

## The philosophers and the crucibles.

### New data on the 17<sup>th</sup>-18<sup>th</sup> century remains from the Old Ashmolean laboratory, Oxford

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**Abstract.** *The differentiation between alchemy and chemistry as separate disciplines is relatively recent. As such, an unbiased understanding of the early history of chemistry requires an approach to actual laboratory activities that avoids anachronistic biases and generalisations. This paper presents the analytical study of an assemblage of early modern chemical vessels used in the laboratory of the Old Ashmolean Museum in Oxford, the first university institution in Britain where chemistry was taught. We seek to explore the nature and range of laboratory activities conducted there in the late 17th and early 18th centuries, at a time when Europe witnessed the rise of the new experimental sciences and major technological advancements. The assemblage comprises crucibles of different types, ceramic distillation equipment and other containers. The analyses of residues found within them indicate that the laboratory's experimental programme focused on some of the most relevant technological as well as philosophical quests of the time, including the production/working of new types of glass and the distillation of zinc. The results reinforce the idea of a tightly connected chemical community operating in early modern Oxford and beyond, whose members included both natural philosophers and industrial entrepreneurs and whose aims straddled the investigation of nature as well as the extraction of profit. Moreover, this archaeological study makes visible the epistemic exchanges between the more scholarly and the more artisanal worlds, thereby making a relevant contribution to the history of early modern science as well.*

## 1. Introduction

The early modern period (ca. 1500-1800) saw exceptional technological progress and industrial development across Europe, fuelled by both the emergence of a consumeristic and mercantile society and an ever-growing scholarly curiosity to understand and control nature. Natural philosophy, the discipline that formulated theories to explain visible phenomena, was supported by growing empiricism and experimentation in the innumerable laboratories that existed across Europe (Anderson 2013). Here, practitioners routinely carried out a host of chemical procedures, ranging from mineral analysis to the distillation of acids, medicines, perfumes and alcoholic beverages, and from the manufacture of glass, porcelain and dyes to attempts at producing the philosopher's stone (Smith 1994; Moran 2005; Newman 2006; Nummedal 2007; Valleriani 2017). On the one hand, high-precision activities such as fire assay and attempts at metallic transmutation triggered a deeper understanding of the physico-chemical principles regulating matter (Debus 1998; Martín-Torres and Rehren 2005a; Newman 2000; 2004; 2006; Newman and Principe 2002; Rehren 1996). On the other hand, new knowledge fostered (and required) technological innovation, which between the late 17<sup>th</sup> and the first half of the 18<sup>th</sup>

century brought improved types of glassware (Von Kerssenbrock-Krosigk 2008; Dungworth and Brain 2009; Beretta 2009), the successful imitation of Chinese porcelain (Tite et al 1986; Wesley 2014) and the distillation of zinc (Etheridge and Dungworth 2012). The study of these issues has traditionally been the domain of historians of science and philosophy, and our understanding of the early modern culture of knowing is by and large text-based. More recently, and with particular impetus in recent years, there has been a growing interest in the practical and material aspects of early science (Anderson 2000; Smith 2007; Martínón-Torres 2011; Nummedal 2011; Werrett 2014). Archaeological remains from laboratories and artisanal workshops and their chemical analysis allow us to approach alchemical practice from a different perspective, and to explore the many overlaps between scholars and craftsmen, and their role in the emergence of the new experimental sciences of the 17<sup>th</sup> century (Zilsel 1942; Rossi 1970; Long 2001; 2011; Smith 2004; 2014; Roberts et al 2007; Klein and Spary 2010; Dupre 2014; Valleriani 2017). Pioneering research in this direction has been conducted with particular focus on early lab-based metallurgical activities (Rehren 1996; Martínón-Torres and Rehren 2005a; 2005b; 2007; Martínón-Torres et al 2008; Mongiatti et al 2009a; 2009b; Martínón-Torres 2012), with other studies including analyses of ceramic tools (Martínón-Torres and Rehren 2009) and glass apparatus (Veronesi and Martínón-Torres 2018). As a further contribution, here we present the results of analyses conducted on materials excavated from the site of one of the most famous laboratories in early modern Europe, the (Old) Ashmolean Museum in Oxford.

### 1.1 The Ashmolean laboratory

Opened in 1683, the Ashmolean laboratory occupied the building that today houses the Museum of the History of Science on Broad Street. The museum spread over three floors, hosting a collection of curiosities, a library, a lecture theatre and the *officina chimica*, the basement laboratory furnished with state-of-the-art equipment (Bennett et al 2000). The museum was part of Oxford University and represented the first institution where chemistry was officially taught in England. The first to be appointed keeper of the museum, director of experiments and chemistry professor was the natural philosopher Robert Plot, who had risen to prominence after publishing the *Natural History of Oxfordshire* in 1677 (Gunther 1923-1945, vol 12). Despite the high profile of this chemical laboratory, documentary evidence is rather silent regarding what type of experiments Plot and his assistant Christopher White pursued during their time here (Sherwood Taylor 1949; Guerrini 1994; Roos 2014). Equally, we know very little about Plot's successors after he retired in 1690. In 1999, excavation works took place at the rear of the building, demonstrating that the whole area had been used as a dump for

materials linked to the 17<sup>th</sup> and 18<sup>th</sup> century basement laboratory of the museum before being covered by limestone slabs (Hull 2003).

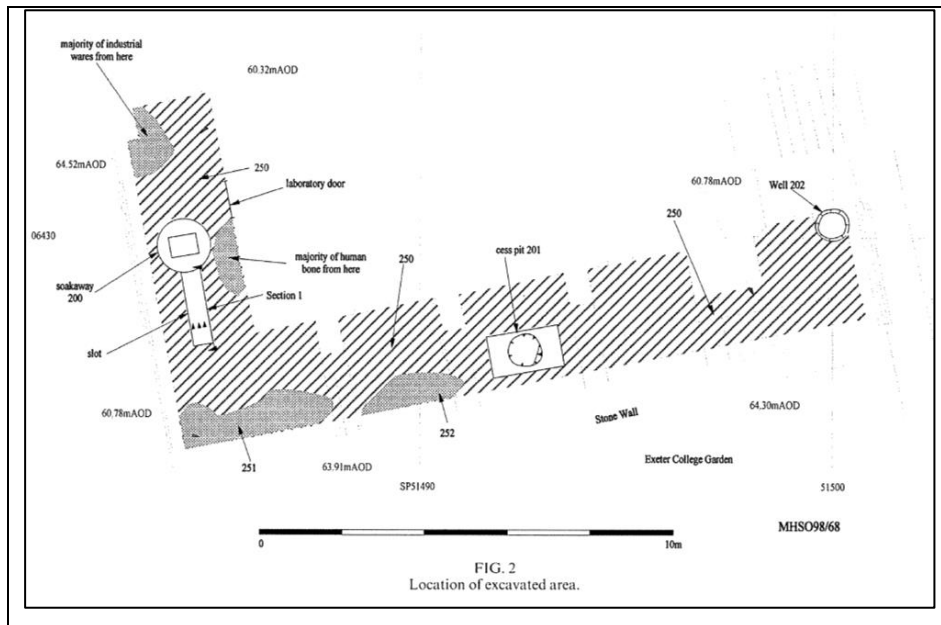


Figure 1. Map of the excavated area at the rear of the History of Science Museum in Oxford (from Bennett et al 2000: 35)

The vast majority of the artefacts recovered come from the dumping context 250, while a smaller number was excavated from the backfilling of smaller stratigraphic features such as the soakaway 200 (fig. 1). The most numerous findings are several thousand fragments of human and animal bones, to be linked with the anatomy teaching. The ceramic assemblage includes sherds of “domestic” pottery, clay tobacco pipes as well as pieces of laboratory equipment (Hull 2003), which constitutes the main focus of the present paper. The assemblage comprises 25 mostly complete crucibles of recognisable type, plus several non-diagnostic fragments, and pieces of equipment indicating distillation activities, such as two retorts and the base of a cucurbit, the globular vessel forming the lower part of a distillation apparatus. Finally, other containers include two flasks (one represented by two fragments), one stoneware bottle, and the fragment of a lid. The crucibles include both open and closed types (fig. 2). Examples of the former range from very small ( $H < 10\text{cm}$ ) rounded specimens to larger ones ( $H > 10\text{cm}$ ) of beaker shape with tapering profile, with or without a pouring spout, through to triangular crucibles of different size ( $H 5\text{-}10\text{cm}$ ). The latter type includes tall ( $H > 15\text{cm}$ ) crucibles with closed profile.

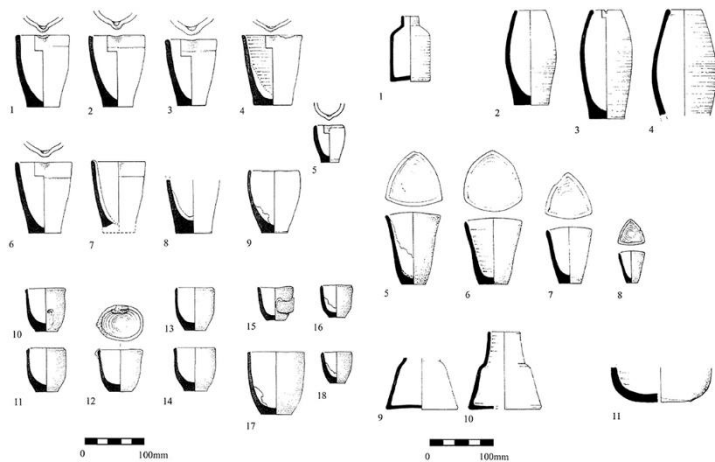


Figure 2. The ceramic crucibles excavated from the rear of the History of Science Museum in 1999 (from Hull 2003: 7-8). Left: Larger (n. 1-9) and smaller open type (n. 10-18); Right: Tall closed type (n. 2-4) and triangular type (n. 5-8). The remaining artefacts on the right-hand side are a bottle (n. 1), two flasks (n. 9-10) and a cucurbit (n. 11).

Dating the assemblage has been challenging since the secondary context they come from does not offer useful stratigraphic information. Moreover, pieces of ceramic apparatus like the ones found at the Ashmolean laboratory have been used, virtually unchanged in style, from the Middle Ages to the modern era (Taylor 1945; Moorehouse et al 1972; Booth 2016). Among the domestic wares are fragments of glazed and decorated pottery dating from the late 17<sup>th</sup> up to the first half of the 19<sup>th</sup> century (Hull 2003: 5-9). The very nature of the operations carried out within the apparatus may represent a further clue as to the chronology of the assemblage. In this sense, both the initial XRF analyses performed by Chris Salter shortly after excavation (reported in Hull 2003: 11-13) and the preliminary SEM-EDS investigation of a few samples by one of us indicates that the emerging chemical activity matches philosophical and industrial interests of the late 17<sup>th</sup> century, and therefore points to a date close to the opening of the Old Ashmolean (Martín-Torres 2012: 26). In the present paper we present a more comprehensive set of data from the analysis of seventeen samples by SEM-EDS, which we amalgamate with previous datasets where appropriate. The questions addressed encompass the type of ingredients and reagents employed and recipes followed by the practitioners at play. At the same time, the results provide a fresh perspective on the historiographic issue of the relationship between craft and science in early modern Europe (Smith 2014; Dupre 2014). The emerging picture reinforces the potential of a material culture-based approach as a source of information in the history of science, and adds to our knowledge of one of the most important laboratories of early modern Europe.

## 2. Materials and methods

The sample selection was informed by an initial assessment carried out on-site using a portable X-ray fluorescence spectrometer (pXRF). For curatorial reasons, only objects that were already fragmentary were targeted for invasive sampling, trying to cover the diversity of typologies and chemical residues identified in the initial screening. Sampling was carried out using a small Dremel drill with a rotating diamond-coated blade. When possible, a section including the ceramic body and the residue was cut from the vessels. In other cases, only the internal residue was sampled by scraping it off with a scalpel. A summary of the samples analysed for the present paper, reporting any code previously received, is presented in **table 1**.

Sample ID (as it appears here)	Type of artefact	Process	Further code (Martínón- Torres 2012)	Further code (Hull 2003)
AN1999.205.9d	Beaker crucible	Lead crystal glass making/working		
AN1999.205.14	Greenish glassy residue (from beaker crucible)	White opaque glass making		OX1411
AN1999.205.17	Beaker crucible	Lead crystal glass making/working	OXn003	
AN1999.205.18	Drug jar	Uncertain		
AN1999.205.19	Beaker crucible?	Uncertain		
AN1999.205.21	Cucurbit	Zinc distillation?		OX1404
AN1999.205.22	Tall closed crucible	Zinc distillation		
AN1999.205.23	Triangular crucible	N/A		
AN1999.205.24	Triangular crucible	N/A	OXn001	
AN1999.205.25	Triangular crucible	White opaque glass	OXn002	
AN1999.205.27	White crust (from tall closed crucible)	Zinc distillation		
AN1999.205.28	Graphitic crucible	Transmutation? Medical alchemy?		
AN1999.205.30	Beaker crucible	White opaque glass making		
AN1999.205.31	Triangular crucible	Uncertain		
AN1999.205.32	Retort	Zinc distillation?		R1
OX1409	Flask	Uncertain		OX1409

**Table 1.** *The samples analysed in this paper, including the type of laboratory vessel they come from and correspondent IDs given to them in previous studies (if applicable). A more comprehensive list, including images of the samples is given in table S1 (supplementary materials).*

The specimens were mounted in epoxy resin blocks, their cross-section ground flat and polished down to 1µm following standard procedures. The blocks were carbon-coated and analysed using a Hitachi S-3400 scanning electron microscope with an Oxford Instruments energy dispersive spectrometer (SEM-

EDS). All analyses were carried out at an accelerating voltage of 20 kV and at constant 10mm working distance, with an acquisition time of 100s. 'Bulk' analyses on the residues were performed at relatively low magnification on areas between 100 and 300  $\mu\text{m}$  wide, while for the characterisation of the ceramic matrix smaller areas at higher magnification were targeted, avoiding larger inclusions more likely to come from tempering materials (Freestone and Tite 1986; Martín-Torres and Rehren 2009). Spot analysis of discrete phases was performed at higher magnification on smaller areas of variable size. Throughout the paper images are presented from the backscattered electron detector (BSE). Results were processed using INCA Oxford Instruments software and are reported in wt% of stoichiometric oxides for ceramic and glassy matrices and for mineral phases, and in elemental wt% for metallic phases. Bulk areas with comparable microstructure were analysed three to five times and the average calculated, whereas discrete phases were normally analysed once. In order to account for corrosion, porosity and to facilitate comparability, all data is shown normalised to 100 wt%. Precision and accuracy of the machine were evaluated through the analysis of certified reference materials with comparable composition. Results (**tables S2-4 in supplementary materials**) indicate an accuracy within 10% relative error for most elements of interest, in the amounts encountered here. Equally, precision appears satisfactory, with a coefficient of variation well below 10% in the vast majority of cases.

### 3. Results

The following four sections outline the results on the ceramic equipment itself (3.1), glass-related activities (3.2), the metallurgy of zinc (3.3) and other samples that could not be conclusively ascribed to any specific activity (3.4).

#### 3.1 The ceramic apparatus

The composition of the chemical vessels sampled is reported in **table 2**. The triangular crucibles have a highly vitrified matrix with abundant sand tempering visible as small rounded quartz grains, and elongated voids parallel to the surface of the vessel and around individual quartz grains (**fig. 3**). These were caused by the expansion and shrinkage of the quartz upon firing and contributed to the resistance of the vessel to thermal and mechanical stress, by preventing the propagation of catastrophic cracks during high-temperature operations (Kilikoglou et al 1998; Tite et al 2001).-The composition of the ceramic matrices of these crucibles show the highest levels of alumina in the assemblage (>38 wt%  $\text{Al}_2\text{O}_3$ ) and very low concentrations of alkali and alkali earth oxides (<3wt% total), denoting an exceptional refractoriness. Post-medieval crucibles with these characteristics were famously made in the Hesse region of Germany from which they were exported all over the world (Martín-Torres and

Rehren 2009). The alumina-rich kaolinitic clay employed for their manufacture and the high-temperature pre-firing afforded these vessels extraordinary thermal and chemical refractoriness (Martín-Torres et al 2006; 2008).

Sample ID	Tempering	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Cl	SO <sub>3</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	FeO	ZnO
AN1999.205.9d	QS+G	0.3	0.7	36.5	56.8	bdl	bdl	1.8	0.2	1.6	2.0	bdl
AN1999.205.17	QS+G	0.3	0.9	34.8	58.3	bdl	bdl	2.1	0.2	1.3	2.2	bdl
AN1999.205.18	QS	0.4	0.4	29.2	64.0	bdl	bdl	1.8	0.3	2.3	1.5	bdl
AN1999.205.19	QS	0.3	0.8	32.6	60.2	bdl	bdl	2.2	0.3	1.3	2.3	bdl
AN1999.205.21	QS	0.9	0.4	30.5	63.6	bdl	bdl	2.2	0.2	1.4	1.0	bdl
AN1999.205.22	QS+G	0.2	0.5	32.1	60.8	bdl	bdl	2.2	0.3	1.4	2.1	0.3
AN1999.205.23	QS	bdl	0.7	39.5	54.8	bdl	bdl	1.2	0.3	1.8	1.7	bdl
AN1999.205.24	QS	bdl	0.6	38.1	55.7	bdl	bdl	1.9	0.3	1.8	1.7	bdl
AN1999.205.25	QS	0.2	0.6	38.8	54.9	bdl	bdl	1.7	0.4	1.8	1.7	bdl
AN1999.205.28	Gr	0.4	0.5	21.0	54.8	bdl	0.5	19.5	bdl	1.3	2.0	bdl
AN1999.205.30	QS+G	0.2	0.6	37.2	56.3	bdl	bdl	2.1	0.4	1.3	1.8	bdl
AN1999.205.31	QS	0.2	0.7	38.7	55.3	bdl	bdl	1.4	0.4	1.8	1.6	bdl
AN1999.205.32	QS+G	0.3	0.7	34.0	59.3	bdl	bdl	2.6	0.2	1.2	1.7	bdl
OX1409	Q+G	0.6	0.8	30.9	60.7	0.5	bdl	2.1	0.3	2.0	2.3	bdl

*Table 1. Average chemical composition by SEM-EDS of the ceramic matrices avoiding large inclusions. QS: quartz sand; S+G: quartz sand + grog; Gr: graphite; Q+G: quartz + grog*



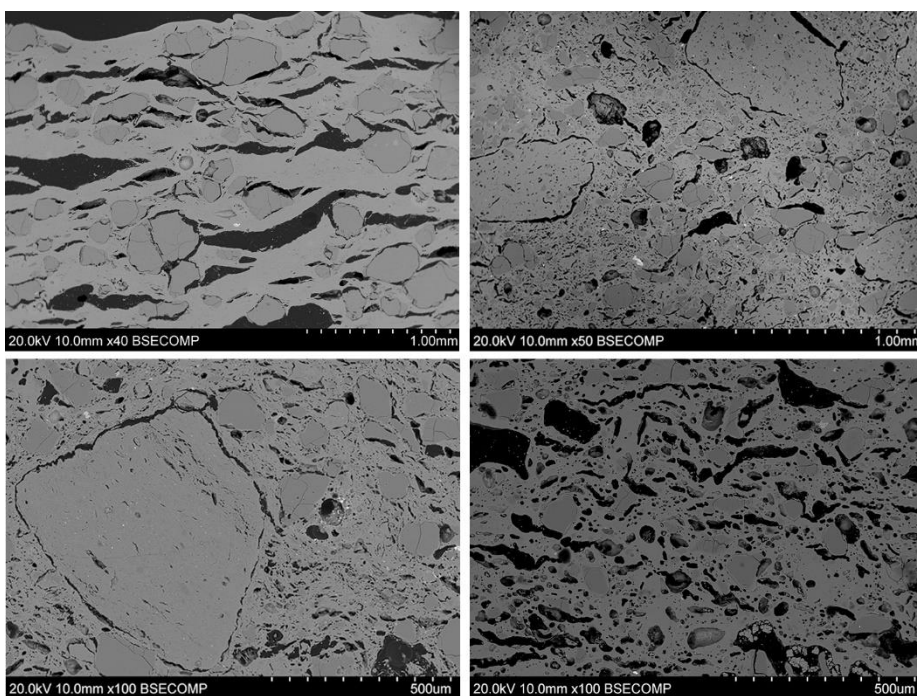


Figure 3. SEM backscattered micrographs showing different crucible ceramic fabrics. **A:** Triangular crucible AN1999.205.23 with sand temper. Note the shattered quartz grains (medium grey) and elongated voids (black) caused by the quartz expansion and subsequent shrinkage during firing at high temperature. **B, C:** Vessels AN1999.205.30 and AN1999.205.32 tempered with sand (medium grey) and grog (typically larger, light grey discrete areas within the matrix). **D:** Example of severely molten and vitrified vessel (AN1999.205.21) showing vesicular bloating pores across the matrix.

Except for the graphitic crucible AN1999.205.28 and the flask OX1409, which will be presented separately, the rest of the vessels share a broadly similar microstructure. Their matrices show signs of distortion and of more or less severe melting of the ceramic body, which in some instances becomes continuous and includes the formation of vesicular bloating pores (**fig. 3D**). Tempering materials are either quartz sand alone or quartz coupled with grog. The former is typical of the triangular crucibles, while the beaker crucibles, the tall crucibles and the retort display the latter combination (**fig. 3**). The grog inclusions are both chemically and microstructurally comparable to the surrounding ceramic but often look more vitrified, suggesting that they were exposed to high temperatures prior to being mixed with the clay and that therefore they are recycled old pots or crucibles (Freestone and Tite 1986; Freestone 1991). The practice of tempering technical ceramics with grog is found in technical texts of the time (Hoover and Hoover 1950: 230; Sisco and Smith 1949: 111; Glauber 1651: 315) and has been reported from numerous archaeological samples of English glasshouses and metalworking sites (Freestone and Tite 1986; Dungworth 2006: 18 and references therein; Bayley and White 2013). The composition of the ceramic matrices of these crucibles shows a slightly higher content of alkali and alkali earth oxides when compared to the triangular vessels (total >3wt%) and slightly lower alumina.

However, levels of these oxides vary between samples, with the grog-tempered vessels usually showing higher values (up to 36.5 wt%  $\text{Al}_2\text{O}_3$ ) than the rest of the samples (up to 32.6 wt%  $\text{Al}_2\text{O}_3$ ). Despite the internal differences, the vessels from the Old Ashmolean are considerably richer in alumina than other post-medieval crucibles from English glasshouses and metallurgical workshops (**fig. 5**) (Vose 1994; Dungworth 2006 and references therein; White and Kearns 2010). Thus, the Ashmolean vessels were manufactured with a particularly high-alumina refractory clay of the kaolinite type, similar to that employed for the German crucibles from Hesse and Bavaria. Indeed, the very minor differences between at least part of the non-triangular crucibles from Oxford and Hesse does not allow to rule out a possible German origin for the former at least (Martín-Torres 2012: 29).

The flask fragment OX1409 has a different microstructure. First, it is not vitrified, indicating a lower temperature both in firing and during use. Second, its texture is coarser, with more diversified and more numerous inclusions in it (**fig. 4**). It contains quartz, though the (sub-)angular shape of the grains and their reddish-pink colour in cross-section points to a type of siliceous temper different from sand (see also Hull 2003: 11). Grog was added to this vessel, too, visible as large angular inclusions, while smaller and rounded clay pellets probably coming with the sand can also be seen (**fig. 4**). Finally, the fragment of black crucible (AN1999.205.28) differs markedly in both its microstructural and compositional profile from the rest of the vessels analysed. The fabric appears black in cross-section due to the presence of graphite flakes added as temper to enhance the clay's refractoriness (**fig. 4**). The extraordinary heat resistance and inert qualities of graphite rendered it an ideal temper for technical ceramics subjected to extreme temperatures and corrosive chemical reactions, and similar crucibles were a highly specialised production originating from Bavaria (Martín-Torres and Rehren 2009). This type is believed to appear in England during the 18<sup>th</sup> century at the Warmley brass works site near Bristol linked to the distillation of zinc (Dungworth and White 2007) (cfr. 3.3). The chemical composition of the matrix in the Ashmolean sample reveals lower alumina when compared to the other crucibles (21 wt%  $\text{Al}_2\text{O}_3$ ), and unusually high levels of potash throughout its profile (19.5wt%  $\text{K}_2\text{O}$ ). The latter is very unlikely to reflect the composition of the clay, and instead probably derives from interaction between the crucible and the corrosive potassium-rich residue found within it (cfr 3.4), which at high temperature caused the vessel to partially melt as shown in **figure 12**. The ratio of alumina to silica of the sample analysed here is roughly in line with black crucibles from various other sites, as well as with the one other fragment of graphitic crucible discovered at the Ashmolean laboratory and displaying a stamp from the Bavarian manufacturer (Martín-Torres 2012) (**fig. 5**)

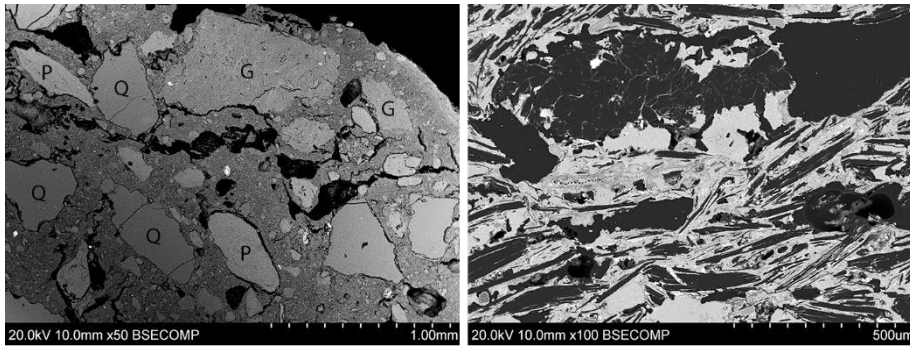


Figure 4. Left: SEM backscattered micrograph showing ceramic matrix of flask sample OX1409 (left) with large quartz grains (Q), grog inclusions (G) and clay pellets (P). Right: Ceramic matrix of sample AN1999.205.28 tempered with flakes of graphite (black).

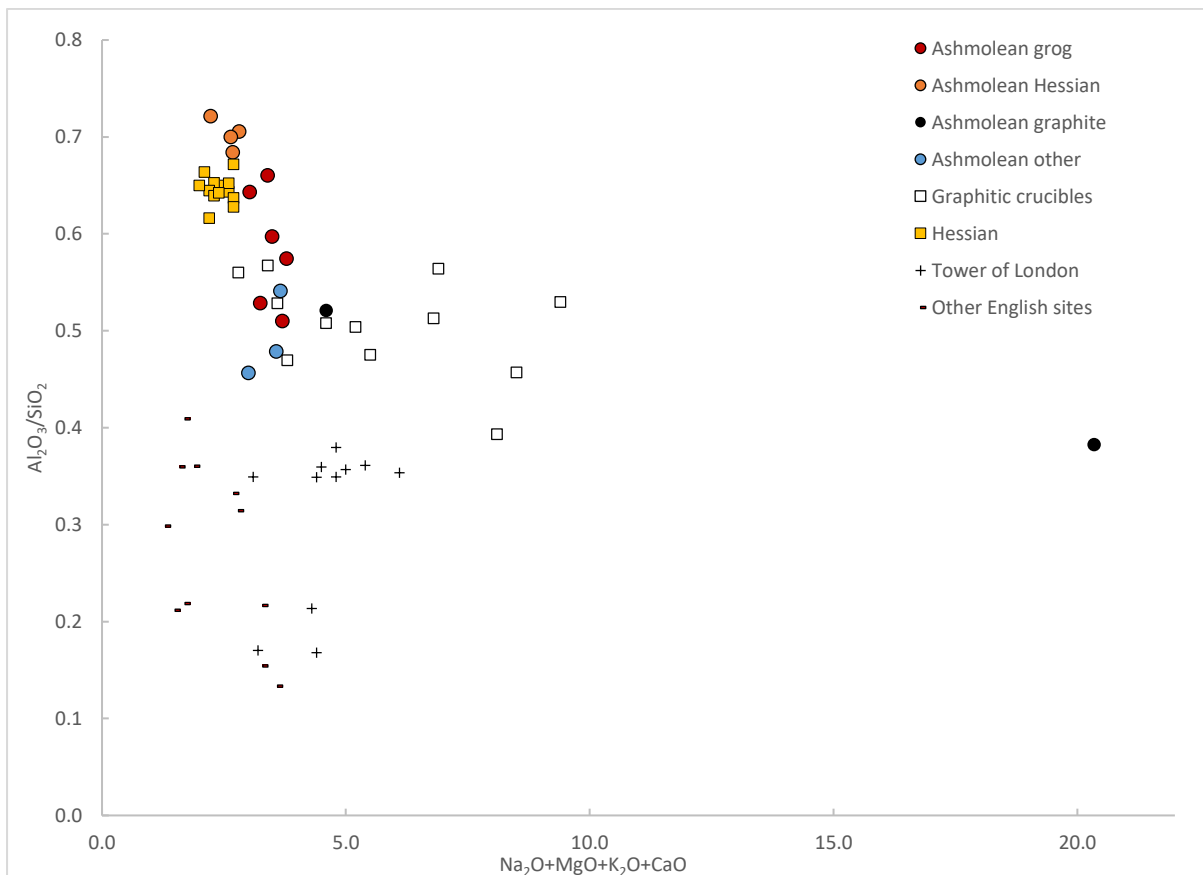


Figure 5. Alumina to silica ratio vs total alkalis in the Ashmolean vessels and in other post-medieval crucibles from England and Europe. Data includes the other fragment of graphitic crucible from the Ashmolean (Martinón-Torres 2012) crucibles made in Hesse and Bavaria (Martinón-Torres and Verrocchio 2008; Martinón-Torres and Rehren 2009; Gardner et al 2018); the Tower of London (White and Kearns 2010) and various English glasshouses (Vose 1994; Dungworth 2006 and references therein). In most cases, the composition of English crucibles is obtained through the analyses of larger areas than those analysed here, and may include a higher proportion of inclusions.

### 3.2 Glass-related activities

Five of the analysed crucibles were found to contain residues that can be related to the manufacture or working of glass-based products. There are, however, important compositional differences as to glassmaking ingredients employed and type of glass obtained. **Table 3** shows the chemical composition of the residue matrices, avoiding larger inclusions where present. The most noticeable difference is that between samples of lead-potash glass and one sample each of antimony-potash and soda-lead glass.

ID	Glass type	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Cl	SO <sub>3</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	FeO	SnO <sub>2</sub>	Sb <sub>2</sub> O <sub>3</sub>	PbO
AN1999.205.9d	Pb crystal (blue)	bdl	bdl	0.1	51.3	0.2	bdl	11.1	bdl	bdl	bdl	bdl	bdl	37.4
AN1999.205.17	Pb crystal (colourless)	0.8	bdl	bdl	59.9	0.3	bdl	11.8	bdl	bdl	bdl	bdl	bdl	27.1
AN1999.205.14	Green glass (Pb-based)	12.5	bdl	bdl	52.5	0.1	bdl	bdl	bdl	bdl	0.8	1.8	bdl	32.4
AN1999.205.25	Opaque white (Sb-based)	0.1	0.3	6.6	38.9	bdl	0.6	17.3	3.3	0.4	1.4	bdl	31.1	bdl
AN1999.205.30	Opaque white (Pb-based)	0.6	bdl	5.2	38	bdl	bdl	14.3	1.0	bdl	0.6	bdl	bdl	40.3

*Table 2. Average bulk chemical composition of glassy residue matrices.*

#### 3.2.1 Lead-based glass

All but one sample of the glass-related residues show a high-lead composition and were found inside beaker type crucibles. Samples AN1999.205.9d and AN1999.205.17 were taken from, respectively, a layer of blue and a layer of colourless glass. The latter had been analysed previously (OXn003 in Martín-Torres 2012) and has been re-analysed here. Both are potash-lead-silica glasses of high purity, with negligible amounts of other oxides. The glass composition is consistent with lead crystal, a novel type of glass patented at the end of the 17<sup>th</sup> century by George Ravenscroft (MacLeod 1987; Moretti 2003; Brain and Brain 2016). The ingredients of lead crystal were very pure and included sand or calcined flints (hence the alternative name of flint glass), saltpetre (potassium nitrate) and litharge (Dungworth and Brain 2005; 2009). The noticeable differences in the levels of the main oxides and particularly in the lead oxide content of the two samples may be indicative of experimentation with a recipe that was still new and required adjustments. Lead oxide was an essential stabiliser for the glass, and analyses performed on samples from different periods show that levels gradually increased during the initial decades of lead crystal production, eventually settling at around 40 wt% PbO to remain virtually unchanged throughout the 18<sup>th</sup> century (Redknap et al 1995; Dungworth and Brain 2013). Sample AN1999.205.9d, with lead oxide in excess of 35 wt% and potash around 11 wt%, is similar to the post-1690s glasses from Dungworth and Brain group 3 and 4. On the contrary, sample

AN1999.205.17 has levels of these oxides that are more similar to Dungworth and Brain group 2, with glasses dated to the 1680s (Dungworth and Brain 2009; 2013) (fig. 7). Lastly, the blue colour of one of the residues can only be intentional considering that the purity of the ingredients employed would not have produced any natural colouration in the glass. Existing analyses of 18<sup>th</sup>-century blue lead crystal show around 0.04 wt% of cobalt oxide (Redknapp et al 1995), a quantity that is below the detection limit of the SEM-EDS. We can only assume that the same colourant was employed for the blue lead crystal in the Oxford crucible.

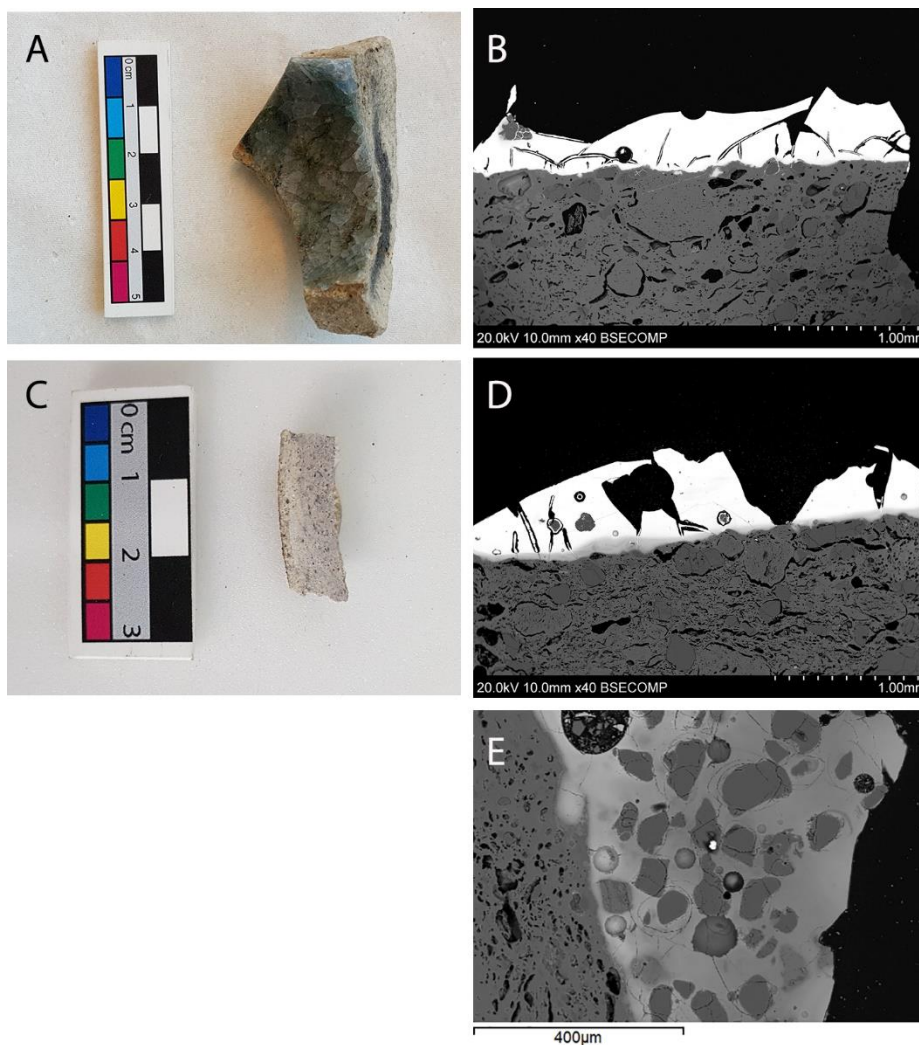


Figure 6. Samples AN1999.205.9d (A) and AN1999.205.17 (C) with evidence of lead crystal adhering the crucibles' walls, and corresponding SEM backscattered micrographs (B,D) showing the bright dense glass layer stuck to the ceramic. Note the area with residual rounded quartz and feldspars in sample AN1999.205.17 (E).

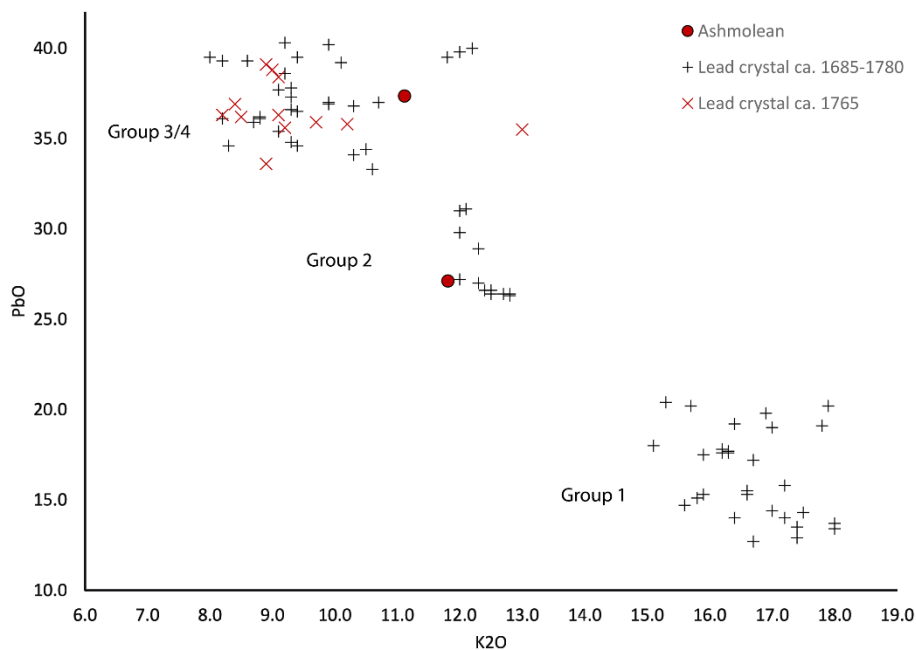
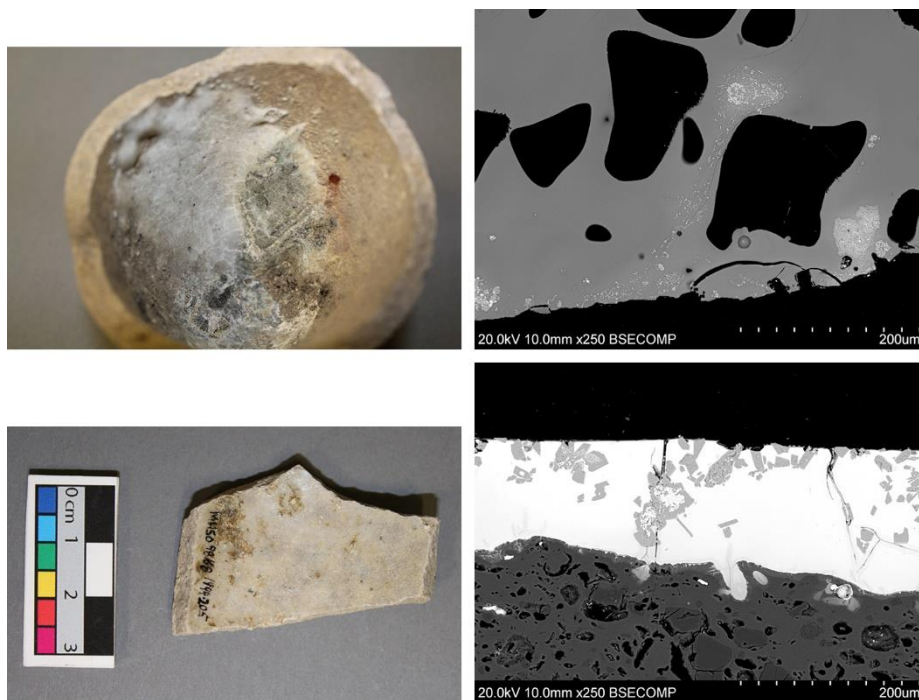


Figure 7 The two samples of lead crystal from the Ashmolean laboratory (red dots) compared to available legacy data (from Dungworth and Brain 2009; 2013 and from Redknapp et al 1995). The cluster forming at the bottom-right corner corresponds to group 1 from Dungworth and Brain, the cluster towards the middle is group 2, while on the upper-left corner is group 3/4.

The other two samples with evidence of high-lead glass are AN1999.205.14 and 30, but unlike the previous ones, they show evidence of tin-based opacification. Again, the compositional differences are suggestive of experimentation. Sample AN1999.205.14 is a small fragment taken from the bottom of a beaker crucible, where two areas of glassy residue can be seen intermixing: one is opaque white, while the other, from which the piece analysed comes, is dark green (**fig. 8**). The glass phase has a soda-lead-silica composition (12.5 wt% Na<sub>2</sub>O, 32.4 wt% PbO, 52.5 wt% SiO<sub>2</sub>) with an enrichment in tin oxide (ca. 2 wt% SnO<sub>2</sub>) due to the presence of rounded tin-rich aggregates within the glass matrix (**fig. 8**). These minute crystals are mainly composed of pure tin oxide, even though analysed levels range from ca. 46 to ca. 93 wt% SnO<sub>2</sub>. The balance is provided by lead oxide, silica and soda, most likely absorbed from the surrounding lead glass and also highly variable (ca. 2-26 wt% PbO and ca. 4-27 wt% SiO<sub>2</sub>) (**see table S5 in supplementary material**). In addition to these crystals, opacification was given by the large sub-rounded grains of partially reacted quartz that can be seen in the glass matrix.

The glassy residue of sample AN1999.205.30 contains 40.3 wt% lead oxide and 38 wt% silica, while the third main component is in this case potash (14.3 wt% K<sub>2</sub>O), indicating the use of a potassium-based

flux instead of soda-based one. The high alumina detected in this sample (ca. 5 wt%  $\text{Al}_2\text{O}_3$ ) is likely caused by absorption of ceramic material, particularly noticeable due to the thinness of the residue (Dungworth 2008). Scattered throughout the glass matrix are angular particles rich in tin oxide (ca 35 wt%  $\text{SnO}_2$ ) and representing the opacifying agent (**fig. 8**). Their composition is very constant and also includes around 42 wt% silica and 20 wt% potash (see **table S5 in supplementary material**), consistent with a formula of  $\text{K}_2\text{SnSi}_3\text{O}_9$ , which has an ideal composition of 42 wt%  $\text{SiO}_2$ , 35 wt%  $\text{SnO}_2$  and 22.5 wt%  $\text{K}_2\text{O}$ ; its nearest mineral analogue is wadeite, with Zr instead of Sn in the formula. This phase, not previously reported in historical glass samples (but see Sakai et al 2000), probably formed when the tin oxide added to the batch reacted with the surrounding glass melt. A further indication to this is the presence in many of the crystals of a bright  $\text{SnO}_2$  core (fig. 8 and S1 in supplementary materials). The absence of tin oxide in the bulk glass composition can be explained by the same diluting effect due to interaction with the crucible (Heck et al 2003).



*Figure 8. Residues of opacified glass. Top: The inside of the crucible from which sample AN1999.205.14 was taken. Note the two intermixing glass areas, opaque white and green. This glass was opacified by the white clusters rich in lead and tin oxide (bright white) and large grains of partially reacted quartz (dark). Bottom: The crucible fragment from which sample AN1999.205.30 was taken (left), with its microstructure in SEM backscattered mode (right). Note the angular  $\text{K}_2\text{SnSi}_3\text{O}_9$  crystals (grey) with a bright tin-rich core (more images in fig. S1, supplementary materials).*

In the period under consideration, the opaque white effect could be achieved through the precipitation of tin oxide in the glass matrix (Tite et al 2008; Tite 2009; Biron and Chopinet 2013; Moretti and Hreglich

2013; Matin 2019; Matin et al 2018). This is the case in sample AN.1999.205.30, where the glass is opacified by a newly-identified tin-rich phase crystallising from the melt in reaction with added tin oxide particles. AN.1999.205.14 is less straightforward, since the presence of numerous residual quartz grains in the soda-lead glass may also point to a semi-finished glass, similar to frit, which often appears white from residual quartz grains and / or gas bubbles (e.g., Rehren and Pusch 2005; Smirniou and Rehren 2011). Nonetheless, the fact that this glass has some tin-rich clusters points more towards intentional opacification through tin calx.

### 3.2.2 Antimony-based glass

The last sample with glass-related evidence (AN1999.205.25) is a fragment of triangular crucible with a yellowish-green glassy drip going down its outer wall (**fig. 9**). This residue is bright and glassy under the SEM (**fig. 9**) and has a potash-antimony oxide-silica composition (ca. 17 wt%, 31 wt% and 39 wt% respectively) (**table 3**). A significant degree of absorption of crucible material is indicated by the high alumina content of the glass (6.6 wt%  $\text{Al}_2\text{O}_3$ ). Moreover, the composition also includes lime (3.3 wt% CaO), iron oxide (1.4 wt% FeO), sulfur (0.6 wt%  $\text{SO}_3$ ) and titania (0.4 wt%  $\text{TiO}_2$ ), plus very minor quantities of magnesia and soda. As in other glass samples analysed here, a potassium-based flux was employed, probably saltpetre or salt of tartar, since the absence of magnesia and phosphate rule out plant or wood ashes as flux (Wedepohl and Simon 2010). Scattered across the glass matrix are small white particles of calcium antimonate (**fig. 9**) with 61-64 wt% antimony oxide and 20-21 wt% lime, the balance made of elements from the surrounding glass. Finally, a few antimony sulfide prills were also discovered in the cracks of the ceramic matrix of the crucible, their presence suggesting the use of mineral stibnite ( $\text{Sb}_2\text{S}_3$ ) as the source of antimony. Besides crucible contamination, the presence of stibnite in the melt, potentially carrying its own gangue, may be the reason to explain the “dirtier” composition of this glass when compared to the other samples analysed, as well as what triggered the crystallisation of calcium antimonate (Shortland 2002; Lahil et al 2008).

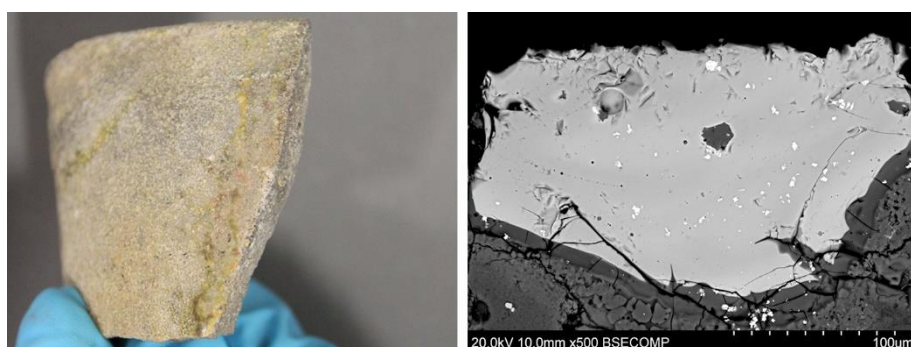




Figure 9. Left: Crucible AN1999.205.25 with yellowish-green antimony-rich glassy residue on the external wall. Right: SEM backscattered micrograph of the residue showing small bright calcium antimonate particles scattered across the glass phase.

Recipes for opaque white glass employing stibnite and salt of tartar are recorded in early modern documents (Moretti et al 2004: 27). Thus, this sample may be an attempt at making an opaque white glass by following a different recipe from that discussed above. In his commentary to Antonio Neri's famous glassmaking treatise *L'Arte Vetraria*, Christopher Merret adds a recipe to make an opaque white glass by using stibnite and saltpetre which would probably have led to a glass like that seen here (Cable 2006: 365). A sample from the same vessel was previously analysed by Martín-Torres (2012), who suggested a possible connection with the alchemical *glass of antimony* (a form of vitrified antimony oxide), a substance particularly appreciated in pharmacology and in the production of telescopic mirrors (Valentine 1678; Roos 2010). However, historical recipes for *glass of antimony* do not include sand or other sources of silica, and laboratory replications have shown that vitrification of the antimony oxide occurs with the silica brought in by the ore itself (Principe 1987). Instead, the amount of silica in sample AN1999.205.25 appears too high for it not to be intentionally added. Regardless of the specific end product, the chymists seem interested in the effects of antimony oxide in glass, possibly as a way to replace lead oxide since the two have similar properties (see for instance Hoover and Hoover 1950: 110).

### 3.3 Zinc metallurgy

Based on the XRF analysis carried out after excavation, Chris Salter reported traces of zinc in several of the artefacts (Hull 2003: 11-12). However, owing to the qualitative nature of the data obtained it was impossible to be more specific about the operations carried out in the vessels. Four samples analysed in the current study can be related to zinc metallurgy. The first two are fragments from the same tall closed-profile crucible of the type shown in **figure 2 no. 2-4**, showing patches of a white crust on its internal walls (**fig. 10**). pXRF performed before sampling signalled a strong enrichment in zinc and the presence of cadmium. Sample AN1999.205.22 is a cross-section through the crucible and the white residue, while sample AN1999.205.27 is a small lump of the crust only. In both cases, a core of metallic zinc was identified upon cutting through the corrosion layer, the metal being very pure with only minor amounts of cadmium (ca. 0.3 wt% Cd) and nickel (ca. 0.1-0.2 wt% Ni) sometimes detected. The proximity of these values to the instrumental limit of detection means that they should be taken as indicative only. Tiny lead-rich droplets also containing small amounts of cadmium were found scattered in the metallic zinc, while all around the metal core is a corroded area composed of zinc oxide and

sulfate of post-depositional origin. Bulk analysis of the ceramic body of the crucible shows minor zinc contamination ( $\leq 2.5$  wt% ZnO). Analyses of crucibles used in metallurgical operations involving zinc often show a higher degree of contamination (Rehren 1999a; Dungworth and White 2007; Etheridge and Dungworth 2012). However, this is not always the case and levels of zinc oxide in the ceramic can be similar to what is seen here (Freestone et al 1985; Craddock et al 1998; Rehren 1999b; Zhou et al 2012).

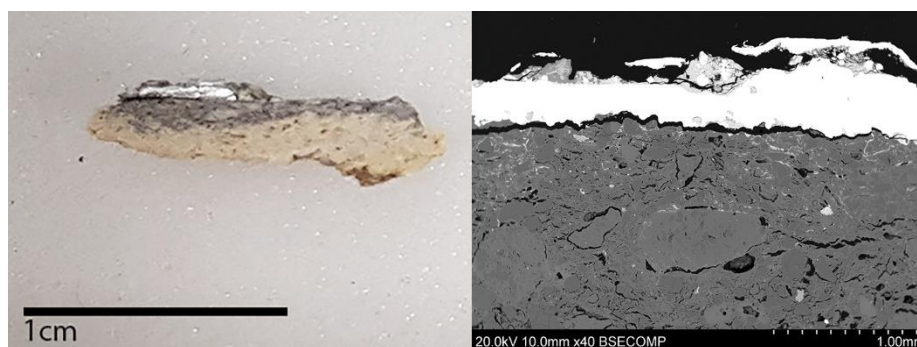
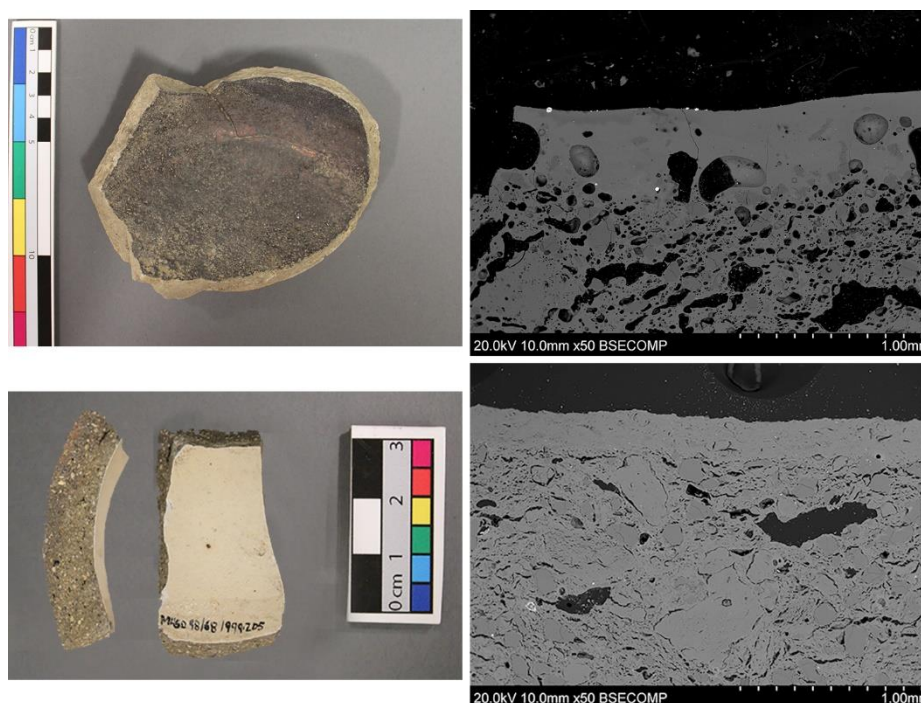


Figure 10. Sample AN1999.205.22 with direct evidence of zinc metallurgy. Left: Section through crucible wall and white residue showing the metallic zinc layer. Right: SEM backscattered micrograph of the section with the bright zinc sitting on top of the ceramic.

The other two samples related to zinc metallurgy come from the cucurbit fragment AN1999.205.21 and the piece of retort AN1999.205.32 (**fig. 11**). Chris Salter reported a strong zinc enrichment on the vitrified layer outside of the cucurbit's bottom (**fig. 11**), and suggested that the vessel had been used as a lid absorbing zinc-rich fumes (Hull 2003: 11). As it was not possible to sample through the outer vitrified layer, a cross-section of the inside wall was analysed instead, including a dark glassy interaction layer for which no zinc signal came from the pXRF analysis conducted during sampling (**fig. 11**). The layer is made of silica (57.5 wt% SiO<sub>2</sub>), alumina (22.6 wt% Al<sub>2</sub>O<sub>3</sub>) and alkalis (9.1 wt% Na<sub>2</sub>O and 4.8 wt% K<sub>2</sub>O), plus smaller quantities of lime (2.9 wt% CaO), titania (1.1 wt% TiO<sub>2</sub>), magnesia (1.6 wt% MgO), phosphate (0.3 wt% P<sub>2</sub>O<sub>5</sub>) and iron oxide (0.3 wt% FeO). Tiny iron prills appear dispersed in the glassy matrix (**fig.11**), their composition including a host of elements among which phosphorus is the most abundant one (10-33 wt% P), followed by titanium ( $\leq 6.3$  wt% Ti), manganese ( $\leq 5.1$  wt% Mn), copper ( $\leq 4.4$  wt% Cu), vanadium ( $\leq 1.1$  wt% V), chromium ( $\leq 0.6$  wt% Cr) and in one occasion around half percent of zinc. In two of them, around 0.3 percent nickel was detected, while one yielded a percent of zinc. The complex composition and small size of the prills indicate that they formed as a consequence of the extremely reducing conditions in the vessel which transformed oxides derived from the ceramic (iron and titanium especially) or from charcoal (phosphate) into metal. Similar features were observed in the

vessels used to distil zinc in the 18<sup>th</sup>-century Warmley brass works near Bristol (Dungworth and White 2007).

Lastly, the retort fragment AN1999.205.32 is also included here given its connection to distillation activities. However, the fragment does not display a crusty residue akin to those described above. Instead, a smooth light-coloured slip covers the internal wall of the retort's lower part and under the SEM this appears very similar to the ceramic, but more compact and not as porous (**fig.11**). The pXRF screening performed on the retort revealed the presence of barium on the internal slipped surface, and of lead, mercury and zinc towards the spout. However, SEM-EDS performed on the small fragment did not detect any relevant enrichment.



*Figure 11. The distillation apparatus with indirect evidence of zinc metallurgy. Top: Inner surface of the cucurbit bottom AN1999.205.22 (left). Note the dark vitrified interaction layer which under the SEM (right) looks glassy and contains small iron-rich prills. Bottom: Retort fragment AN1999.205.32. The internal surface is coated with a smooth white slip (left), which under the SEM (right) looks less porous than the ceramic matrix.*

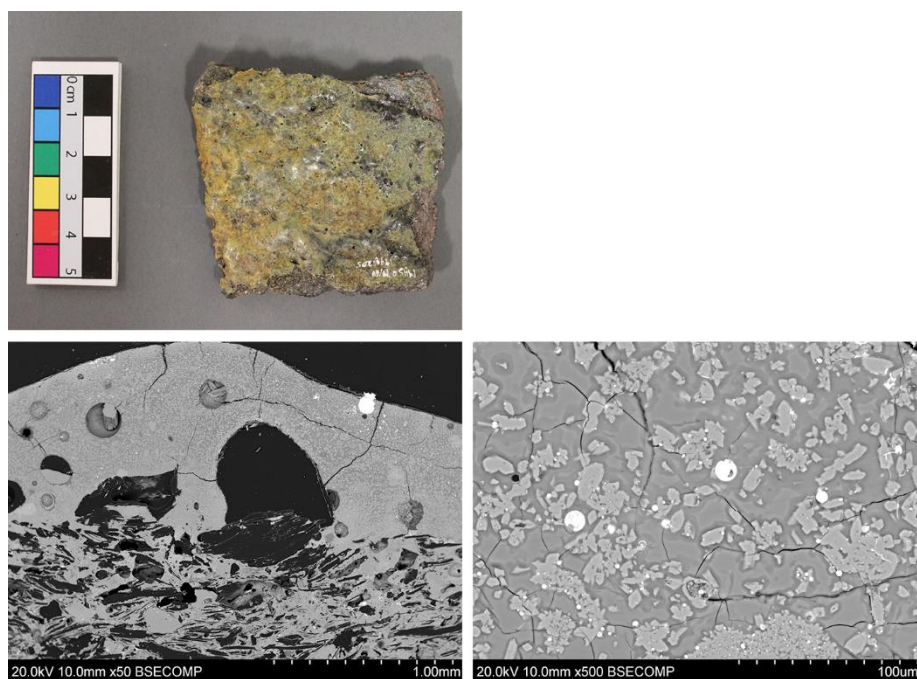
Notably, none of the samples directly or indirectly related to zinc metallurgy contain any consistent trace of copper. This allows to rule out brassworking, whereby closed-profile vessels were necessary to retain the zinc vapour and allow the copper to absorb it (Bayley 1998; Day 1988; 1998; Rehren 1999b; Craddock 1998; Craddock and Eckstein 2003). Here, it appears that zinc metal was the sole focus of the laboratory practitioners' attention. This interest would be consistent with the still rather mysterious

character of this substance in early modern metallurgy and natural philosophy, at a time when zinc was not widely considered as a metal in its own right (de Ruelle 1995; Craddock 1998; Rehren and Martín-Torres 2008). The peculiar volatility of zinc, which boils at around 907 °C, did not allow smelting procedures akin to those in use for other metals because this would have resulted in most of the zinc being lost in vapour. Instead, extracting zinc from its ores required a specific distillation technology in order to condense the fumes into droplets of metal that could be subsequently recovered thanks to specialised vessels. Variants of this technology had been known in India and in China for centuries (Craddock et al 1998; Freestone et al 1985; Zhou 2007; Zhou et al 2012; Craddock 2018), but it was not until the 1730s that a patent for zinc distillation was obtained in Europe, by William Champion from Bristol (Day 1988; 1998; Etheridge and Dungworth 2012). A range of vessels and other residues related to Champion's zinc distillation process have been analysed (Dungworth and White 2007). However, except for some general similarities such as the presence of cadmium, the segregation of tiny lead prills in the zinc phase, and the same zinc oxide/sulfate corrosion of the metallic areas, the remains from Oxford are much smaller in scale when compared to both Champion's Warmley Brass Works and Chinese and Indian operations. For this reason, the activity carried out in the Old Ashmolean laboratory appears more experimental and possibly linked to early attempts at distilling zinc in England not long before Champion's patent.

### 3.4 Other operations

The remaining specimens analysed cannot be ascribed to either glassmaking or zinc distillation activities, but illustrate a wider range of operations at the laboratory. These samples are the crucible fragments AN1999.205.28, AN1999.205.31 and AN1999.205.19 and the fragment from a drug jar AN1999.205.18. The chemical composition of the most relevant phases can be found in **table 5** in the supplementary material. The most interesting specimen is the fragment of graphitic crucible AN1999.205.28, which displays extensive vitrification and deformation of the surface and a thick yellow-green residue coating one side of the vessel's wall (**fig. 12**). This is for the most part made of molten ceramic, as shown by the very diffuse interface between crucible and residue and further suggested by the strong enrichment in silica (53.3 wt% SiO<sub>2</sub>), alumina (18.1 wt% Al<sub>2</sub>O<sub>3</sub>) and titania (1.7 wt% TiO<sub>2</sub>). Other elements making up the glassy residue include potash (14.6 wt% K<sub>2</sub>O), iron oxide (4.6 wt% FeO) and lime (3.3 wt% CaO), plus small quantities of copper and silver (1.2 wt% and 1 wt% respectively). The residue contains a large number of sub-angular kalsilite (KAlSiO<sub>4</sub>) crystals, which often are clustered together and sometimes contain traces of sulfur, silver and copper. The crystals are interspersed with numerous silver-rich globules, typically a few micrometres in diameter and with variable proportions of silver, copper and sulfur. One larger prill of around 100 µm across is almost pure

silver with some 1.3 wt% gold (**fig. 12**). The process that led to the formation of the phases described is unclear. The presence of silver droplets suggests operations involving precious metals, potentially with an eye to alchemical goals of metallic transmutation. The natural philosopher George Starkey conducted extensive experimentation mixing antimony with various other metals when trying to make the philosopher's stone, and interestingly he reports the failure of a Hessian crucible as a result of the extremely corrosive salts he employed (Newman and Principe 2004: 217). In his work diaries, Robert Boyle combines ground silver and copper with either sulfur or tartar in order to produce medicinal substances (Boyle 2004). The addition of tartar and its reaction with the crucible's ceramic may have caused the formation of the kalsilite crystals, and would explain the extremely high potash levels detected in the ceramic. In the heat, tartar would decompose and leave behind just potassium oxide – a very aggressive flux towards all silica-rich materials, including the best Hessian crucibles.



*Figure 12. Sample AN1999.205.28 from graphitic crucible. Note the thick yellowish-green residue coating the inside wall (top). Bottom: SEM backscattered micrographs showing the glassy residue layer sitting on top of the crucible (left,) and detail of the clusters of grey kalsilite crystals interspersed with small metallic droplets (right) (composition in table 5 of supplementary materials).*

The chemical operations that produced the rest of the samples discussed in this section are harder to infer. AN1999.205.31 is a crucible fragment whose surface is also molten and severely altered by heat and possibly chemical attack (**fig. 13 A**). As in the case of the graphitic crucible just discussed, the very high alumina in the vitrified layer (18.6 wt%  $\text{Al}_2\text{O}_3$ ) is likely due to interaction with the ceramic. The

other enrichments hint at an extra source (or sources) of lime (18.7 wt% CaO), magnesia (2.6 wt% MgO), phosphate (1.6 wt% P<sub>2</sub>O<sub>5</sub>) and the oxides of iron (3.5 wt% FeO) and manganese (0.6 wt% MnO). The alkali enrichment is most likely from the ash of charcoal present in the charge. The glass phase contains sub-angular iron-rich crystals (**fig. 13 B**). Sample AN1999.205.19 comes from the base of a crucible with a black glassy residue dripping down the outside surface (**fig. 13 C**). The analysis of the residue shows that the three main components are silica, alumina and iron oxide (57.5 wt%, 21.5 wt% and 12.8 wt% respectively), plus a slight enrichment in fuel ash-related oxides (1.3 wt% MgO, 1.9 wt% K<sub>2</sub>O, 2.9 wt% CaO and 0.4 P<sub>2</sub>O<sub>5</sub>). The glass matrix contains scattered elongated iron-rich crystals bearing small amounts of nickel (0.3-1.2 wt% NiO) (**fig.13 D**). The drug jar fragment AN1999.205.18 belongs to the same vessel type shown in **fig. 2** (n.5). It is made of a white ceramic body with a thin orange deposit on its internal wall (**fig. 13 E-F**). The deposit is very corroded and yielded very low analytical totals. Other than silica and alumina, most likely from molten ceramic, this residue is enriched in lime and iron oxide. The vessel does not show other noticeable areas of discolouration, ceramic distortion or other visible signs of use in high-temperature operations. It is thus likely that pieces of laboratory equipment like this were employed for gentle heating or simply to store chemicals, either in the main room of the laboratory or possibly in the store room adjacent to it (Bennett et al 2000).

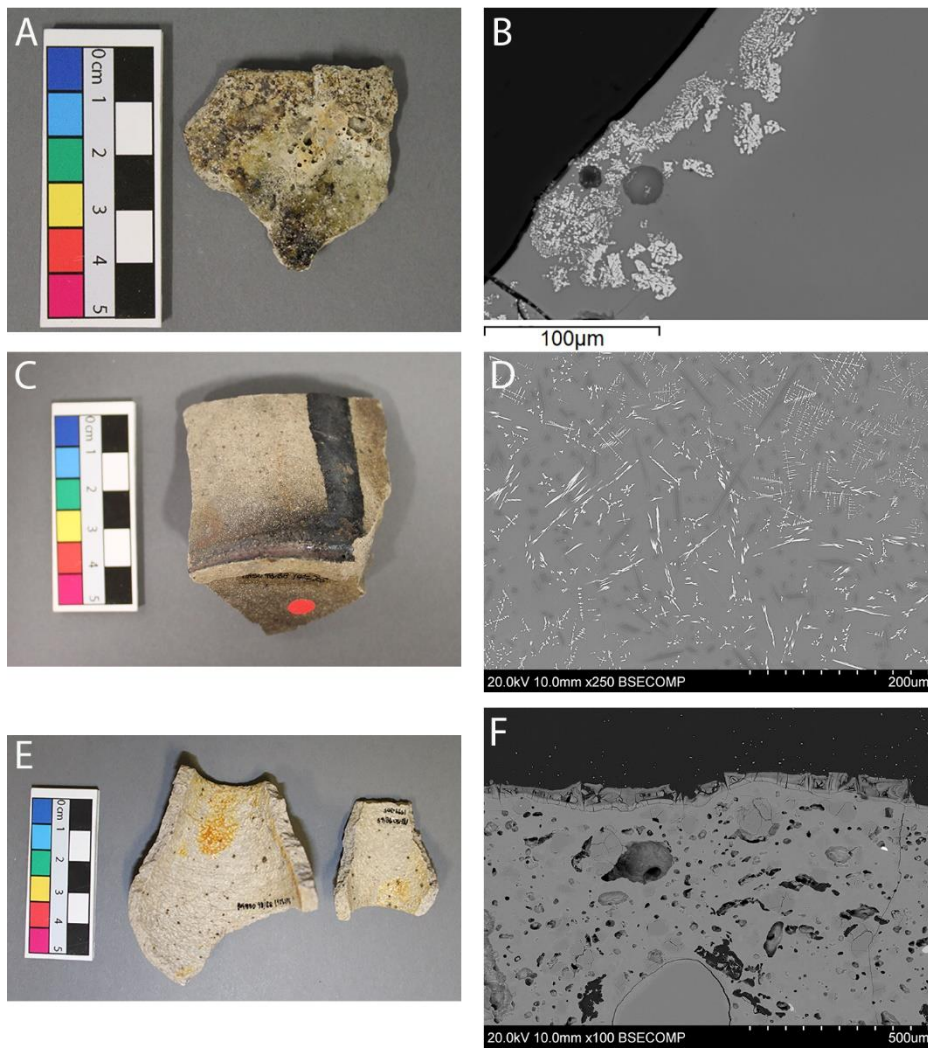


Figure 13. Samples of uncertain interpretation. The two crucible fragments AN1999.205.31 (A-B) and AN1999.205.19 (C-D) show glassy residues stuck to the vessels and iron-rich particles floating in them. E-F: Drug jar AN1999.205.18 made of a white clay body. Note the orange spots on the inner walls which under the SEM look very thin and corroded.

#### 4. Discussion

The results of the analytical investigation on the equipment from the Ashmolean laboratory bear witness to the variety that characterised its research agenda. A wide chemical diversity emerges from a rather limited selection of the objects recovered, suggesting that the laboratory's activity was wide-reaching, multifaceted and markedly experimental. Although the necessarily limited size of the assemblage analysed demands some caution, the laboratory appears especially active in the fields of glass technology and zinc-related metallurgy, areas of major technological innovation between the second half of the 17<sup>th</sup> and the first half of the 18<sup>th</sup> centuries. In the 1660s, not long before the opening of the Museum, the Royal Society had sponsored the English translation of Antonio Neri's *L'Arte Vetraria*, the first treatise to collect hundreds of recipes for making glasses of all sorts (Cable 2006). The translation attracted erudite attention and stimulated experimental research and debate, effectively rendering glass a current topic in scholarly circles (Birch 1756; Hunter and Davis 1999, vol. 6: 476). The invention of lead crystal less than a decade prior to the opening of the Ashmolean Museum must be seen within this atmosphere of excitement, and indeed it was met with interest by the scientific community, so much so that Robert Plot, soon to become the first director of experiments of the Ashmolean laboratory, visited George Ravenscroft's glasshouse (Plot 1677: 253). The wealth of information available after *The Art of Glass* was published sparked both economic and more philosophical interests on the production of colour and opacity effects in glass.

Against this background, the five glass-related crucible fragments each tell a different story. At the most basic level, AN1999.205.9d shows the melting of a lead glass whose composition is in line with the late 17<sup>th</sup> century state of the art. Neither here nor in the other sample with similar composition (AN1999.205.17) could we find conclusive evidence for primary glassmaking. Clusters of residual quartz and feldspar grains were found preserved in some parts of both samples (**fig. 6**), and may represent remains of glass making ingredients, although both glasses are too poor in alumina to be made with such a feldspar-rich mix. In the case of fragment AN1999.205.9d, the residual crystals might be related to the addition of a cobalt-rich ore or a silica-rich preparation to impart the blue colour (Hartwig 2001). The situation seems more uncertain in AN1999.205.17, where the residual grains may represent some sort of reagent added to the glass-rich melt or instead come from an altogether separate event carried out in the same crucible. Sample AN1999.205.14 is remarkable for its substitution of soda for potash in the lead glass matrix. A soda-rich lead glass was employed in Venice as a base composition to make coloured glass in imitation of gemstones (Moretti et al 2004; Cable 2006). However, this used plant ashes as fluxing agent, which does not seem the case in the Ashmolean glass, where fluxes appear very pure. While lead crystal was normally fluxed with saltpetre, Robert Plot also mentions borax (sodium borate) among the ingredients he sees upon visiting Ravenscroft's glasshouse (Plot 1677: 253). The use



of borax is therefore a possibility, but SEM-EDS could not confirm its presence in the samples analysed here. The numerous partly-dissolved quartz particles found in the glass analysed here (**fig. 8**) could indicate a glass making experiment, but this hypothesis seems undermined by the sharp contact of the melt to the ceramic body, and the generally clean composition, which points more towards the re-melting of pre-existing glass. It is quite possible that the operation was aimed at obtaining an opaque white glass, as indicated by the clusters of minute tin-rich crystals, a typical sign of added colourant particles as opposed to crystals grown in the melt (Shortland 2002). At any rate, further sampling of the white glassy area is required in order to offer a final interpretation on this rather puzzling sample. Sample AN1999.205.30 could add further light on the experimentation with white lead glass. Here, the base glass is of the potash-lead composition typical for the late 17<sup>th</sup> century. The high alumina content of more than 5 wt% could indicate the absorption of ceramic material during the melting of the chemically aggressive raw batch. Irrespective of this, it appears that the operator added tin oxide to the melt which then reacted with some of the silica and potash to form crystals of the composition  $K_2SnSi_3O_9$ , an analogue to the mineral wadeite,  $K_2ZrSi_3O_9$ , which itself occurs in alkali-rich igneous rocks that are very low in silica – a setting mimicked here by the potash-lead glass. This phase has not been reported previously in historic glass samples, suggesting that it did not take on as a viable recipe to opacify lead glass. Similarly, sample AN1999.205.25 has elevated alumina levels of more than 6 wt%, indicative of the absorption of ceramic material during high-temperature melting, together with a range of other minor oxides not normally present in high-quality lead glass of the time. What is interesting here is that the glass is based on more than 30 wt% antimony oxide rather than on lead oxide. It is quite possible that the experimenter replaced the red-yellow litharge typically used for lead glass preparations with the similarly red-yellow *glass of antimony*, amorphous antimony oxide, a substance of high interest at the time both as a pharmaceutical and as a chemical substance. Taken together, and considering the purpose of the Ashmolean laboratory as primarily a research and teaching place,<sup>1</sup> the findings demonstrate the range of innovative compositions (lead crystal, both colourless and blue, soda-lead glass, opacification with complex potash-tin-silicates, antimony-based glass) that were studied here. It would be tempting to include the extremely potash-rich graphite-tempered crucible fragment AN1999.205.28 here; however, the presence of silver- and even gold-rich prills, and records preserved in Boyle's work diaries lead to a different interpretation (see below).

The metallurgy of zinc is another technological field that in this period attracted a great deal of scholarly and entrepreneurial attention, resulting in efforts at experimenting with such material. In his 1677

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<sup>1</sup> While the research orientation of the laboratory seems undeniable, it is important to keep in mind that entrepreneurial ambitions did in part fuel the experimental agenda of the laboratory, that the substances produced were often sold and that Robert Plot is known to have entered several contracts with potential buyers of his innovative procedures (see Roos 2014).

*Natural History of Oxfordshire*, Plot discusses zinc ores and mentions the Mendip Hills district near Bristol as the place to find the best calamine in the world (Gunther 1923-1945 vol. 12: 338). Thanks to accumulated experience in metallurgy, experimentation and the ever-growing exposure to this “new” metal coming from abroad, the knowledge around zinc’s elusive nature changed dramatically during the 17<sup>th</sup> century (de Ruelle 1995; Craddock 1998; Day 1998; Martín-Torres and Rehren 2002; Rehren and Martín-Torres 2008). In this sense, William Champion’s 1738 patent for the extraction of zinc through distillation marks a ground breaking discovery and the apex of an artisanal tradition on working with zinc minerals stretching back centuries (Craddock 1998). The evidence collected from the four zinc-rich samples here all points to their use during distillation of metallic zinc – a new concept at the time for a metal and in fact conducted several decades before the formal recognition of zinc as an element in its own right, in 1746. The occurrence of minor amounts of cadmium is consistent with this, given the similarly volatile nature of this metal, and its geochemical close link to zinc ores.

The crucible fragment AN1999.205.28, heavily vitrified and heat-deformed, has been mentioned earlier as unusual and contains three significant components: the extreme potash content, of nearly 20 wt% - exceeding the amount of potash in pure potassium feldspar, the presence of graphitic temper, and numerous prills rich in precious metals. The latter would seem to point towards the exploitation of silver-rich ores; however, apart from some copper there are few if any minor components here that would support such an indication (i.e. barite or calcite gangue, or lead-rich host ore). Instead, the most likely interpretation here is a combination of silver and copper with *tartar* (potassium bitartrate  $KC_4H_5O_6$ ) to produce medicinal substances (Boyle 2004). Only graphite would be resistant against attack, and this may explain why the operator here chose a heavily graphite-tempered crucible to conduct his experiments. The formation of kalsilite, evident in the more vitrified parts of the sample, is normal for such alkali- and alumina-rich but silica-deprived melts. Thus, what we see may not be an economic-geological attempt of silver prospection or of metallic transmutation, but rather a medical recipe implemented through a series of high-temperature chemical reactions.

## 5. Conclusions

This paper has presented new data from the laboratory apparatus from the Old Ashmolean Museum in Oxford. The materials analysed include ceramic crucibles with residues adhering to the inner and outer surfaces, distillation equipment and other types of vessels employed in various chemical operations. The results indicate that the experimental agenda pursued at the site was strongly characterised by what today we would call industrial chemistry, with a breadth of interests that may appear unusual

today, but is characteristic of early modern chymical<sup>2</sup> tradition. The experimental programme involved the testing of technological innovations in artisanal fields such as glass, metallurgy, and iatro-chemistry, the precursor to chemical-based pharmacology. The various substances and processes that the analyses revealed had major economic relevance and eventually resulted in patents being issued and monopolies secured (MacLeod 1987; Brain and Brain 2016, Etheridge and Dungworth 2012). This may partly explain why Plot was so silent regarding the laboratory activities, many of which may have been kept as trade secrets. At the same time, they also show the deep attraction that leading natural philosophers of the late 17<sup>th</sup> and early 18<sup>th</sup> century had towards cutting-edge industrial activities carried out both in England and overseas, and the connection they established with some of the most innovative entrepreneurs of the time. What ultimately emerges from investigating the material culture of the Ashmolean laboratory is a chymical community characterised by a lively interaction between its members, whose aims and motivations were often different but who worked with the same or similar materials and were well aware of one another. The Museum was one of the crossroads of such community, a place where technological and scientific advances could be tested using state-of-the-art equipment, and where the resulting knowledge could be taught and disseminated. Finally, this study also argues for further consideration of the practical dimension of early modern science and for the permeable boundaries between scholarly and artisanal endeavours, where exchanges, overlaps and mutual interaction were the norm.

### **Acknowledgments**

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<sup>2</sup> When referring to early modern practices that, as shown here, largely cross the boundaries between science and craft/industry, historians now tend to use the neutral term chymistry, which conveys the multifaceted character of this phenomenon (on this see Newman and Principe 1998 and Principe and Newman 2001).

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