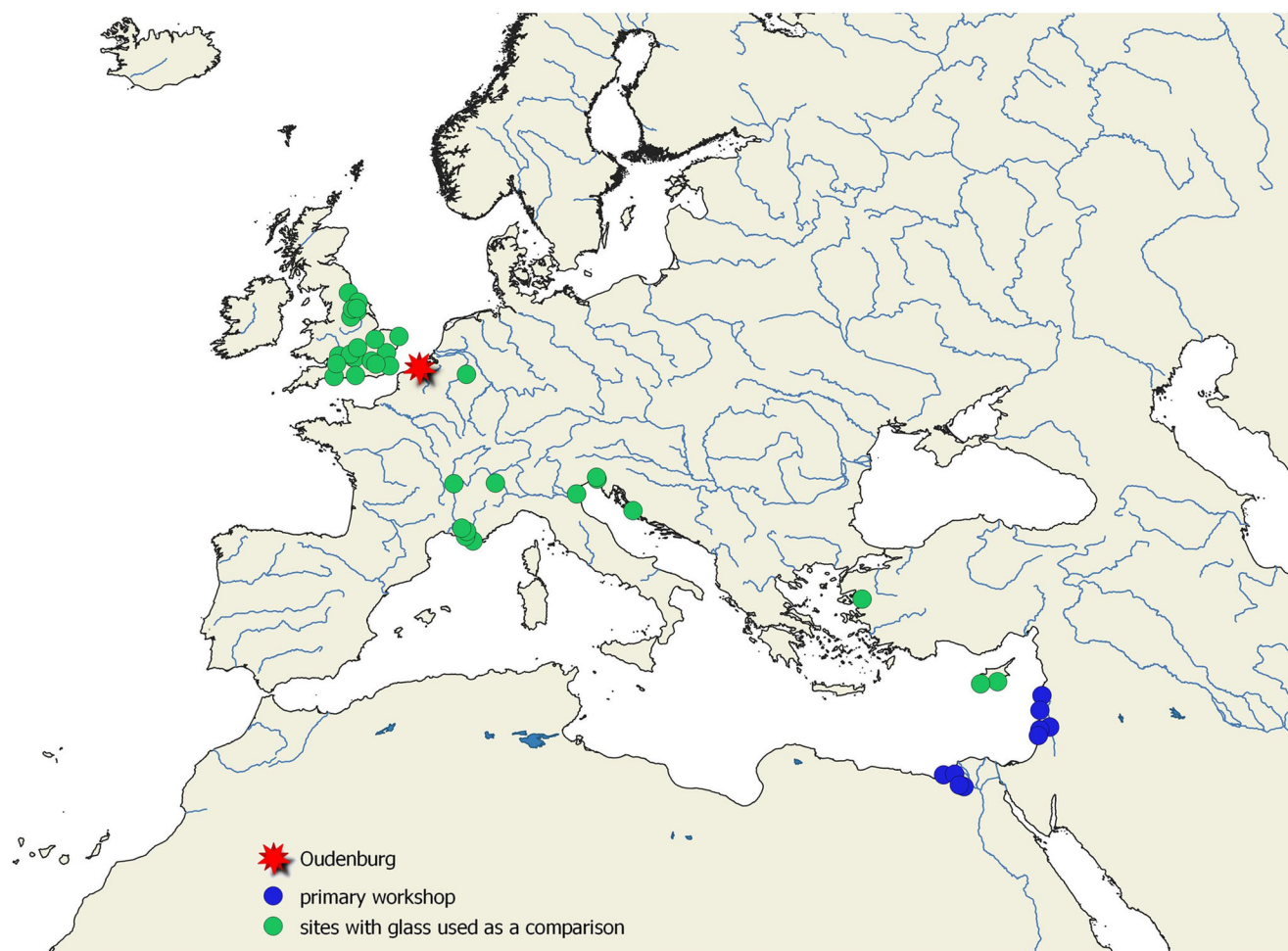


Fig. 1 Map of the location of Oudenburg, other sites with archaeological glass used as a comparison and some primary workshops

collector which allows an increase of the linear dynamic range by an additional three orders of magnitude. This feature is particularly important for laser ablation analysis as dilution of the sample is impossible compared to ICP-MS with liquid sample introduction. This allowed for the quantification of major, minor and trace elements for any concentration of 58 elements in a single run. For each sample, the chemical composition of at least two points was measured.

Five reference standards were measured periodically to calibrate the instrument: NIST610, Corning B, Corning C, Corning D (Pearce et al. 1997; Vicenzi et al. 2002; Wagner et al. 2012) and APL1 (an in-house reference of IRAMAT-CNRS Orléans). Using these references, a response coefficient k was determined for each element analysed as a function of Si. In order to determine the concentration of each element in the unknown samples, the coefficient k previously obtained along with the hypothesis that the sum of all elements equals 100 were used (Gratuzé 1999, 2013).

Accuracy and precision of the measurements were verified overall 13 times with Corning A and NIST 612

analysed quantitatively as unknown samples and verified from the literature (Brill 1999; Pearce et al. 1997; Vicenzi et al. 2002; Wagner et al. 2012), the data are given in Appendix A. The coefficients of variation of major and minor oxides are within 5% while they are between 5 to 10% for trace elements.

In order to characterise the chemical homogeneity of the glass, elemental maps were recorded on a Bruker M4 Tornado μ XRF (VUB—Brussels) using a Rh tube at 40 kV and 700 μ A under vacuum. A polycapillary lens was used to focus the X-ray beam to a circular spot of diameter 25 μ m (Mo-K α) while a Si drift detector was used to detect the characteristic X-ray photons (de Winter et al. 2017). μ XRF was used to obtain chemical composition maps and assess the homogeneity of the glass.

Multidimensional principal component analysis (PCA) on Euclidian distances were carried out on Matlab. The oxides chosen for this analysis are SiO₂, Al₂O₃, Fe₂O₃, TiO₂, Na₂O, K₂O, MgO, CaO (wt%) as well as trace elements Li, B, Sr, Zr, La and Ce (ppm), because these are known for representing

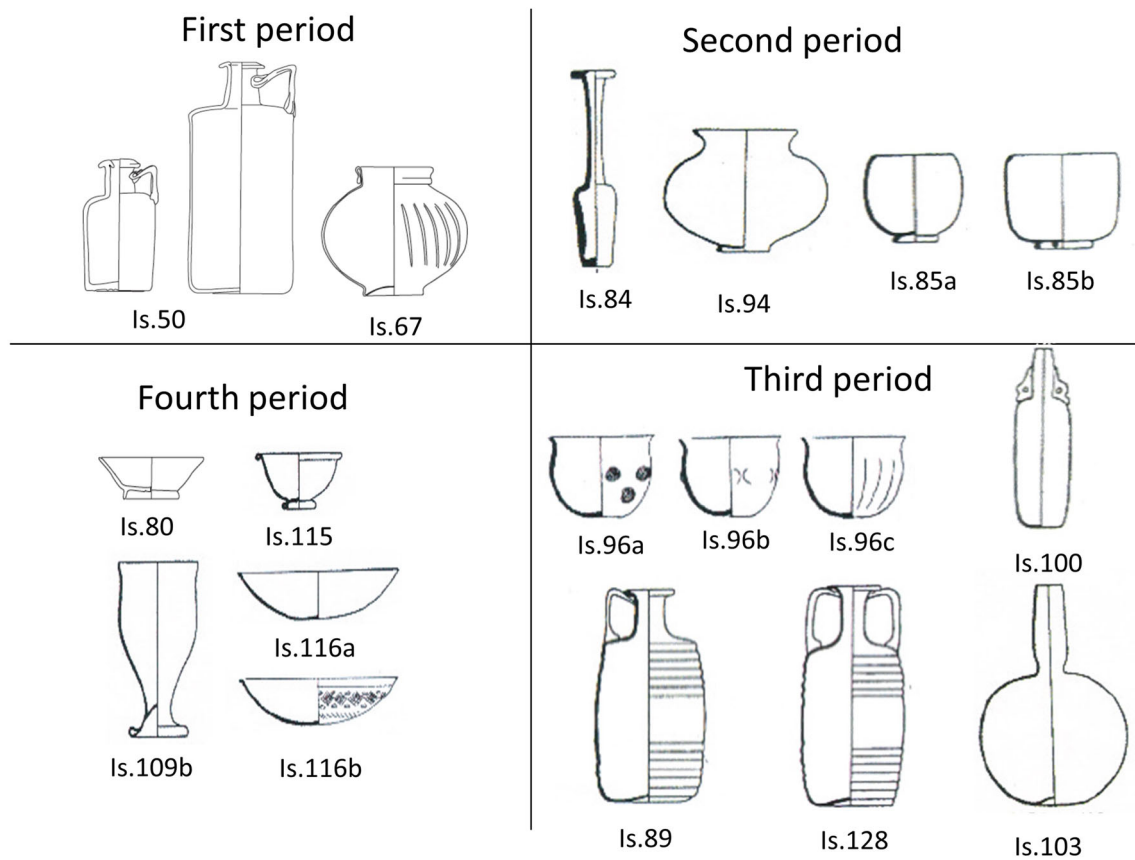


Fig. 2 Drawings of the analysed vessels organised in time period

geological markers of the sand and anthropogenic factors (Brems and Degryse 2014; Ceglia et al. 2017; Phelps et al. 2016). The data used as input was given as standardised variables, i.e. the difference between the value and the mean of the data set normalised to its standard deviation.

Results and discussion

The complete chemical composition of the glass samples along with their typology and description is given in Appendix B. All glasses are soda-lime silicates with typical Roman glass chemical compositions. It appears clearly that natron was used as flux seeing the high level of soda (ranging between 13.1 and 20.7 wt%) in combination with the low concentrations of MgO (below 1 wt%) and K₂O (below 1.2 wt%) while the Cl content is rather high (above 0.7 wt%) (Shortland et al. 2006).

Most of the 90 colourless glass fragments have slight blue-green or yellowish hues apart from the yellow-green HIMT glass. This is due to the decolouring agents, manganese and antimony oxides, intentionally added to neutralise the colouring caused by the natural presence of iron oxide ranging between 0.3 and 1.8 wt%.

Glass grouping

The set was subdivided into different groups based on the chemical composition of the glasses. In order to obtain consistent groups and go further in the analyses of the glass, we followed three main steps.

First, the dataset is approached based on the presence of the decolouring agents antimony and/or manganese; the cutoff values used are based on Gliozzo (2017) who proposes that natural levels of antimony (Sb₂O₃) and manganese oxide (MnO) are respectively 0.012 and 0.025 wt%. Above those values, antimony and manganese must have been either intentionally added or present through glass recycling. A lower value for Sb₂O₃ of 0.0036 wt% has even been proposed by Brems and Degryse (2014). This initial step leads to five broad groups of glass defined by no decolouring agent, antimony (Sb) as the only decolouring agent, manganese (Mn) as the only decolouring agent, the simultaneous presence of manganese and antimony (Mn-Sb) and finally HIMT following the existing grouping proposed in the literature (Gliozzo 2017; Jackson 2005).

Glass grouping based solely on the presence of decolourants would not be sufficient to understand thoroughly the variability of the dataset thus leading to the second step where we deepen the analysis and refine these groups based

on the chemical composition of the matrix and trace elements characteristic of the sand.

Within the group of glass containing manganese as the only decolouring agent, two samples (OU7_1 and OU7_4) are clearly different as their soda concentration is higher while their lime and manganese oxide concentrations are significantly lower than the other manganese glasses (Appendix B). These two samples are thus put together in subgroup Mn-b.

To go further in the analysis of the Mn-Sb glass, it is useful to look at oxides such as TiO_2 and Fe_2O_3 as well as trace elements such as Hf and Zr which are characteristic of the sand (Fig. 3). From this analysis, four subgroups can be distinguished. While Mn-Sb-a and Mn-Sb-d clearly cluster apart from the rest of the Mn-Sb glass, Mn-Sb-b and Mn-Sb-c are somewhat similar. We have chosen to separate them into two groups based on Fig. 3a. OU9_8 was classified as Mn-Sb-c but plots a bit apart on Fig. 3b. This sample also has high trace elements characteristic of recycling (39,472 ppm Pb and 1755 ppm Cu).

In the third step, we verify these groupings based on PCA using standardised values of SiO_2 , Al_2O_3 , Fe_2O_3 , TiO_2 , Na_2O , K_2O , MgO , CaO (wt%), Li, B, Sr, Zr, La and Ce (ppm). Figure 4 shows how the set of glasses plots according to the first two principal components accounting for almost 75% of variability. In this case, PCA is used to determine how the glass matrix and oxides characteristic of the sand and natron, other than decolouring agents, are related to the groups established here.

PCA (Fig. 4) confirms that HIMT clusters apart and that Mn-a and Mn-b are not grouped together. It also reveals complex patterns not only with the Mn-Sb group but also within the Sb-glass itself. Despite the intricacy of Mn-Sb, the four

subgroups plot separately and corroborate the previously discussed considerations.

This three-step analysis leads us to define nine subgroups within the glass set. Their averages and standard deviations are given in Table 1.

As introduced by Freestone et al. (2000), Fig. 5 shows the concentrations of trace elements normalised to the upper continental crust (Kamber et al. 2005). HIMT and mixed manganese-antimony glass have the highest concentrations in trace elements, whereas glass containing antimony only, or manganese only show the lowest amounts.

Sb group ($n = 9$): Nine glasses contain antimony as the only decolouring agent. Glasses from this group have the smallest concentration of iron, lime and alumina (0.38 ± 0.09 , 5.98 ± 0.76 and 1.87 ± 0.15 wt% respectively). Even though this group is quite homogeneous, there is some variability within the group for example regarding the soda, titanium and zirconium content (18.3 ± 0.6 wt%, 395 ± 75 ppm and 39.1 ± 6.3 ppm) and certain samples (OU4_4, OU8_10, OU1_9 and OU5_10) resemble glass from group 4 in the study by Foy et al. (2004). As observed in Fig. 5, antimony glass is the most pristine of all the groups present here, but it can be observed in Appendix B that some antimony glasses have slightly higher trace element concentrations while only one sample (OU5_10) dating from the first to second century contains higher lead content (222 ppm).

Mn-Sb group ($n = 58$): The majority of the glasses in this set contains both antimony and manganese and can be subdivided into four groups (Mn-Sb-a to Mn-Sb-d) based on the concentrations of iron, titanium, antimony and

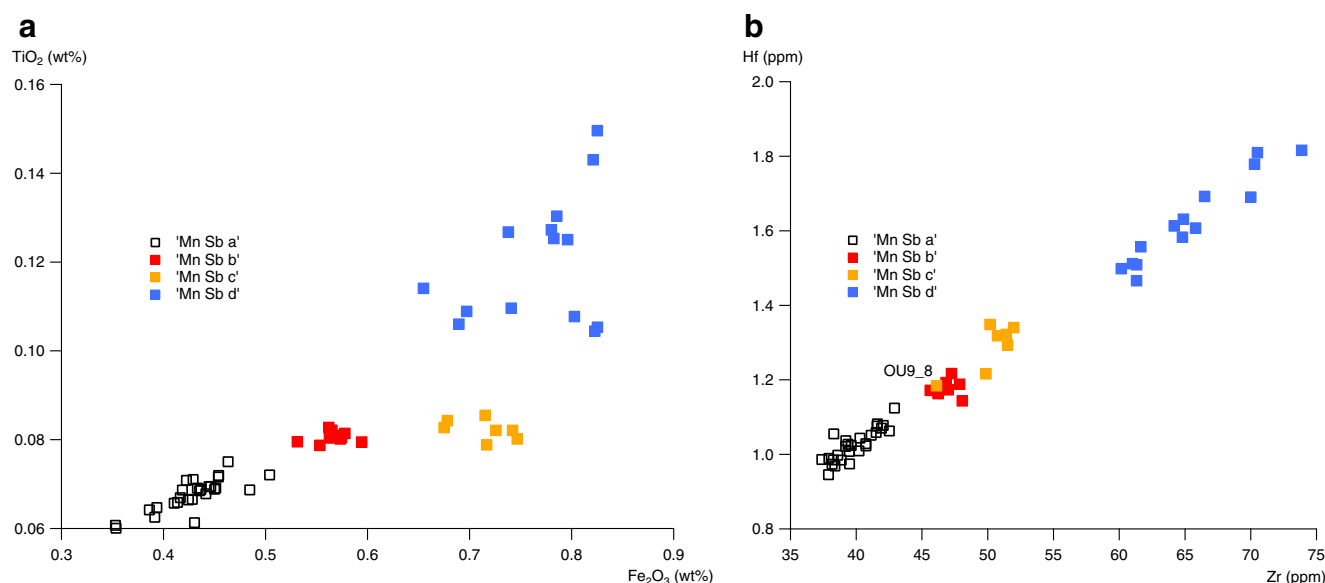


Fig. 3 **a** TiO_2 concentrations as a function of Fe_2O_3 concentrations. **b** Hf concentrations as a function of Zr concentrations. These biplots show that different subgroups of Mn-Sb glass can be defined

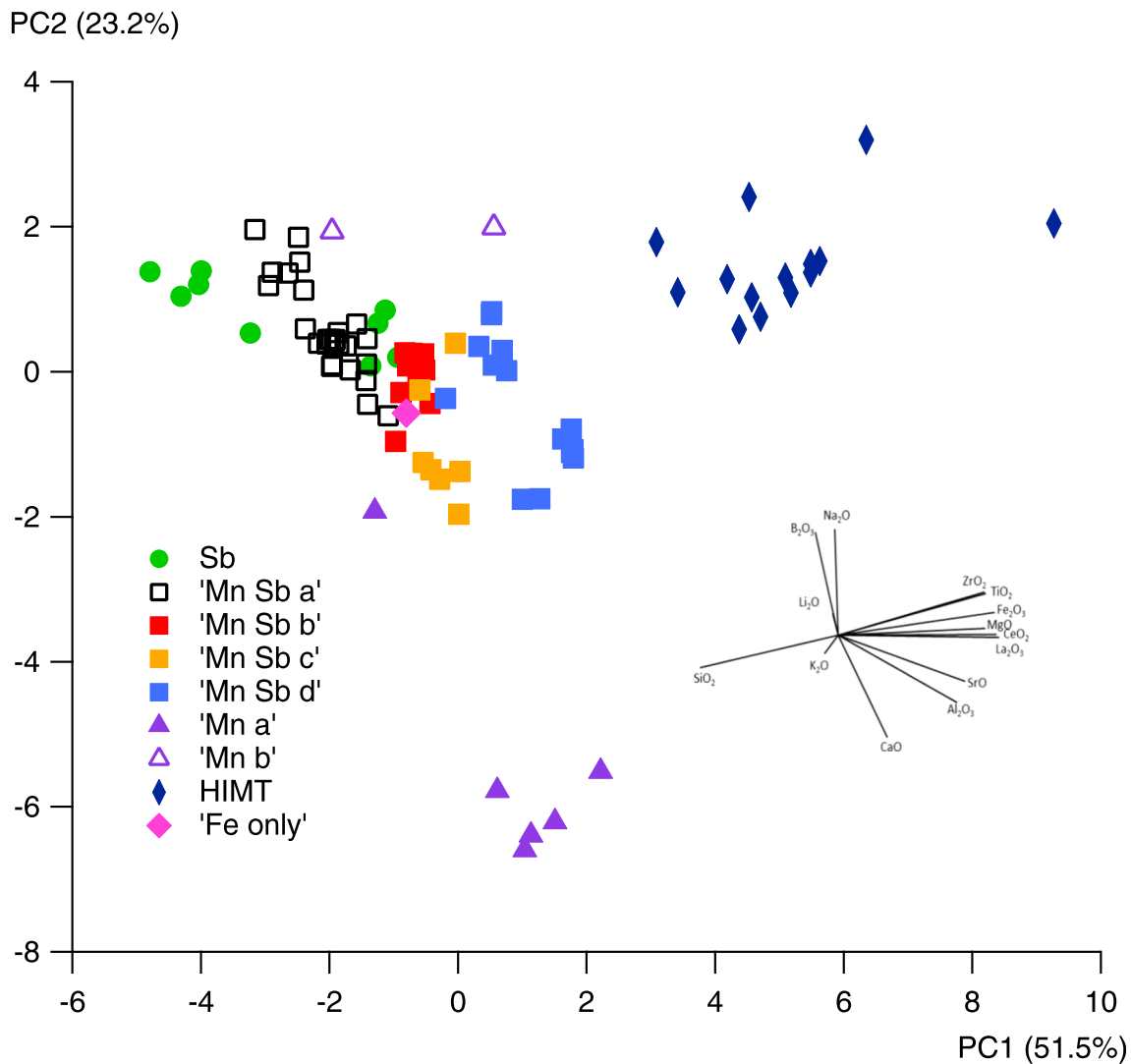


Fig. 4 PCA bi-plot of principle components 1 and 2 showing that the grouping based solely on the presence of decolouring agents is not sufficient to understand the full variability of the dataset

manganese (Fig. 4) with 27 ex. of 'Mn-Sb a', 10 ex. of 'Mn-Sb b', 7 ex. of 'Mn-Sb c' and 14 ex. of 'Mn-Sb d'. The two latter oxides are anticorrelated seeing that there is an increase in manganese and a decrease in antimony from Mn-Sb-a to Mn-Sb-d. The differences between those groups are also visible for trace elements (Fig. 5). Mn group ($n = 8$): The presence of manganese as the only decolouring agent can be found in eight glasses. Two subgroups can be identified. Mn-a subgroup with six samples has low soda (14.4 ± 1.3 wt%), high lime (8.60 ± 0.78 wt%), alumina (2.97 ± 0.25 wt%) and manganese oxide (1.56 ± 0.31 wt%) while the Mn-b subgroup with two samples has higher soda 20.4 ± 0.4 wt%, lower lime 6.11 ± 0.5 wt%, alumina 2.03 ± 0.19 wt% and manganese oxide 1.07 ± 0.08 wt%. This is observed from PCA results (Fig. 4) revealing that two samples differ in chemical composition (OU7_4 and OU7_1) with the rest of the

glasses from the group so that two subgroups of manganese glass can be defined. OU3_4 has an intermediate alumina concentration between these two sub-groups, which is why it plotted slightly apart from the rest of the Mn glass on Fig. 3. However, it is more appropriate to be classified in Mn a based on its soda and lime concentration.

HIMT group ($n = 14$): Fourteen glasses correspond to the HIMT group defined by high amounts of iron, manganese and titanium oxides (respectively 1.33 ± 0.21 , 1.79 ± 0.28 and 0.32 ± 0.06 wt%).

Fe alone ($n = 1$): The only glass sample that contains neither manganese nor antimony is a pale blue sample from the third to fourth century and attributed to a hemispherical bowl with slightly everted rim (type Isings 96). The chemical composition of this glass falls within the range of common blue-green Roman glass with 0.55 wt% Fe_2O_3 .

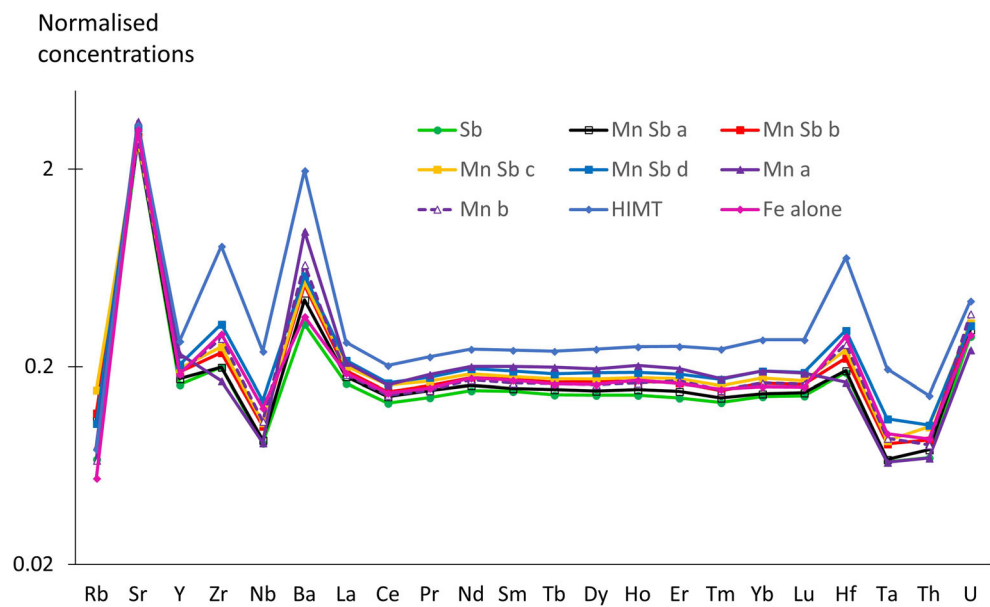
Table 1 Mean (μ) and standard deviation (σ) of the glass groups

Group	n	wt%																											
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	MnO	Sb ₂ O ₃	Na ₂ O	K ₂ O	MgO	CaO	Cl	P ₂ O ₅	Li	B	Ti	V	Cr	Co	Ni	ppm								
Sb	μ	70.6	1.87	0.38	0.07	0.02	0.51	18.3	0.39	0.49	5.98	1.17	0.02	2.80	181	395	7.22	7.91	1.10	2.81									
	σ	1.8	0.15	0.09	0.01	0.01	0.17	0.6	0.07	0.19	0.76	0.06	0.01	1.15	20	75	1.52	1.85	0.25	0.63									
Mn-Sb-a	μ	69.6	2.03	0.43	0.07	0.23	0.42	18.6	0.54	0.48	6.45	1.00	0.05	14.2	181	405	10.6	8.48	2.77	4.40									
	σ	0.4	0.09	0.03	0.00	0.09	0.04	0.4	0.11	0.04	0.35	0.11	0.01	11.6	13	22	1.8	0.85	0.63	0.63									
Mn-Sb-b	μ	68.6	2.21	0.57	0.08	0.36	0.28	18.8	0.58	0.62	6.70	0.95	0.07	15.5	156	483	12.4	9.99	5.66	6.14									
	σ	0.8	0.04	0.02	0.001	0.05	0.06	0.8	0.06	0.03	0.11	0.05	0.01	4.8	5		0.4	0.62	0.73	0.23									
Mn-Sb-c	μ	68.9	2.41	0.71	0.08	0.42	0.33	17.0	0.70	0.57	7.05	0.85	0.12	17.2	160	493	14.8	11.4	8.49	8.09									
	σ	1.6	0.13	0.03	0.00	0.05	0.09	0.5	0.12	0.02	0.36	0.05	0.02	10.5	7	14	1.0	0.6	4.33	0.49									
Mn-Sb-d	μ	68.6	2.31	0.77	0.12	0.60	0.25	17.9	0.57	0.81	6.86	0.93	0.08	15.9	158	721	17.2	13.5	11.6	9.29									
	σ	0.5	0.15	0.06	0.01	0.15	0.06	0.8	0.29	0.10	0.53	0.11	0.06	7.1	6	87	1.3	1.5	3.0	1.10									
Mn-a	μ	69.5	2.97	0.55	0.07	1.56	0.01	14.4	0.43	0.59	8.60	1.04	0.10	2.72	99.0	395	26.5	10.4	8.50	9.62									
	σ	1.0	0.25	0.15	0.01	0.31	0.01	1.3	0.12	0.08	0.78	0.08	0.02	1.38	28.7	33	9.9	2.1	2.08	1.94									
Mn-b	μ	67.1	2.03	0.58	0.10	1.07	0.01	20.4	0.41	0.67	6.11	1.29	0.04	9.13	183	612	16.2	11.2	4.97	7.84									
	σ	1.6	0.19	0.20	0.02	0.08	0.01	0.4	0.06	0.16	0.50	0.16	0.02	6.01	10	145	4.2	1.5	2.30	0.44									
HIMT	μ	66.0	2.56	1.33	0.32	1.79	0.01	18.8	0.45	0.94	6.49	1.00	0.06	10.2	179	1898	35.9	38.2	10.4	13.9									
	σ	1.0	0.13	0.21	0.06	0.28	0.01	0.5	0.08	0.05	0.39	0.10	0.02	6.4	19	383	8.0	8.3	2.8	1.9									
Fe alone	1	71.0	2.01	0.55	0.11	0.09	0.00	17.3	0.30	0.74	6.51	1.23	0.02	4.24	154	643	11.9	15.6	2.02	4.57									
Cu	Zn	15.8	2.31	21.7	16.2	5.46	388	5.14	39.1	5.14	39.1	39.1	39.1	39.1	39.1	39.1	39.1	39.1	39.1	39.1									
8.96	5.0	8.8	16.0	0.41	69	0.43	6.3	0.21	0.03	0.27	0.05	1.36	1407	7	0.44	0.75	0.09	0.39	0.39	0.39									
3.14	41.6	2.60	18.5	8.75	392	5.54	39.9	1.31	0.55	0.20	0.12	12.8	3515	171	5.76	10.0	1.28	5.29	5.29	5.29									
28.6	25.2	0.14	2.9	1.97	21	0.24	1.6	0.07	0.21	0.07	0.04	12.9	298	19	0.30	0.6	0.07	0.22	0.22	0.22									
18.5	48.6	2.82	24.3	9.31	418	5.99	46.9	1.54	1.02	0.48	0.15	25.9	2377	205	6.22	10.6	1.36	5.79	5.79	5.79									
207	6.6	0.06	1.7	1.30	9	0.15	0.7	0.03	0.18	0.19	0.03	12.6	471	13	0.10	0.2	0.02	0.14	0.14	0.14									
92	65.7	3.11	13.1	12.1	400	6.23	50.2	1.60	1.13	0.72	0.18	106	2716	213	6.54	11.5	1.43	6.05	6.05	6.05									
392	26.7	0.06	5.7	21.2	10	0.21	1.98	0.03	0.15	0.71	0.07	163	788	12	0.10	0.34	0.03	0.13	0.13	0.13									
618	64.1	3.18	19.0	7.30	461	6.52	65.5	2.07	1.22	0.33	0.17	28.4	2072	230	7.01	11.8	1.50	6.44	6.44	6.44									
200	33.2	0.17	3.5	14.8	44	0.30	4.3	0.13	0.21	0.19	0.06	34.4	461	20	0.37	0.65	0.06	0.33	0.33	0.33									
189	13.5	4.05	1.92	12.7	496	7.40	33.9	1.27	2.98	0.04	0.05	0.74	72.9	38.0	6.86	11.5	1.54	6.60	6.60	6.60									
11.2	1.8	0.42	0.71	28.4	40	0.67	2.37	0.07	1.68	0.05	0.03	0.30	43.2	32.6	0.61	0.7	0.13	0.55	0.55	0.55									
6.9	34.3	2.92	2.12	12.0	403	5.91	55.3	1.63	3.26	0.35	0.13	12.7	55.9	261	5.93	10.2	1.29	5.65	5.65	5.65									
131	20.0	0.43	0.73	16.9	37	0.46	10.3	0.30	0.45	0.41	0.12	15.5	59.9	118	0.60	1.0	0.12	0.53	0.53	0.53									
162	40.9	4.24	4.26	7.69	476	8.58	163	3.67	3.80	0.41	0.12	20.4	65.5	780	8.65	14.4	1.90	8.08	8.08	8.08									
134	10.1	2.65	0.63	5.01	450	5.83	58.5	1.90	0.30	0.31	0.01	0.61	8.32	142	6.04	10.5	1.32	5.77	5.77	5.77									
6.00																													

Table 1 (continued)

wt%	ppm																
	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Hf	Ta	W	Au	Pb	Bi	Th
1.03	0.27	0.88	0.14	0.84	0.17	0.47	0.07	0.46	0.07	0.98	0.07	0.15	0.05	42.9	0.07	0.76	0.80
0.09	0.02	0.08	0.01	0.08	0.01	0.04	0.01	0.04	0.01	0.14	0.01	0.21	0.08	67.5	0.03	0.10	0.16
1.07	0.29	0.97	0.15	0.89	0.19	0.50	0.07	0.48	0.07	1.03	0.08	0.20	0.05	189	0.05	0.85	0.87
0.04	0.01	0.04	0.01	0.05	0.01	0.03	0.00	0.02	0.00	0.04	0.00	0.15	0.03	372	0.02	0.05	0.04
1.19	0.31	1.03	0.17	0.99	0.21	0.56	0.08	0.54	0.08	1.18	0.09	2.65	0.10	758	0.05	0.95	0.93
0.04	0.01	0.02	0.01	0.04	0.01	0.01	0.00	0.02	0.00	0.02	0.00	1.07	0.08	524	0.02	0.03	0.03
1.23	0.32	1.08	0.17	1.02	0.22	0.59	0.08	0.57	0.08	1.29	0.09	0.69	0.06	5838	0.11	1.11	0.95
0.04	0.02	0.05	0.01	0.06	0.01	0.02	0.00	0.03	0.00	0.06	0.00	1.05	0.07	14,393	0.19	0.03	0.03
1.31	0.33	1.14	0.18	1.10	0.23	0.62	0.09	0.62	0.09	1.63	0.12	0.78	0.10	410	0.07	1.13	0.92
0.07	0.01	0.06	0.01	0.06	0.01	0.03	0.00	0.04	0.01	0.12	0.01	0.85	0.11	432	0.02	0.11	0.06
1.38	0.38	1.21	0.20	1.15	0.25	0.66	0.09	0.62	0.09	0.89	0.07	0.14	0.00	6.04	0.00	0.77	0.69
0.12	0.02	0.12	0.02	0.12	0.03	0.08	0.01	0.05	0.01	0.04	0.01	0.03	0.00	2.22	0.00	0.03	0.06
1.14	0.30	1.02	0.16	0.96	0.20	0.57	0.08	0.54	0.08	1.38	0.10	0.13	0.01	59.0	0.02	0.90	1.05
0.07	0.02	0.02	0.01	0.07	0.02	0.05	0.01	0.08	0.01	0.28	0.01	0.07	0.01	75.4	0.01	0.12	0.04
1.67	0.42	1.50	0.24	1.45	0.31	0.85	0.13	0.89	0.13	3.81	0.22	0.30	0.02	114	0.02	1.60	1.22
0.13	0.03	0.12	0.02	0.12	0.03	0.08	0.01	0.11	0.01	0.70	0.03	0.09	0.03	101	0.01	0.19	0.14
1.18	0.30	0.99	0.16	0.96	0.21	0.55	0.08	0.51	0.08	1.51	0.10	0.04	0.00	7.03	0.00	0.96	0.82

Fig. 5 Concentration of trace elements normalised to the upper continental crust (Kamber et al. 2005), HIMT and mixed glass have the highest concentrations of trace elements and antimony glass the lowest



Comparison of the compositional groups at Oudenburg to contemporary sites

Overall, the compositions of Oudenburg glasses are similar to what has been described in the literature regarding Roman glass found at many sites since the 1960s (Sayre 1963; Sayre and Smith 1961). There are however slight differences appearing in the proportion of certain glass types found as well as links that can be drawn with other sites. A comparison with some assemblages from the literature as well as the averages determined by Gliozzo (2017) are also given in Table 2 showing variabilities and similarities that can exist within certain glass groups. For example, with the Mn-glass, soda content can go from 15.18 wt% in Italian glass to 18.35 wt% in Romano-British material, a difference which cannot be explained only by the use of different analytical techniques. General averages for these chemical composition groups have been given by Gliozzo (2017) in a recent review.

Within the antimony decoloured glass group, some chemical composition variations exist as in many other glass assemblages in Italy (Maltoni et al. 2016; Silvestri et al. 2008), in Egypt (Rosenow and Rehren 2014), in Romano-British sites (Foster et al. 2010) and in the Netherlands (Huisman et al. 2009). In this assemblage, there is only one first to second century sample containing higher lead content (222 ppm). It has been suggested that earlier antimony glass tends to contain higher lead contents (Paynter 2006; Paynter and Jackson 2018), which goes well with the argument that different antimony sources would have been exploited through time (Lobo et al. 2014). There are also variations in the lead content of the Sb glass from Bocholtz (Huisman et al. 2009) suggesting that the Sb glass reaching the northwestern provinces was likely to be a product of the use of different antimony sources.

Mn-a is also very similar to many contemporary manganese-containing glasses, to cite a few examples in Italy CL2 from the Iulia Felix shipwreck in the Adriatic Sea (Silvestri et al. 2008), FC colourless 2 from the domus of Tito Macro in Aquileia, Italy (Maltoni et al. 2016). Both Mn-a and Mn-b differ from manganese-containing glass found in the British sites, where soda levels are intermediate between Mn-a and Mn-b (Foster et al. 2010) even though it might be that these differences are enhanced by different analytical procedures.

The chemical composition of Mn-Sb glass of Oudenburg is within the same ranges and shows similar variability as the well-known groups of glasses with both Mn and Sb discussed in the literature (Baxter et al. 2005; Braun 1983; Foster and Jackson 2009; Gliozzo et al. 2015; Jackson 2005; Silvestri 2008; Silvestri et al. 2008). However, the presence of this large amount of Mn-Sb glass is different from Bocholtz (Netherlands), where the majority of the glass was Sb-decoloured and only three samples contained both Mn and Sb (Huisman et al. 2009). Although Oudenburg is located relatively close, the glass supply in those two sites is quite different. The difference in glass composition can be explained through the fact that Bocholtz was a high-status Roman burial, whereas Oudenburg was a military fort. The type (burial versus fort), the status and the network (civilian versus military) of the site appear to make a more important difference in the glass composition consumed than the geographical localisation.

Several subgroups of HIMT glass have been described (Nenna 2014) but the HIMT glass here resembles rather the HIMT 1 found in Romano-British sites (Foster and Jackson 2009) or the Italian Group E (Mirti et al. 1993) which are contemporary. This suggests that from its appearance

Table 2 Comparison with Sb-, Mn-decoloured glass and HIMT from the literature (*n.a.* not analysed)

Sb		Mn							
Source of data	Gliozzo 2017	Silvestri et al. 2008	Rosenow and Rehren 2014	Huisman et al. 2009	Foster et al. 2010	UK	Maltoni et al. 2016	Aquilaia Italy	Gliozzo 2017
Group	Sb-decoloured	Grado, Italy	Sb-decoloured	Sb glass	Colourless 1		FC colourless 1		Mn-decoloured
Type	Vessel	CL1/1	Vessel	Vessel	Vessel		Vessel		Vessel
Date	1st–7th cent	201–250	1st–2nd cent	late 2nd early 3rd cent	3rd–4th cent		2nd–4th cent		1st–7th cent
<i>n</i>	390	60	8	23	46		7		104
SiO ₂	71.3	70.3	68.4	70.3	<i>n.a.</i>		708		68.7
Al ₂ O ₃	1.88	1.98	2.00	1.93	1.90		2.14		2.30
Fe ₂ O ₃	0.35	0.35	0.53	0.37	0.40		0.46		0.64
TiO ₂	0.06	0.06	0.08	0.08	0.06		0.08		0.10
MnO	0.01	0.01	0.02	0.02	0.04		0.02		1.10
Sb ₂ O ₃	0.53	0.82	0.63	0.57	0.35		0.59		0.02
Na ₂ O	18.5	19.8	17.2	16.3	19.0		18.1		16.7
K ₂ O	0.46	0.41	0.53	0.36	0.39		0.39		0.88
MgO	0.43	0.32	0.59	0.34	0.49		0.49		1.09
CaO	5.47	4.76	6.84	5.29	5.74		5.93		7.53
Cl	<i>n.a.</i>	1.45	0.98	<i>n.a.</i>	<i>n.a.</i>		1.41		<i>n.a.</i>
P ₂ O ₅	<i>n.a.</i>	0.02	<i>n.a.</i>	0.04	0.02		0.04		<i>n.a.</i>
SO ₃	<i>n.a.</i>	0.28	0.29	<i>n.a.</i>	<i>n.a.</i>		0.29		<i>n.a.</i>

Mn		Sb–Mn		HIMT		
Source of data	Silvestri et al. 2008	Foster et al. 2010	UK	Maltoni et al. 2016	Aquilaia Italy	Gliozzo 2017
Group	CL2	2a	2b	FC colourless 2		Mn/Sb-decoloured
Type	Vessel	Vessel		Vessel		Vessel
Date	201–250	3rd–4th cent		1st–5th cent		1st–7th cent
<i>n</i>	12	5	8	10		480
SiO ₂	70.3	<i>n.a.</i>	<i>n.a.</i>	69.4		69.1
Al ₂ O ₃	2.59	1.81	2.37	2.67		2.11
Fe ₂ O ₃	0.20	0.48	0.40	0.42		0.55
TiO ₂	0.07	0.09	0.06	0.07		0.09
MnO	1.39	1.14	0.92	1.49		0.51
Sb ₂ O ₃	0.03	0.07	0.02	<0.04		0.36
Na ₂ O	15.2	18.5	18.3	15.5		17.9
K ₂ O	0.51	0.29	0.61	0.55		0.73
MgO	0.57	0.67	0.41	0.53		0.75
CaO	7.83	5.77	8.02	8.09		6.5
Cl	1.18	<i>n.a.</i>	<i>n.a.</i>	1.12		<i>n.a.</i>
P ₂ O ₅	0.14	0.03	0.06	0.09		<i>n.a.</i>
SO ₃	0.14	<i>n.a.</i>	<i>n.a.</i>	0.17		<i>n.a.</i>

throughout the Roman Empire, from Italy to the British Isles as well as in the regions in between, the same HIMT glass was accessible.

Looking for chemical composition similarity can help defining batches or single objects. Two samples can be considered analytically identical if their chemical composition differences are within experimental error, i.e. two standard deviations of the measurements of Corning A and NIST 612 as unknown samples to verify accuracy and precision (Schibille et al. 2016a; Freestone et al. 2006). In Appendix B, ten sets of analytically identical samples were identified. Based on typological comparisons, the two glasses from set 1 (OU3_5 and OU1_7) have to be assigned to two different objects, whereas the two fragments of set 2 (OU4_4 and OU8_10) seem to originate from one single object though it cannot be fully excluded that they could be attributed to two identical objects. The same goes for the five fragments from set 3 (OU8_6, OU5_6, OU7_8, OU8_1 and OU7_6) that are likely to come from one single object. For the three samples from set 4 (OU9_11, OU7_10 and OU6_7), it is not possible to ascertain whether the fragments come from one single or various identical objects, although the three fragments come from contemporary windowpanes. Six further couples of glasses fragments from the end of the third to the beginning of the fourth century AD resemble each other very closely so that they can be put together in six further sets even though their chemical composition difference does not fall within two standard deviations for all elements. The glasses are OU6_5 and OU6_1 for set 5; OU7_7 and OU6_10 for set 6; OU5_4 and OU5_3 for set 7; OU6_8 and OU8_9 of set 8; OU3_2 and OU9_8 for set 9; OU8_3 and OU9_6 for set 10. In each of these sets, the fragments can be attributed to the same typology and could thus come from the same objects. As highlighted by Freestone et al. (2006), the important archaeological implication of the recognition of batches is that it allows us to identify punctuated acquisition of glass. In this case, only the two samples from set 1 have a significance regarding glass supply as they do not represent fragments of one single object. The limited number of batches here thus suggests many supply waves at Oudenburg, which corroborates the presence of five different building phases. On the contrary, more batches of glass fragments coming from different objects have been identified at other sites. The difference with Oudenburg is that those glass sets such as in Byzantine glass weights from the British Museum (Schibille et al. 2016a), a rural Romano-British settlement (Freestone et al. 2006) or the early Christian basilica of Maroni Petrera (Cosyns and Ceglia 2018) were either tightly-defined contexts or specific productions.

Yet, such conclusions should be drawn with care as there can be some variability in the chemical composition of glass batches. Indeed, if two pieces of glass have chemical compositions within one standard deviation, it is very likely that they stem from the same batch but not necessarily vice versa. As

discussed by Freestone et al. (2006), glass from one single batch can be inhomogeneous in its chemical composition not only due to the variability that there can be in a primary glass furnace (Freestone et al. 2000) but also to the possible inhomogeneities in crucibles during secondary production (Paynter 2008). It is thus possible that more glasses would have been produced from the same batch despite inhomogeneities.

Relation between glass provenance and decolourants

Silica sources can be differentiated based on TiO_2 , SiO_2 and Al_2O_3 as Egyptian sands tend to have higher TiO_2 and lower Al_2O_3 than Syro-Palestinian coastal sands (Brems and Degryse 2014; Degryse and Shortland 2009; Freestone 2005b; Gliozzo 2017; Picon and Vichy 2003; Schibille et al. 2016b). A biplot of $\text{TiO}_2/\text{Al}_2\text{O}_3$ versus $\text{Al}_2\text{O}_3/\text{SiO}_2$ (Fig. 6) is particularly useful to identify the primary production of the glass because TiO_2 comes from the heavy minerals present in the sand source while Al_2O_3 comes from feldspar and reflects the mineralogical purity of the sand (Schibille et al. 2016a, b). Data taken from the literature was also added on Fig. 6 so as to compare the provenance of glass from Oudenburg to raw glass containing either manganese or antimony as the only decolouring agent (Gratuze forthcoming) as well as HIMT glass from Cyprus (Ceglia et al. 2017) analysed in the same conditions, with the same technique.

Mn-a group is the only one which has a chemical signature similar to glass from the Levant, whereas the glass groups Sb only, HIMT and Mn-b as well as the glass containing no decolouring agent resembles Egyptian glass. This can be put in direct relation with various Roman sites where antimony-decoloured and HIMT glass were also considered to be of Egyptian origin, while the manganese-decoloured glass was generally attributed to a Levantine provenance (Gliozzo 2017; Nenna 2014; Schibille et al. 2016b). The provenance and supply patterns at Oudenburg would thus be the same as in France (Gratuze n.d.), Cyprus (Ceglia et al. 2017), Croatia (Coutinho et al. 2017), Turkey (Rehren et al. 2015) and Italy (Gallo et al. 2013; Maltoni et al. 2016; Silvestri et al. 2008). In the north-western provinces (Tienen and Maastricht), i.e. geographically closer to Oudenburg, it has been shown through isotopic analyses that the glass could come either from the Levant, from Egypt or even from Italy (Degryse and Schneider 2008). Furthermore, considering that Mn-a group is of Levantine origin goes well with the slightly lower concentration of soda compared to the other groups. As it is likely that natron was imported from Wadi Natrun in Egypt (Shortland et al. 2011), it is legitimate to assume that it was less available in the Levant compared to Egypt (Freestone et al. 2015). Within the general group of Mn-decoloured glass, Mn-a is similar to Levantine glass, whereas Mn-b is more likely to have an Egyptian provenance. This provides a very good example that

groups based on the presence of decolouring agents should not be considered as provenance groups but rather ‘technological groups’ as highlighted by Gliozzo (2017).

Although it is generally considered that Mn-decoloured glass prior to the appearance of HIMT was produced in the Levant, it cannot be fully excluded that certain Egyptian primary workshops were also producing Mn-decoloured glass. Indeed, excavations have found that glass decoloured with manganese was also present in Egypt (Nenna et al. 2003). Moreover, as discussed by Foster and Jackson (2010) regarding glass from Romano-British sites, there can be different manganese subgroups within one assemblage and the diversity of glass production centres is vaster for decoloured than for naturally coloured glass.

Figure 6 not only demonstrates a higher frequency of glass likely to have an Egyptian origin compared to the Levant, but also that the mixed glasses plot closer to the use of Egyptian glass. This can be merely ascribed to the larger use of Egyptian material rather than Levantine when the glasses were mixed in secondary workshops in the northwestern provinces of the Roman Empire. It can also be observed clearly that HIMT glass must come from a different sand than Sb-decoloured glass, although they come from Egypt. HIMT glass being a group clustering apart from the others is obviously based on all the different oxides and confirms that this group corresponds to a technological and provenance change (Freestone et al. 2005).

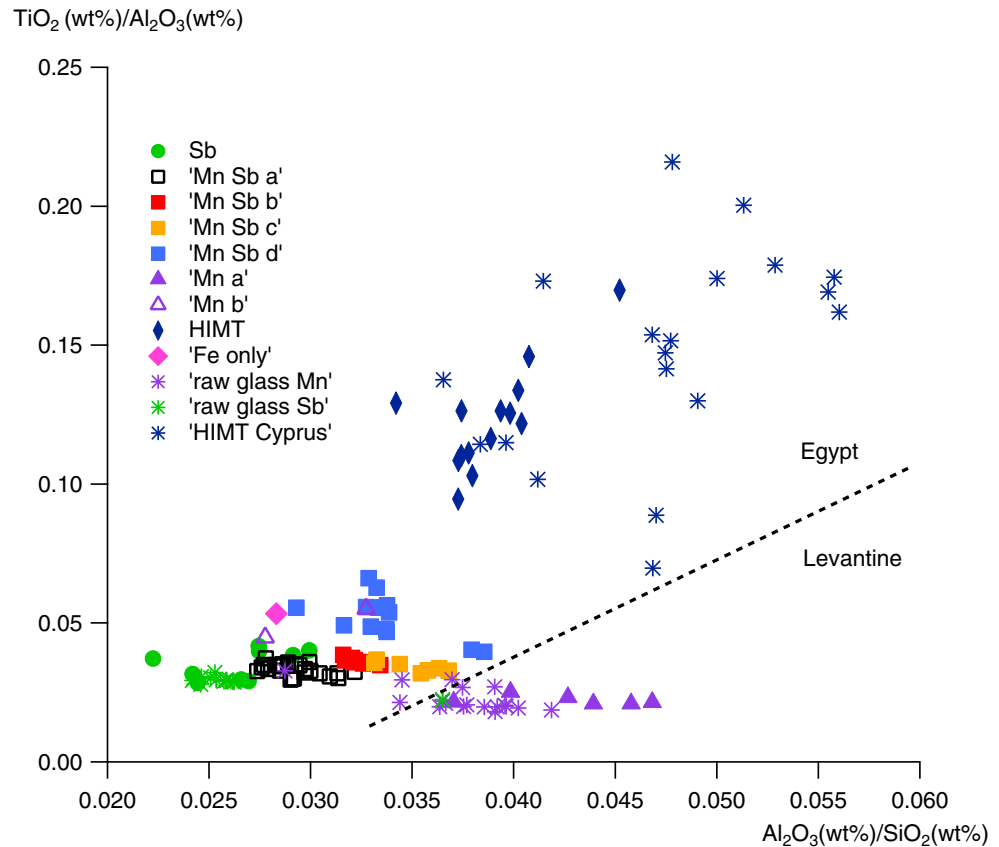
The provenance differences showing that Mn-a and Mn-b were prepared using different sands and the connection between HIMT and Mn-b can be ascertained with other minor oxides and trace elements (Brems et al. 2015; Schibille et al. 2016a) such as titanium and niobium whose correlation is characteristic of a specific geology (Ceglia et al. 2017).

Although there is no direct correlation between decolouring agents and provenance, it is interesting to compare them to observe some general trends. While there is a correlation between iron oxide and the decolouring agent in the glass attributed to an Egyptian provenance (i.e. Sb and HIMT), this is not the case in the glass from the Levant where the amounts of manganese added is not correlated to the amount of iron already present in the glass. This would imply different choices in the production: while in Egypt just enough of decolouring agent would have been added to compensate for iron, this would not have been the case in the Levant, where an excess of manganese was added.

Relation between typo-chronology and decolourants

In British and Italian glass collections, Sb-glass prevails in the early stage, peaks in the second to third century AD and declines as from the fourth century AD being gradually replaced by Mn-glass (Foster et al. 2010; Maltoni et al. 2016; Paynter and Jackson 2018; Sainsbury 2017). Even though the early

Fig. 6 Bi-plot of the Oudenburg glass showing the ratio of TiO_2/Al_2O_3 as a function of the ratio of Al_2O_3/SiO_2 . Data from the manganese or antimony decolourized raw glass from shipwrecks and glass workshops (Gratuze forthcoming) and the HIMT glass from Cyprus (Ceglia et al. 2015) is also showed as a comparison. The dashed line separates the origin of the primary production regions as proposed by Schibille et al. (2016a, b)



presence of Mn-glass should not be fully excluded, for example, Mn-decoloured glass has been found at other earlier sites like in Aalter (Belgium) (Van Der Linden et al. 2009), it became more and more common in later periods, gradually replaced Sb-glass and culminated in the late Roman period, being followed by HIMT glass. The chronology in the use of Mn-glass in Oudenburg corroborates this general trend as it only starts to become common around the late second century AD. An interesting point is that pristine non-recycled Sb-decoloured glass is present throughout all the periods from the pre-fort phase to the fifth fort phase (i.e. mid of the first century AD to the beginning of the fifth century). Its presence in later periods of the fort indicates that either some glass vessels were kept without being re-worked or that there was some minor antimony glass production even in later periods.

In all four larger time-periods, the most represented group is Mn-Sb (above 50%). This implies that both Mn- and Sb-decoloured glass must have been simultaneously available at the production sites to be mixed together. Going more into detail, the group containing the highest amounts of manganese (Mn-Sb-d) is present only as from the end of the third century AD, whereas the group containing the highest amounts of antimony (Mn-Sb-a) is not present after the end of the third century AD. This can be put directly in relation with the chronological use of antimony and manganese in glass. Both types of decoloured glass were thus available and mixed together, but their proportions evolved towards the prevalence of Mn-glass as previously mentioned.

In Oudenburg, as in other contemporary sites, HIMT appears from the beginning of the fourth century onwards and immediately gains a large importance in the glass consumption of the site (26% in the period end of third—beginning fourth century). Even though HIMT rapidly occupied a significant proportion of the glass consumption, most of the sampled glass falls within the Mn-Sb group. This is slightly different from fourth century AD Roman-British sites where HIMT overwhelms glass assemblages (Foster and Jackson 2009). This could be due to the fact that the material studied by Foster and Jackson is slightly later or to geographical differences.

A relation between provenance, chronology and the site's history is particularly visible in the case of Mn-decoloured glass consumption. The Levantine Mn-a glass was present at the site already from the end of the second century while the Egyptian Mn-b glass started being used only one century later at the same time as HIMT from Egypt came in. This supports the idea that early Mn-decoloured glass is Levantine, while later Mn-decoloured glass could come from both region. Such patterns are comparable to later sites in Britain (Foster and Jackson 2009), Italy (Maltoni et al. 2016) and Cyprus (Ceglia et al. 2015), where the Levantine I glass group with a provenance from the Levant appeared from the fifth century AD onwards. However, the already limited supply of glass

from the Levant to the site of Oudenburg came to an end and was totally replaced by glass from Egypt around the fourth century. This coincides with the reoccupation of the fort at Oudenburg by fresh military troops in the Constantinian era (Vanhoutte 2007) and implying the arrival of new loads of glass materials. At the time of this resettlement, the Egyptian provenance of the glass appears to be dominating the market in military networks of this region. As military camps were introducing glass in the region where they were installed and their network contributed to the general trade routes (Stern 1999), it could thus be hypothesised that the glass supply in the continental northwestern provinces increasingly favoured Egyptian production to the detriment of the Levantine one.

Generally, trends in the use of manganese or antimony in glass seem to be related to chronology rather than to the quality of the object (Gliozzo 2017). Yet, a relation between glass function and decolourant can be noticed here. Sb-glass groups contain only vessels, whereas windowpanes are either decolourised using manganese or recycled manganese and antimony glass. This is in agreement with the fact that Sb-glass was rather reserved for high-status ware such as facet-cut beakers (Baxter et al. 1995; Foster et al. 2010; Paynter 2006). Until the late third century, glass with high contents of manganese seem to have been reserved to produce bottles and window glass, whereas in later periods, tableware of higher quality was produced using HIMT. Such relations between glass function and chemical composition have also been noted in other cases such as in the glass assemblages of Romano-British towns (Baxter et al. 2005) or in the Iulia Felix, an Italian shipwreck found in the Italian area of the Adriatic Sea (Silvestri et al. 2008).

Following on Gliozzo's advice (2017) to compare not only glass function but also typology with chemical composition, we have found only some limited common features. Like in Romano-British assemblages (Jackson 2005; Jackson et al. 2015), fragments corresponding to late second-early third century cylindrical cups with double annular footstand (Isings 85b) are either purely Sb-glass or Mn-Sb-glass with the highest amounts of antimony (Mn-Sb-a). This can be related to the shipload of the wreck Ouest-Embiez 1 at the French Cotes d'Azur (Fontaine and Foy 2007) containing glass vessels of a purely Sb-decolourized composition. Almost all the HIMT glass of Oudenburg corresponds to the later hemispherical cups with slightly everted rim (type Isings 96/AR60/Trier 40). This type has also been found with an HIMT composition in Egypt (Rosenow and Rehren 2014), at Romano-British sites (Jackson et al. 2015) and in Italy (Gliozzo et al. 2016; Maltoni et al. 2016), because Isings 96 came into fashion at the same time as HIMT started being used. At Oudenburg, this long-lasting and largely represented typological group also contains glass belonging to the Sb or the Mn-Sb-a, the mixed group with the highest concentrations of antimony.

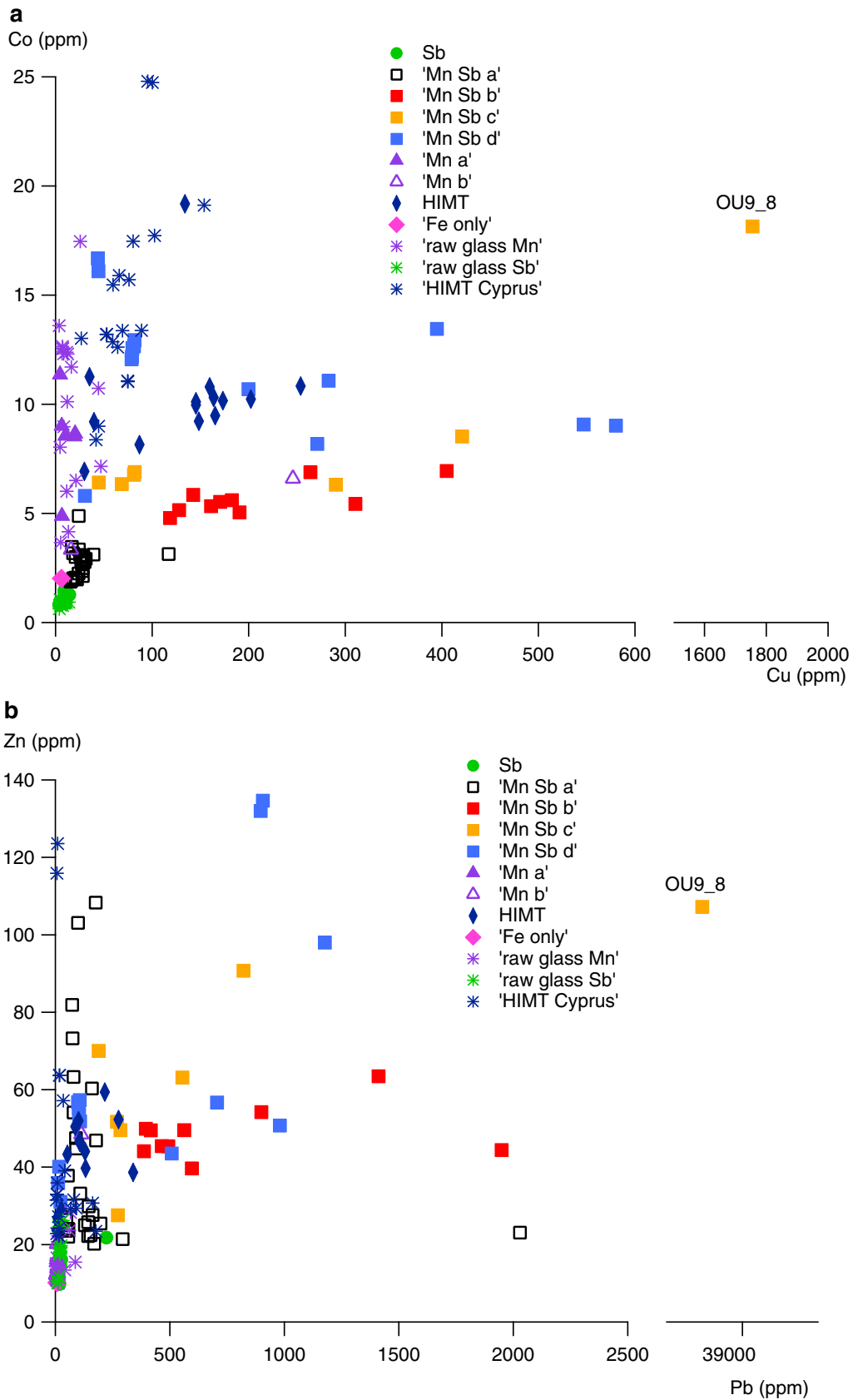


Fig. 7 a Co content as a function of Cu content. b Zn content as a function of Pb content. Antimony and manganese glass have the lowest contents in those trace elements characteristic of recycling

The effect of glass recycling on chemical composition mixing and homogeneity

As a second step in the life-cycle of Roman glass, recycling, remelting and mixing was a common practice. This process, probably introduced in the Flavian period (69–96 AD), became geographically widespread and revolutionised glass production technologies (Degryse et al. 2006). Evidence for recycling comes from late first-early second century AD written sources (e.g. Martial, Statius and Juvenal) and the cargo of shipwrecks such as the late second century AD Iulia Felix wreck yielding at least 140 kg of broken glass fragments, traded in view of recycling (Silvestri 2008; Silvestri et al. 2008). It can also be attested via archaeometric studies and has been the subject of many analyses and discussions in the recent archaeometric literature (Ceglia et al. 2017; Freestone 2015; Jackson et al. 2015; Paynter and Jackson 2018).

One indicator of glass recycling is the simultaneous presence of antimony and manganese oxides as they do not require to be present together to fulfil their decolouring role (Silvestri et al. 2008). Glass containing both manganese and antimony is widespread and generally considered to be due to glass recycling so that different glass groups were mixed together.

Trace elements, in particular Co, Cu, Zn and Pb used as glass additives and minor oxides such as P_2O_5 and K_2O coming from prolonged contact with ashes that float in the firing chamber are useful to detect recycling (Freestone et al. 2002; Paynter 2008). As can be seen in Fig. 7 and Appendix B, Sb-glass is the group that contains the smallest amounts of recycling markers. Only one sample from the Sb group contains above 100 ppm of Pb. Even though this could attest mixing with a glass containing higher amounts of Pb, it could also suggest the use of another antimony ore, richer in lead such as galena or bindheimite, whereas other glasses would have been produced using stibnite (Biek and Bayley 1979; Jackson 2005). Manganese-containing glasses have higher trace elements concentrations than Sb-glass, thus suggesting that they could have been recycled or that the raw materials used to produce Mn-glass were less pure. This goes well with the chronology as Mn-glass in Oudenburg arrives later, at a time where more recycling occurred in the northwestern provinces of the Roman Empire (Jackson and Paynter 2015). The groups Mn-a, Mn-b and Sb have similar trace elements concentrations compared to the decoloured glass from the Iulia Felix containing either manganese (C12) or antimony (C11/1) (Silvestri 2008).

Glasses with both antimony and manganese constitute the group which contains by far the highest concentrations of recycling markers because they were clearly recycled. The trace elements concentrations in the Mn-Sb group studied here are much higher than those of colourless Mn-Sb group of the Iulia Felix shipwreck which has a comparable chemical composition otherwise (Silvestri et al. 2008). These concentrations

Fig. 8 a Ratio of Y(ppm)/Zr(ppm) as a function of ratio Ce(ppm)/Zr(ppm). **b** Ratio Fe_2O_3 (wt%)/ TiO_2 (wt%) as a function of Fe_2O_3 (wt%)/ Al_2O_3 (wt%). Data from the literature using the same technique was added (Ceglia et al. 2015; Gratuze forthcoming). These ratios suggest that Mn-Sb-d was obtained from a mixture between antimony and HIMT glass which appear to be the two endpoints

are also higher than those of the group Ic1b of the Iulia Felix with a calculated recycling index of 54% (i.e. Ic1b glass includes 54% of recycled antimony glass) (Silvestri 2008). Thus, the mixed Sb-Mn-glass from Oudenburg must originate from a production with extensive recycling.

It is generally considered that a glass containing more than 100 ppm of Cu, Co, Zn or Pb has been recycled (Freestone et al. 2002; Gliozzo 2017; Silvestri et al. 2005), although more recently even lower amounts have been proposed (Ceglia et al. 2017). In any case, these elements allow for a rough estimation of the extent of recycling and the purity of the adopted raw materials. If the content of Cu, Co, Zn or Pb is above 100 ppm, then the glass was very likely recycled but not necessarily vice versa. In the set of samples studied here, over 70% of both groups Mn-Sb and HIMT contain glass where all those trace elements are above the threshold value (100 ppm), while this is the case for only around 10% of both the Mn only and Sb only groups (Fig. 7).

An interesting point is to look at which glass groups could have been mixed together. To do this, trace elements ratios such as Y/Zr and Ce/Zr (Fig. 8a) as introduced by Cholakova et al. (2016) as well as ratios of iron, aluminium and titanium oxides (Fig. 8b) are very useful. In both Fig. 8a, b, it can be observed that the mixed glass groups Mn-Sb-a, Mn-Sb-b and Mn-Sb-c could have been obtained by a mixture of manganese glass (either Mn-a or Mn-b) with antimony glass. The production of Mn-Sb-d, instead, could not be the outcome of simply mixing manganese glass with antimony glass but is the result of mixing antimony-decoloured glass with HIMT. Indeed, Mn-Sb-d has lower Y/Zr and Ce/Zr than manganese and antimony glass so that Mn-Sb-d would have required a source of glass with much lower ratios (such as HIMT).

HIMT glass seems to appear at the earliest from the early fourth century AD onwards which fits well with the dating of Mn-Sb-d which is also present during the same time period. From a chronological point of view, it is thus also likely that Mn-Sb-d could represent mixtures of antimony glass and HIMT. The other mixed glass groups are dated to earlier phases and could thus not have been prepared by mixing antimony glass with HIMT. To our knowledge, it is the first time that a glass mixture between antimony and HIMT is observed, which would corroborate the concept of ‘great big melting pot’ (Jackson et al. 2015) that points to an increased recycling and mixing of glass towards the later Roman periods.

If glass fragments from different compositions are mixed, the question of homogeneity is crucial to see how well glass

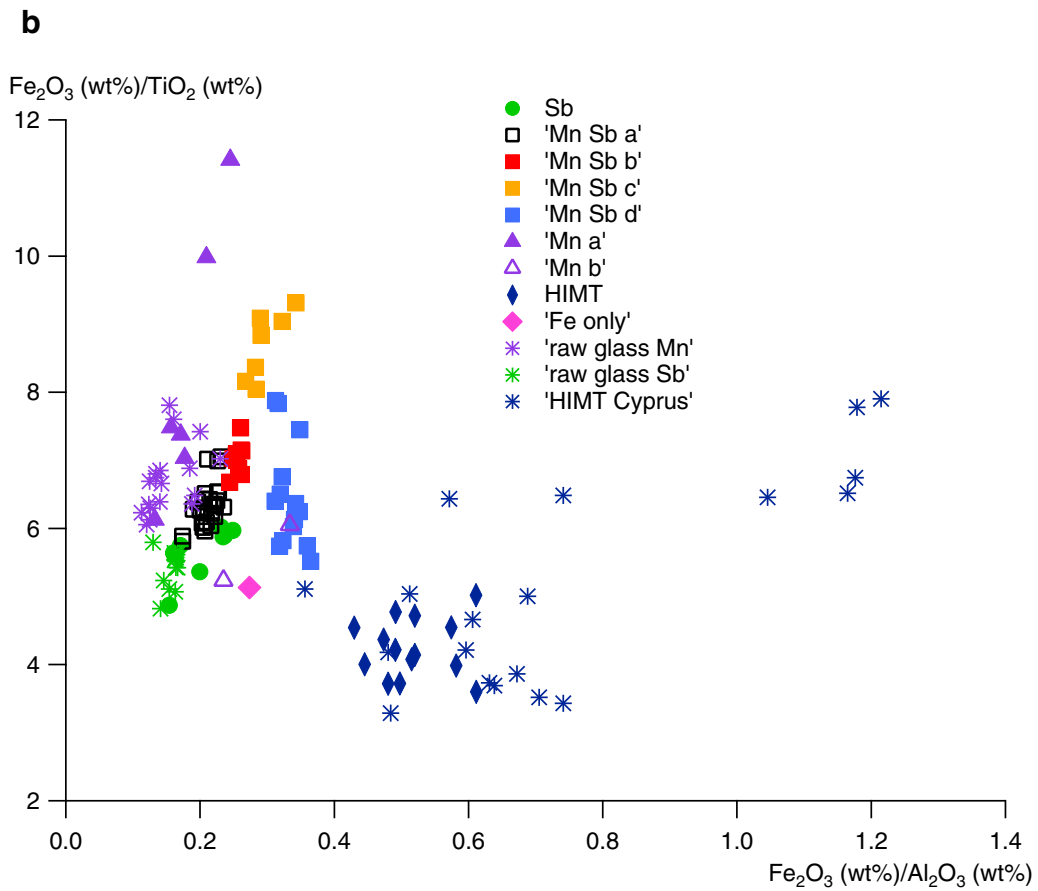
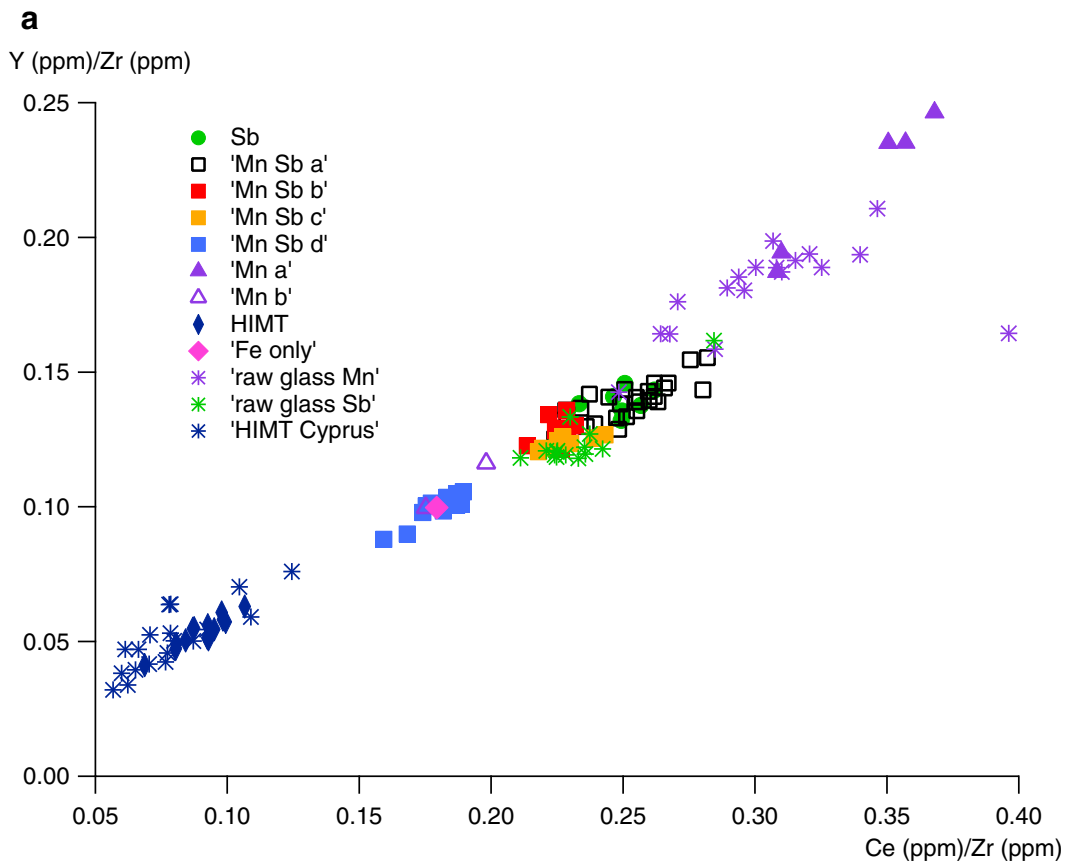
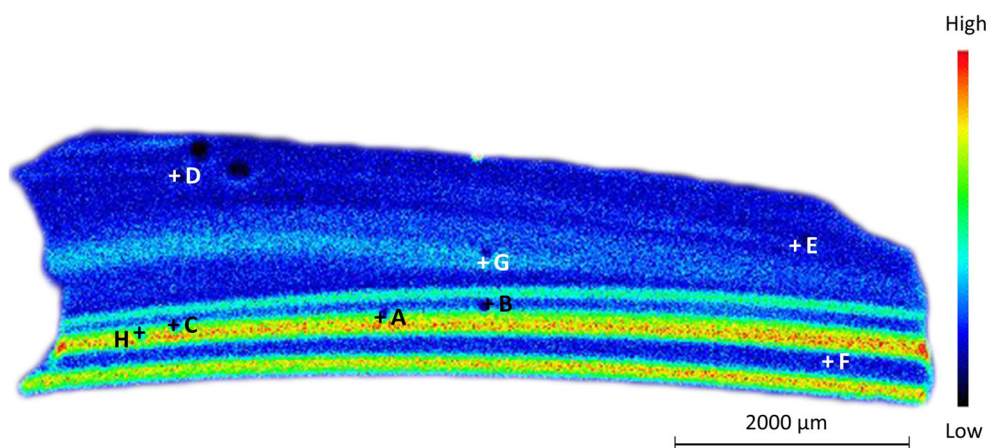


Fig. 9 μ XRF manganese heatmap of OU5_1 showing stripes containing different decolouring agents: points A, H and C contain high amounts of Mn, whereas other points contain both Sb and Mn in equivalent proportions. The concentrations of MnO and Sb_2O_3 given below come from LA-ICP-MS



	A	B	C	D	E	F	G	H
MnO (wt%)	0.83	0.44	0.87	0.26	0.26	0.26	0.38	0.97
Sb_2O_3 (wt%)	0.22	0.36	0.20	0.39	0.41	0.41	0.32	0.16

cullet was mixed. Thanks to μ XRF mapping, it was possible to observe that none of the glass containing only manganese or antimony was heterogeneous. This confirms the hypothesis that decolouring would have been carried out in primary workshops (Freestone 2015; Gliozzo 2017). This argument is based on the general homogeneity of the large quantities of antimony-decoloured glass found in shipwrecks such as *Ouest-Embiez I* (Fontaine et al. 2007; Foy et al. 2004) and on the presence of decolouring agents found in Egyptian primary glass (Nenna et al. 2003).

However, the mixture of Sb- and Mn-decoloured glass can be heterogeneous. In one glass containing both antimony and manganese (OU5_1), stripes of different chemical composition have been detected using μ XRF mapping: some containing high amounts of manganese and some equal quantities of manganese and antimony (Fig. 9). On Fig. 9, the concentration in MnO and Sb_2O_3 determined by LA-ICP-MS is also given. This fragment comes from a free-blown hemispherical cup (type Isings 96) typologically dated to the second half of the third up to the first half of the fourth century AD and found in the latest layers of the fort. It could be attributed to Mn-Sb-a based on the iron concentration, but heterogeneities in its chemical composition make it too difficult to attribute the fragment to one group.

Some other samples of Mn-Sb glass show similar heterogeneities (OU9_1, OU9_8, OU8_4, OU5_2, OU4_10, OU2_5, OU2_6). In these samples, bands appear but the variability in chemical composition is not as strong as for OU5_1 so that these samples can still be considered to belong to the various subgroups. It should also be noted that OU9_8 is the

sample with the highest trace elements characteristic of recycling (Fig. 5).

These bands are similar to the inhomogeneous samples from San Giusto (Gliozzo et al. 2017) in which bands of lead were observed. However, in the Oudenburg samples, the bands are related to variations in the content of the decolouring agents antimony and manganese. This evidence points to the inhomogeneous mixing of different glass compositions and implies that the glass could have been produced by the mixing of partially melted cullet. Due to glass blowing, the zones with different chemical compositions would be stretched into the stripes appearing in Fig. 9. This is a direct proof of ancient glass remixing and recycling techniques, which have been discussed in the literature for British sites (Jackson and Paynter 2015; Paynter and Jackson 2016). It is difficult to determine whether these bands would have been the result of inhomogeneous mixing at a primary or secondary stage even though it has been suggested by Nenna (2003) and Freestone (2015) that mixed Mn-Sb glass could already have been produced at a primary stage.

Conclusions

Based on the chemical composition and the detailed chronology of 90 glass samples from the site of Oudenburg in Belgium analysed by means of LA-ICP-MS, it was possible to explore correlations between glass recipes, provenance and dating for this dataset. To approach this dataset, three steps were followed with the aim of dividing the glass set into coherent subgroups. Groups were first determined based on the

presence of the decolouring agents antimony and/or manganese. This was then refined by taking into consideration the chemical composition of the glass matrix as well as some markers characteristic of the sands used. PCA was used to confirm the groups that were formed but also showed the high complexity of the data.

The first objective of this study was to complement the landscape of Roman glass analysis in Europe and fill a gap pointed out by (Gliozzo 2016). Oudenburg represents a relatively long-lived site in the northwestern provinces along continental trade routes between the Rhine region and the British islands. At Oudenburg, the same glass groups as in other sites are present, the main difference being that this site relied mostly on antimony-manganese containing glass recycled glass and to a lesser extent on HIMT glass in the later periods. It is possible that maritime routes could have supplied the British Isles with glass from Egypt, whereas this network would have been limited in Oudenburg (Foster et al. 2010). Even though Bocholtz is geographically close, the chemical composition repartition of the glass is quite different. This can be explained by the status and function of the site: while Bocholtz is a high-status burial, Oudenburg is a Roman fort with very different needs and trade networks.

Secondly, diachronic shifts in the use of different decolouring agents were observed and these can be related to certain glass types and provenances in the glasses analysed here. Sb-decoloured glass was present through all the periods of the fort and is akin to Egyptian glass. This group is found only in vessels and shows the least recycling, thus suggesting that antimony would have been reserved for higher status glass made out of pristine sands. Mn-glass gradually replaced Sb-glass. Amongst the glass decoloured solely with manganese, two subgroups could be identified: Mn-a with a chemical signature characteristic of a Levantine origin and Mn-b similar to Egyptian glass. This latter subgroup is related to the appearance of the Egyptian HIMT glass as from the fourth century AD. The majority of the Oudenburg samples contain both manganese and antimony. The simultaneous presence of antimony and manganese can be attributed to cullet recycling due to the elevated trace elements contents and to the inhomogeneous presence of both decolouring elements in one sample.

Thirdly, this dataset allowed for the observation of interesting mixing patterns. It was noted for the first time that Sb-glass could have been mixed with HIMT based on minor and trace elements and heterogeneous mixing of Mn- and Sb-glass could be observed.

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