

Glass and alchemy in early modern Europe: An analytical study of glassware from the Oberstockstall laboratory in Austria

Umberto Veronesi^{*}, Marcos Martinón-Torres

Abstract: Glass distillation equipment from an early modern alchemical laboratory was analyzed for its technology of manufacture and potential origin. Chemical data show that the assemblage can be divided into sodium-rich, colorless distillation vessels made with glass from Venice or its European imitation, and potassium-rich dark brown non-specialized forms produced within the technological tradition of forest glass typical for central and north-western Europe. These results complete our understanding of the supply of technical apparatus at one of the best-preserved alchemical laboratories and highlight an early awareness of the need of high quality instruments to guarantee the successful outcome of specialized chemical operations. This study demonstrates the potential of archaeological science to inform historical research around the practice of early chemistry and the development of modern science.

The scientific and technological advancements of the early modern period were the outcome of great experimenters as well as of ingenious craftsmen who aimed to find practical solutions to their questions and needs by looking at phenomena or manipulating substances. A crucial yet often overlooked role in this process was played by the wide array of specialized apparatus and technical equipment employed in laboratories, which ultimately made these efforts possible. Among those, glass is a key material that enabled the production of new ideas. For example, Galileo Galilei was very concerned with finding the right material for his telescope lenses,¹ as was Newton a few decades later;² in the same vein, Robert Boyle's mechanical philosophy, whose impact reverberates in modern analytical chemistry, was based on a theory of matter formulated after observing metals dissolve and re-precipitate within glass distillation vessels.³ Indeed, distillation was a central procedure in early chemistry as testified by numerous treatises,⁴ and as a chemical reaction with specialized equipment it crossed the boundaries of disciplines, from the production of alcoholic beverages and medicines to

^{*}U. Veronesi MSc, Prof. M. Martinón-Torres

UCL Institute of Archaeology

³¹⁻³⁴ Gordon Square, WC1H 0PY, London UK

E-mail: umberto.veronesi.13@ucl.ac.uk

metallurgy and up to the works on metallic transmutation. As a kind of artificial stone with peculiar properties, glass attracted the attention of craftspeople and natural philosophers from prehistoric times; at the same time, as the need of transparent, strong and stable glassware of peculiar shapes for laboratory reactions is likely to have triggered important developments in glass technology.⁵

The scientific analysis of archaeological remains of laboratories has demonstrated great potential to further our knowledge of early modern science and technology. In particular, it is possible not only to understand the provision, manufacture and development of specialized equipment, but also to infer the reactions carried out based on the residues within it. However, while there have been several studies concerned with ceramic laboratory equipment such as crucibles or cupels, which successfully combined scientific analysis, history and archaeology,⁶ chemical glassware has rarely been the subject of similar scholarly attention. Against this background, the material recovered from the 16th century laboratory of Oberstockstall in Lower Austria provides a very fitting case study. The assemblage includes hundreds of ceramic instruments, but also numerous glass finds: a large distillation column, a still head, and several receivers, vials and other vessels of transparent glass, in addition to several bottles of dark brown or black color (figure 1).⁷ Previous analytical work has shown that one of the main processes at this laboratory involved the assay of argentiferous minerals, and demonstrated the high technical standard of the crucibles employed, which were obtained from specialized suppliers and displayed excellent material properties and standardization.⁸ Distillation was routinely carried out by assayers, for example to obtain mineral acids in order separate gold from silver that came naturally alloyed and early modern manuals give full description of the process.⁹ Through the scientific analysis of glassware recovered at Oberstockstall, this paper seeks to complete our understanding of the supply of technical instruments to the laboratory. Were specialized glasses specifically chosen for the purpose? What type of glass was used and was it imported from the best manufacturers? Answering these questions will shed light on the role of technical glassware within the laboratory and, more broadly, on the role glass as a scientific material in the development of modern science.

Pre-modern glass was made by melting silica (typically quartz sand or pebbles) with a mineral or vegetable flux; lime (often present as an impurity in the raw materials) acted as a stabilizer, and other additives or colorants could be included to adjust the final appearance. Two major glassmaking traditions were predominant in 16th century Europe. First, since the 9th century, glass in central and north-western Europe had been produced in glasshouses located in vast forested areas where the wood needed as fuel and as a flux (the latter in the form of ashes), was abundant and readily available. This glass type has a specific chemical signature that could vary according to the plants used, but whose main characteristics are high levels of potassium and/or calcium together with other elements brought by the flux such as magnesium, manganese and phosphorous.¹⁰ These and other impurities

in the raw materials imparted characteristic dark green or brown colors to the glass, which was used for domestic items as well as for windows to glaze houses and churches. On the other hand, in southern Europe a technology prevailed which made use of halophytic plant ashes, producing a glass with high sodium but lower levels of potassium and calcium.¹¹ Venice had for centuries been the leader in the production of this type of sodic glass and its most famed product, *cristallo*, was exported all over the world. The secret of the Venetian glass industry was the use of extremely pure raw materials, partly imported from the Levant and partly sourced in the region, and of a sophisticated technology of ash purification and mineral additives that allowed craftspeople to obtain perfectly colorless glasses.¹² In the 16th century, some glassmakers left Venice and started to produce glasses imitating the Venetian technology in other European cities. Antwerp was one such production center, as were Amsterdam and London,¹³ but both Venetian and *façon de Venise* glasses have been found at many locations from the Iberian peninsula to the Balkans as a testimony to the great impact Venice had in early modern glass technology.¹⁴

For the present paper, 34 glass fragments recovered from the same pit in Oberstockstall were analyzed for their chemical composition using scanning electron microscopy with energy dispersive Xray spectrometry (SEM-EDS). Based on their shape they can be ascribed to both distillation vessels and more general-purpose domestic forms, but they all relate to the context of the laboratory. The fragments range from colorless through blue-green to dark brown, their thickness varying from as little as 0.3mm to 3.2mm. In general, the distillation vessels appear to have been free-blown, are generally thinner and lack signs of decoration, whereas the dark bottles were blown into moulds and usually show a moulded ribbed decoration (figure 1). Details on the analytical methodology are given in the supporting information.



Figure 1 – A distillation column (left, height 41.8cm) and a bottle with ribbed decoration (right) (photos: Marcos Martinón-Torres).

The results show that the Oberstockstall laboratory contained glassware made within both the potassium-rich and the sodium-rich technological traditions mentioned above (see the Supporting Information for details), and there is a close correspondence between composition and typology. Forest glass is present in non-specialized forms, like bottles and flat discs, and comprises all the dark brown glasses as well as the naturally colored blue-green ones. Conversely, those glasses which appear to have been decolorised through the addition of manganese are all of the sodium-rich type. This group only includes thin-walled fragments of globular shape ascribable to distillation vessels or vials, even though the size of the fragments does not always allow a precise form identification. The levels of the oxides of sodium and potassium in this type of glass shows a certain degree of variability, with a group of samples clustering tightly and others appearing more scattered (figure 2). This might be the indication of differences in raw materials used, technological choices or of the presence of more than one supplier of glass objects. Comparison of the Oberstockstall data with contemporary soda-ash glasses indicates that the one specimen with higher soda (OB14) is likely to be a Venetian import of the type called "vitrum blanchum".¹⁵ Similar compositions outside Venice are reported from Antwerp.¹⁶ The rest of the assemblage is likely to constitute a Venetian imitation, a type which tends to show higher levels of potash than genuine Venetian products. These chemical differences have been explained as resulting from the use of a different types of soda-rich ashes and/or the dilution with potassium-rich forest ashes or glass cullet.¹⁷ This last possibility is suggested by the somewhat lower sodium and higher calcium of the sodium-rich glasses from Oberstockstall when compared to other facon de Venise examples (figure 2). This difference might arise from the dilution through clippings of forest glass, which would be responsible for the increase in potassium and calcium seen.



Figure 2 – Scatterplots comparing the concentrations of alkalis between Oberstockstall and contemporary European glasses. Data for Venetian and Venetian imitation glass was taken from Ref. 13 while data for Central European glass is from Ref. 14b.

Regardless of where exactly the laboratory practitioners of Oberstockstall sourced their glassware, it is worth noting that a special type seems to have been chosen specifically for the distillation vessels and different from the rest of the objects with which the space was equipped. As

mentioned above, sodium-rich glasses were less common in central and north-western Europe than their potassium-rich counterparts and normally they only occur as fine tableware and expensive ornamental items, whether imported or not. Any occurrence of such glasses can therefore carry a particular significance. This becomes clear in the case of Oberstockstall, where the special importance of the distillation vessels is further stressed by the rest of the assemblage being made of a different and more common glass type. One reason for the choice of higher quality glass may have been the need for better performance since the containers would be subjected to repeated cycles of distillation and heating while exposed to aggressive reagents, meaning they had to withstand remarkable thermal as well as chemical stress. In this sense a sodium-rich glass, much richer in silica and therefore more resistant to decay than a potassium-rich one, would certainly do a better job. This preoccupation shows through the pages of technical writings and other early modern documents: Daniel Sennert, a prominent 17th century physician, complains that the war is preventing him from obtaining laboratory glassware from his favorite suppliers, thus having to work with a lower quality material that constantly shatters.¹⁸ Hieronymus Brunschwig and Johann Rudolf Glauber both give advice on the subject in their treatises, the former explicitly mentioning Venetian or Bohemian products,¹⁹ while the latter prescribing a "strong and firme glass" that can better retain the distillation vapors.²⁰ The addition of broken glass waste to make stronger glass is suggested in the Ordinal of Alchemy by Thomas Norton²¹. Whether that was indeed a common practise we do not know, but it was mentioned above that the addition of recycled glass to the batch might be documented in the Oberstockstall assemblage. There is yet another reason, perhaps even more important though less acknowledged in the documents, why Venetian glass may have been preferred. This can be seen in the words of Vannoccio Biringuccio who mentions that cucurbits should be of a glass "as clear and uniform as possible" with no "bubbles and indentations".²² The higher transparency and clarity of Venetian glass allowed to monitor every detail of what was going on inside, and Biringuccio seems indeed to value his glassware for both strength but also clarity. A color change or the formation of particles in the liquid being distilled were vital signs about the reaction's progress ²³ and being able to recognise them could make the difference between success or failure. Forest glass was usually dark, thicker and full of small bubbles, which might have hindered the correct execution of the process.

The growing scholarly attention to early laboratories has challenged the traditionally presumed un-scientific nature of early experiments.²⁴ In particular, precision and reproducibility were key concerns, and chemical operations such as fire assay and distillation relied on exact quantification and therefore greatly stimulated scientific and technological advancements.²⁵ It has been argued that experimental reproducibility was a fundamental reason behind the choice of high-quality crucibles,²⁶ and the case of Oberstockstall suggests that similar concerns may have been behind the choice of

glassware for distillation equipment. The glass was probably sourced from several manufacturers, from Venice itself as well as from centers reproducing Venetian technology in Central Europe. It is possible that a Venetian item could not be told apart from a *Venetian style* one or that in fact no substantial difference was even perceived by the end users. After all, this glass was produced using very similar raw materials by former Venetian glassmakers who, starting in the 16th century, began to leave the island of Murano looking for fortune elsewhere.²⁷ It is not surprising therefore that the beginning of Venetian glass industry's decline coincided with the spread of the secret technology that had been zealously kept for centuries.

By considering early modern scientific developments from the perspective of a laboratory's technical equipment, this paper has exemplified the potential of an approach that integrates history and the scientific analysis of archaeological heritage. The materiality of glass distillation apparatus from the site of Oberstockstall was brought to the fore and used to investigate the rationale behind the supply of technical glassware. It was shown that the challenges set by the specific purpose of the distillation-related objects demanded a glass of special quality, different from the one used for nonspecialized forms. The vessels needed to be resistant to chemical and thermal stresses, as well as clear to allow to visually follow the reactions. Venetian products and their European imitations granted such high standards owing to a finely tuned manufacturing process whose outcome was a clear and strong material particularly apt for special products such as expensive tableware and also, as this paper demonstrates, scientific instruments. Our growing understanding of early laboratory equipment emphasises that innovations in science in general, and in analytical chemistry in particular, from the exact quantification of a mineral composition to Galileo's revolutionary discoveries, were only possible thanks to the high quality of technical equipment that populated laboratories. The archaeology of early chemistry thus offers great potential for a fertile cross-disciplinary dialogue on early modern science.

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Keywords: Alchemy, distillation, glass, laboratory, SEM-EDS

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What sort of operations did alchemists carry out in their laboratories? And what tools did they use? Can answering similar questions help us better understand the development of science and technology in the Renaissance? This paper shows that the information gained through the archaeometric analysis of artefacts coming from laboratories can be a powerful tool to explore how alchemy was practiced in early modern Europe.

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Analytical methodology

Small glass fragments were cut by means of a rotating saw and embedded in epoxy resin blocks, ground and polished down to 1µm following standard procedures prior to undergoing scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS). Analyses were performed in the Wolfson Archaeological Science Laboratories at the UCL Institute of Archaeology using a Hitachi S-3400 machine with an Oxford Instruments X-sight energy dispersive spectrometer. Operating conditions were kept constant throughout the campaign, with accelerating voltage of 20kV and 10mm working distance. Bulk chemical data was obtained on areas of exposed uncorroded glass measuring between 100 and 150 μ m across for 100s. Usually three areas were analyzed, the average calculated and the results normalized to 100 wt% to account for oscillations in beam intensity and to facilitate comparison. To test instrumental accuracy glass certified standards Corning A and D were analyzed several times during the course of the campaign (Tables 1-2). The results indicate satisfactory levels of accuracy for the major elements, with error normally within the 10% mark for Na₂O, MgO, SiO₂, K₂O, CaO, MnO, FeO and Al₂O₃ in concentrations ≥0.5%. For trace elements analysis, a technique with lower detection limit such as LA-ICP-MS could provide data useful to provenance the glass samples more precisely and discriminate between genuine Venetian and imitation products. However, this falls outside of the scope of the present paper.

Corning A	1	2	3	Avg	Recomm.	%error
Na ₂ O	14.5	14.2	14.5	14.4	14.3	0.6
MgO	2.7	2.7	2.6	2.7	2.7	0.2
AI_2O_3	0.8	0.8	0.8	0.8	1.0	25.8
SiO ₂	68.8	69.0	68.7	68.9	66.6	3.3
CI	0.1	0.1	0.1	0.1	0.1	13.5
K ₂ O	2.9	3.0	2.9	2.9	2.9	2.3
CaO	4.9	5.1	4.9	5.0	5.0	1.3
TiO ₂	1.0	1.0	0.9	1.0	0.8	20.5
MnO	1.1	1.0	1.0	1.0	1.0	4.6
FeO	0.9	1.0	1.0	1.0	1.1	10.1
CuO	0.7	0.7	0.9	0.8	1.2	54.8
Sb ₂ O ₃	1.4	1.4	1.4	1.4	1.8	22.1

Table 1. SEM-EDS data on certified standards Corning A. Results are shown as wt% oxides.

Table 2. SEM-EDS data on certified standards Corning D. Results are shown as wt% oxides

Corning D	1	2	3	Avg	Recomm.	%error
Na ₂ O	1.3	1.3	1.3	1.3	1.2	10.9
MgO	4.1	4.1	4.0	4.0	3.9	2.4
AI_2O_3	5.1	5.1	5.1	5.1	5.3	3.6
SiO ₂	57.3	57.3	57.3	57.3	55.2	3.7
P_2O_5	3.8	3.7	3.7	3.7	3.9	5.1
SO ₃	0.2	0.3	0.3	0.3	0.3	7.1
CI	0.2	0.2	0.2	0.2	0.2	21.5
K ₂ O	11.6	11.5	11.5	11.5	11.3	1.9
CaO	14.4	14.4	14.5	14.5	14.8	2.4
TiO ₂	0.5	0.6	0.5	0.6	0.4	45.1
MnO	0.6	0.5	0.6	0.6	0.6	3.5
FeO	0.4	0.5	0.5	0.5	0.5	9.7
CuO	0.1	0.1	0.1	0.1	0.4	70.1
Sb ₂ O ₃	0.2	0.2	0.2	0.2	1.0	81.8
PbO	0.2	0.2	0.2	0.2	0.2	21.1

	lnv.	Turno	Color	No O	MaQ	AL O	80	D O	50	CI	KO	6.0	TiO	MnO	E ₀ O
U	number ¹	туре	COIOI	INa ₂ O	lvigO	Al ₂ O ₃	5102	P ₂ U ₅	503	CI	K 2∪	CaU	HO ₂	WINO	FeO
OB3	704	vial	Colorless	10.8	3.4	1.6	66.1	0.3	0.2	0.6	4.6	11.0	bdl	0.8	0.6
OB5	703	vial	Colorless	10.1	3.2	1.2	66.5	0.2	0.3	0.6	6.1	10.5	bdl	0.8	0.6
OB6	647	beaker	Colorless	11.2	3.4	1.2	66.9	0.2	0.3	0.6	3.9	10.8	bdl	0.8	0.6
OB7	649	beaker	Colorless	11.1	3.4	1.2	66.9	0.2	0.3	0.6	4.0	10.8	bdl	0.9	0.6
OB9	700	vial	Colorless	10.7	3.3	1.4	66.6	0.2	0.2	0.5	4.4	11.2	bdl	0.8	0.6
OB10	640	beaker	Colorless	11.1	3.4	1.3	67.1	0.2	0.3	0.6	3.8	10.7	bdl	0.9	0.6
OB14	N/A	vial	Colorless	12.5	3.5	1.1	67.9	0.2	0.3	0.8	2.2	10.5	bdl	0.6	0.6
OB15	N/A	N/A	Colorless	11.3	3.4	1.3	67.0	0.2	0.3	0.6	3.7	10.6	bdl	0.9	0.6
OB16	N/A	vial spout?	Colorless	11.2	3.5	1.2	67.4	0.2	0.3	0.6	3.7	10.3	bdl	0.9	0.6
OB17	N/A	vial	Colorless	10.5	3.3	1.3	66.6	0.2	0.3	0.6	5.1	10.6	bdl	0.9	0.5
OB20	N/A	vial?	Colorless	11.2	3.3	1.3	66.9	0.2	0.3	0.6	4.0	10.8	bdl	0.9	0.6
OB27	N/A	vial	Colorless	12.1	3.4	1.2	67.4	0.2	0.3	0.8	3.8	9.8	bdl	0.7	0.5
OB28	N/A	vial	Colorless	11.0	3.4	1.3	67.2	0.2	0.2	0.7	4.2	10.4	bdl	0.9	0.6
OB29	N/A	bottle?	Aqua	11.6	3.4	1.2	68.7	0.2	0.2	0.7	4.0	10.9	bdl	0.8	0.6
OB30	N/A	vial	Colorless	11.2	3.4	1.2	67.1	0.2	0.3	0.7	3.8	10.6	bdl	0.9	0.7
OB31	N/A	bottle?	Aqua	11.2	3.4	1.2	67.0	0.2	0.3	0.7	3.8	10.7	bdl	0.9	0.6
OB33	N/A	vial? Bottle?	Aqua	9.2	3.4	1.5	66.0	0.4	0.2	0.5	5.0	12.1	bdl	0.9	0.7
OB1	N/A	N/A	Olive	0.2	3.2	1.3	51.6	0.9	0.3	bdl	21.1	20.3	bdl	0.6	0.5
OB2	N/A	flat glass	Blue-green	2.3	2.7	1.7	62.6	0.8	bdl	0.2	12.3	16.1	bdl	0.8	0.5
OB4	665	flat disc	Brown	0.4	3.0	2.0	49.5	1.2	0.3	bdl	21.1	21.1	bdl	0.8	0.6
OB8	661	flat disc	Olive	0.6	2.6	3.5	50.3	1.0	0.3	bdl	20.9	19.0	0.3	0.4	1.0
OB11	634d	bottle	Brown	0.2	2.9	1.3	52.6	0.9	0.3	bdl	20.8	19.8	bdl	0.8	0.4
OB12	634b	bottle	Olive	0.2	2.8	1.2	52.4	0.9	0.3	bdl	20.9	19.9	bdl	0.9	0.5
OB13	663	flat disc	Olive	0.5	2.6	3.5	50.2	1.0	0.3	bdl	21.0	19.1	0.3	0.4	1.1
OB18	N/A	bottle	Blue-green	0.3	4.1	1.6	51.2	1.2	0.3	bdl	17.6	22.1	0.2	0.8	0.6
OB19	662	flat disc	Olive	0.5	2.9	3.9	48.8	1.4	0.2	bdl	18.2	22.2	0.2	1.0	0.6
OB21	N/A	bottle	Blue-green	1.4	4.5	2.6	48.4	1.6	0.2	bdl	16.7	22.3	0.2	1.0	1.1
OB22	626	bottle	Blue-green	1.4	4.5	2.7	48.9	1.6	bdl	bdl	16.6	22.1	bdl	1.0	1.1
OB23	622	bottle	Brown	0.2	3.3	2.0	48.5	1.2	0.2	bdl	21.7	21.3	bdl	0.8	0.9
OB24	N/A	bottle	Blue-green	0.3	4.3	1.7	51.2	2.9	0.3	bdl	19.6	18.5	bdl	0.5	0.6
OB25	N/A	bottle	Blue-green	1.3	4.4	2.6	48.6	1.6	bdl	bdl	16.7	22.2	0.2	1.1	1.2
OB26	N/A	small bottle	Blue-green	1.8	2.5	1.6	61.5	0.8	0.2	0.2	12.8	17.4	bdl	0.7	0.4
OB32	N/A	flat disc	Colorless	3.1	2.1	1.2	67.0	0.7	bdl	0.4	11.0	13.2	bdl	0.9	0.4
OB34	N/A	bottle?	Olive	0.2	2.9	1.2	52.6	0.9	0.4	bdl	20.8	19.8	0.2	0.7	0.5

Table 3. Average SEM-EDS results of glass samples shown as wt% of oxides and normalized to 100%. Samples are arranged by ID number and are divided between soda glass and forest glass.

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